

NaBH₄/[bmim]BF₄: a New Reducing System to Access Vinyl Selenides and Tellurides

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Um método simples e geral foi desenvolvido para a síntese de selenetos e teluretos vinílicos a partir de alquinos terminais e dicalcogenetos de diorganofla usando NaBH₄ e [bmim]BF₄ como solvente reciclável. Este método eficiente permite a formação preferencial de calcogenetos vinílicos de configuração *Z* na maioria dos exemplos estudados. Também foi observado que na reação envolvendo fenilacetileno, (*E*)-bis-fenilcalcogenoestirenos foram obtidos com bons rendimentos e alta seletividade. O líquido iônico foi reutilizado três vezes sem perda da eficiência.

A general and simple method for the synthesis of vinyl selenides and tellurides starting from terminal alkynes and diorganyl chalcogenides using NaBH₄ and [bmim]BF₄ as a recyclable solvent was developed. This efficient and improved method furnishes the corresponding vinyl chalcogenides preferentially with *Z* configuration. We also observed that when the same protocol was applied to phenyl acetylene, (*E*)-bis-phenylchalcogeno styrenes were obtained in good yields and high selectivity. The ionic liquid was reused up three times without lost of efficiency.

Keywords: ionic liquids, hydrochalcogenation, vinyl chalcogenides

Introduction

Vinyl chalcogenides have been found to be a very useful tool in organic synthesis, since they are very versatile intermediates for the selective construction of isolated or conjugated olefins.¹⁻¹⁰ Besides, organoselenium and organotellurium compounds have attracted increased interest because of their unique biological and pharmacological properties.^{11,12} In this way, various methods are mentioned for the preparation of vinyl chalcogenides and the most common protocols involve the addition of organo chalcogenol, or the respective chalcogenolate anions, to terminal or internal alkynes.^{1-10,13-20} On the other hand, ionic liquids (ILs) are receiving much attention in organic synthesis, both as recyclable solvents and/or catalysts.²¹⁻³² Because product isolation or catalyst recycling is very easy in ILs and, in some cases, rate accelerations and/or selectivity improvements are also observed, they are regarded as environmentally friendly green solvents. In despite of the high versatility of vinyl chalcogenides and the green feature of ILs, their use as solvent for

hydrochalcogenation reaction of alkynes was scarcely explored.³³ As a continuation of our studies toward the development of new and cleaner methods for the synthesis of organochalcogenides,³³⁻⁴⁰ we report herein the full results on the hydrochalcogenation of alkynes using NaBH₄ and [bmim]BF₄ as recyclable solvent for the synthesis of vinyl selenides and tellurides (Scheme 1).

Experimental

General remarks

Proton nuclear magnetic resonance spectra (¹H NMR) were obtained at 200 MHz on a Bruker DPX-200 NMR spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃ or tetramethylsilane (TMS) as the internal reference. Data are reported as follows: chemical shift (δ), multiplicity, coupling constant (*J*) in Hertz and integrated intensity. Carbon-13 nuclear magnetic resonance spectra (¹³C NMR) were obtained at 50 MHz on a Bruker DPX-200 NMR spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical

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shifts are reported in ppm, referenced to the solvent peak of CDCl₃. Column chromatography was performed using Merck Silica Gel (230-400 mesh) following the standard methods. Thin layer chromatography (TLC) was performed using Merck Silica Gel GF₂₅₄, 0.25 mm thickness. For visualization, TLC plates were either placed under ultraviolet light, or stained with iodine vapor, or acidic vanillin. The reactions were monitored by TLC for disappearance of starting material. Reactions were conducted in flame-dried glassware equipped with tightly fitted rubber septa and under a positive atmosphere of dry nitrogen. Reagents were handled using standard syringe techniques.

General procedure for the synthesis of vinyl chalcogenides

To a mixture of alkyne (2.0 mmol) and diphenyl diselenide (0.156 g; 0.5 mmol) in [bmim]BF₄ (0.5 mL) under N₂ atmosphere, NaBH₄ (0.045 g; 1.2 mmol) was added at room temperature. Then, the temperature was slowly raised to 60 °C. The reaction progress was followed by TLC and after the time described on Table 1 the reaction mixture was extracted using ether (5 × 3 mL). The solvent was evaporated under reduced pressure and the residue was purified by column chromatography over silica gel eluting with hexanes or a mixture hexanes/ethyl acetate. For the synthesis of the vinyl tellurides analogs it was used 1.2 mmol of the alkyne. Spectral data of the products prepared are listed below.

(2Z)-3-(Phenylseleno)prop-2-en-1-ol (**3a**)³³

Yield: 0.135g (63%). ¹H NMR (200 MHz, CDCl₃) δ 7.54-7.43 (m, 2H); 7.27-7.22 (m, 3H); 6.58 (dt, *J* 9.4 and 1.4 Hz, 1H); 6.19 (dt, *J* 9.4 and 6.0 Hz, 1H); 4.24 (dd, *J* 6.0 and 1.4 Hz, 2H); 3.02 (br s, 1H); ¹³C NMR (50 MHz, CDCl₃) δ 134.54, 132.52, 131.81, 129.14, 127.07, 122.83, 61.26.

2-(Phenylseleno)prop-2-en-1-ol (**4a**)³³

Yield: 0.051g (24%). ¹H NMR (200 MHz, CDCl₃) δ 7.54-7.43 (m, 2H); 7.27-7.22 (m, 3H); 5.86 (s, 1H); 5.40 (s, 1H); 4.15 (s, 2H); 3.02 (br s, 1H); ¹³C NMR (50 MHz, CDCl₃) δ 141.37, 133.83, 129.24, 128.12, 127.72, 118.19, 66.25.

(3Z)-2-Methyl-4-(phenylseleno)but-3-en-2-ol (**3b**)⁴¹

Yield: 0.150g (62%). ¹H NMR (200 MHz, CDCl₃) δ 7.57-7.51 (m, 2H); 7.28-7.24 (m, 3H); 6.43 (d, *J* 10.0 Hz, 1H); 6.00 (d, *J* 10.0 Hz, 1H); 2.15 (br s, 1H); 1.41 (s, 6H); ¹³C NMR (50 MHz, CDCl₃) δ 136.78, 132.82, 132.63, 128.99, 127.09, 120.59, 72.77, 29.48 (2C).

(1Z)-3-Methyl-1-(phenylseleno)pent-1-en-3-ol (**3c**)⁴¹

Yield: 0.230g (90%). ¹H NMR (200 MHz, CDCl₃) δ 7.54-7.51 (m, 2H); 7.29-7.25 (m, 3H); 6.48 (d, *J* 10.1 Hz, 1H); 5.93 (d, *J* 10.1 Hz, 1H); 2.09 (br s, 1H); 1.66 (q, *J* 7.5 Hz, 2H); 1.35 (s, 3H); 0.96 (t, *J* 7.5 Hz, 3H); ¹³C NMR (50 MHz, CDCl₃) δ 135.49, 132.95, 132.39, 128.97, 127.03, 121.21, 75.34, 35.00, 27.16, 8.21.

(Z)-(2-Phenylseleno-vinyl)-1-cyclohexanol (**3d**)³⁹

Yield: 0.229g (81%). ¹H NMR (200 MHz, CDCl₃) δ 7.57-7.52 (m, 2H); 7.31-7.26 (m, 3H); 6.47 (d, *J* 9.8 Hz, 1H); 6.04 (d, *J* 9.8 Hz, 1H); 1.68-1.51 (m, 10H); 1.83 (br s, 1H); ¹³C NMR (50 MHz, CDCl₃) δ 136.31, 133.02, 132.66, 129.10, 127.21, 121.26, 73.92, 37.42 (2C), 25.31, 22.08.

(2Z)-3-(Phenyltelluro)prop-2-en-1-ol (**3h**)⁴¹

Yield: 0.169g (64%). ¹H NMR (200 MHz, CDCl₃) δ 7.74-7.69 (m, 2H); 7.29-7.25 (m, 3H); 6.92 (dt, *J* 9.6 and 1.4 Hz, 1H); 6.53 (dt, *J* 9.6 and 5.0 Hz, 1H); 4.24-4.22 (m, 2H); 2.50 (br s, 1H); ¹³C NMR (50 MHz, CDCl₃) δ 138.99, 137.38, 136.53, 129.13, 127.50, 107.40, 64.07.

2-(Phenyltelluro)prop-2-en-1-ol (**4h**)⁴¹

Yield: 0.063g (24%). ¹H NMR (200 MHz, CDCl₃) δ 7.82-7.76 (m, 2H); 7.33-7.20 (m, 3H); 6.25 (t, *J* 1.7 Hz, 1H); 5.57 (t, *J* 1.7 Hz, 1H); 4.21 (s, 2H); 2.46 (br s, 1H); ¹³C NMR (50 MHz, CDCl₃) δ 138.96, 129.86, 129.33, 127.94, 123.31, 112.06, 69.19.

(3Z)-2-Methyl-4-(phenyltelluro)but-3-en-2-ol (**3i**)⁴¹

Yield: 0.187g (64%). ¹H NMR (200 MHz, CDCl₃) δ 7.81-7.75 (m, 2H); 7.29-7.17 (m, 3H); 6.68 (d, *J* 9.8 Hz, 1H); 6.43 (d, *J* 9.8 Hz, 1H); 2.26 (br s, 1H); 1.36 (s, 6H); ¹³C NMR (50 MHz, CDCl₃) δ 141.28, 140.37, 137.75, 129.02, 127.42, 104.45, 73.31, 29.14.

(1Z)-3-Methyl-1-(phenyltelluro)pent-1-en-3-ol (**3j**)⁴¹

Yield: 0.211g (69%). ¹H NMR (200 MHz, CDCl₃) δ 7.79-7.74 (m, 2H); 7.27-7.20 (m, 3H); 6.72 (d, *J* 10.0 Hz, 1H); 6.37 (d, *J* 10.0 Hz, 1H); 2.04 (br s, 1H); 1.63 (q, *J* 7.5 Hz, 2H); 1.31 (s, 3H); 0.95 (t, *J* 7.5 Hz, 3H); ¹³C NMR (50 MHz, CDCl₃) δ 140.50, 139.97, 137.60, 128.90, 127.27, 105.15, 75.51, 34.70, 26.79, 7.99.

(Z)-(2-Phenyltelluro-vinyl)-1-cyclohexanol (**3k**)³⁹

Yield: 0.222g (67%). ¹H NMR (200 MHz, CDCl₃) δ 7.79-7.74 (m, 2H); 7.28-7.16 (m, 3H); 6.69 (d, *J* 9.9 Hz, 1H); 6.47 (d, *J* 9.9 Hz, 1H); 1.67-1.25 (m, 10H); 1.87 (br s, 1H); ¹³C NMR (50 MHz, CDCl₃) δ 140.92, 137.87, 129.09, 127.42, 118.85, 104.95, 74.43, 36.82, 25.21, 21.80.

(*Z*)-1-(Butyltelluro)prop-2-en-1-ol (**3n**), 2-(Butyltelluro)prop-2-en-1-ol (**4n**)⁴²

Yield: 0.207g (85%). ¹H NMR (200 MHz, CDCl₃) δ 6.77 (ddd, *J* 9.6, 1.2 and 1.5 Hz, 0.1H); 6.40 (ddd, *J* 9.6, 5.2 and 5.4 Hz, 0.1H); 6.16 (dd, *J* 1.6 and 1.5 Hz, 0.9H); 5.52 (dd, *J* 1.3 and 1.2 Hz, 0.9H); 4.19 (s, 1.8H); 4.11 (dd, *J* 3.6 and 3.3 Hz, 0.2H); 2.72 (t, *J* 7.5 Hz, 1.8H); 2.60 (t, *J* 6.8 Hz, 0.2H); 1.85-1.70 (m, 2H); 1.45-1.25 (m, 2H); 0.96-0.89 (m, 3H); ¹³C NMR (50 MHz, CDCl₃) δ 137.41, 127.11, 122.73, 104.68, 70.12, 64.95, 34.03, 33.72, 25.08, 24.88, 13.39, 7.14, 6.11.

(*Z*)-(2-Butyltelluro-vinyl)-benzene (**3p**)⁴³

Yield: 0.232g (80%). ¹H NMR (200 MHz, CDCl₃) δ 7.35 (d, *J* 10.6 Hz, 1H); 7.31-7.13 (m, 5H); 6.90 (d, *J* 10.6 Hz, 1H); 2.62 (t, *J* 6.7 Hz, 2H); 1.75 (quint, *J* 6.7 Hz, 2H); 1.32 (sex, *J* 6.7 Hz, 2H); 0.85 (t, *J* 6.7 Hz, 3H); ¹³C NMR (50 MHz, CDCl₃) δ 138.89, 136.68, 128.23, 127.46, 127.13, 105.29, 33.87, 24.85, 13.35, 8.95.

(*E*)-1-Phenyl-1,2-bis-(phenylseleno)ethene (**5a**)⁴⁴

Yield: 0.177g (85%). ¹H NMR (200 MHz, CDCl₃) δ 7.52-7.09 (m, 15H); 7.02 (s, 1H); ¹³C NMR (50 MHz, CDCl₃) δ 139.51, 133.01, 132.08, 131.13, 130.62, 130.44, 129.24, 129.15, 128.59, 128.28, 128.24, 127.43, 127.40, 126.04.

(*E*)-1-Phenyl-1,2-bis-(phenyltelluro)ethene (**5b**)³⁹

Yield: 0.211g (82%). ¹H NMR (200 MHz, CDCl₃) δ 7.73 (d, *J* 6.8 Hz, 2H); 7.56 (d, *J* 6.8 Hz, 2H); 7.37 (s, 1H); 7.17-7.33 (m, 11H); ¹³C NMR (50 MHz, CDCl₃) δ 144.61, 139.06, 137.64, 129.67, 129.49, 128.70, 128.30, 128.20, 127.93, 127.61, 120.97, 116.03, 115.76, 115.09.

General procedure for the reuse of ionic liquid

The same procedure as for hydrochalcogenation of alkynes described before was followed. After work-up, [bmim]BF₄ was diluted with dichloromethane (3.0 mL) and filtered over Celite. The solution was dried over MgSO₄ and the volatiles were removed under vacuum. The recovered ionic liquid was reused for the next reaction.

Results and Discussion

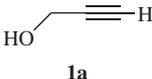
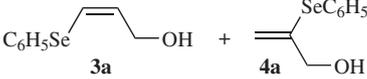
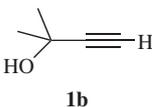
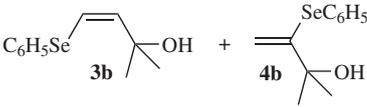
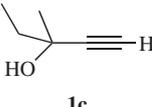
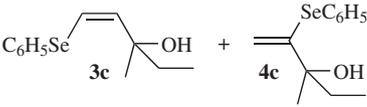
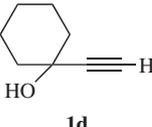
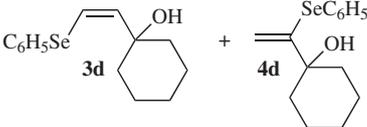
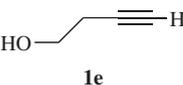
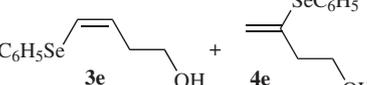
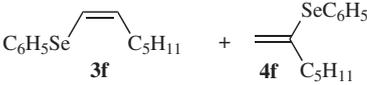
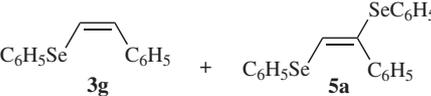
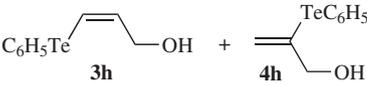
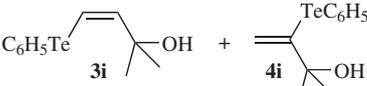
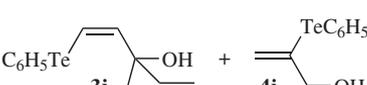
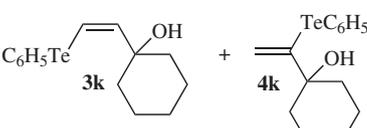
Initially, we chose propargyl alcohol (**1a**; 2.0 mmol) and diphenyl diselenide (**2a**; 0.5 mmol) to establish the best conditions for the hydroselenation reaction. We examined the temperature, amounts of IL and NaBH₄ and the use of N₂ atmosphere. It was found that using 1.2 mmol of NaBH₄ and 0.5 mL of IL at room temperature and under N₂

atmosphere, the reaction proceeded slowly furnishing the product in 30% yield after stirring for 26 h. However, when the mixture was heated at 60 °C, the desired products **3a** and **4a** were obtained in good yields (87%) after 6 h (Table 1, entry 1). When the same protocol was performed without N₂ it was observed, after 10 h, incomplete consume of **2a** and the products were isolated in 48% yield. When the reaction was performed in the presence of [bmim]BF₄ alone, without NaBH₄, no reaction took place in all conditions tested and the starting materials were recovered. Besides, using a smaller amount of NaBH₄ (1.0 mmol), the desired products **3a** and **4a** were obtained only in 52% yield. The use of a slighter amount of **1a** (1.5 mmol) afforded also lower yield of products (68%).

Since the best conditions were established, the protocol was extended to other alkynyl alcohols with diphenyl diselenide (Scheme 1). In all the studied cases, a mixture of (*Z*)-**3** and gem-**4** was obtained in good yields (Table 1, entries 1-5) and, except for homopropargyl alcohol **1e**, the anti-Markovnikov adduct **3** was obtained in higher amount than the Markovnikov one **4** (Table 1, entry 5). For the reaction of phenylselenolate anions with sterically hindered alcohols, it was observed that steric factors are important, because both, the **3:4** ratio and the reaction time, increased with the R group size (Table 1, compare entries 2-4 with 1 and 5). This regioselectivity is similar to that reported for the methods which use organic solvents.^{1,13-20} When the same protocol was applied to phenyl acetylene **1g**, (*E*)-1,2-bis-phenylseleno styrene **5a** was obtained in 85% yield after 3 h at 60 °C (Table 1, entry 7). This result is similar to that obtained under solvent-free conditions.³⁴

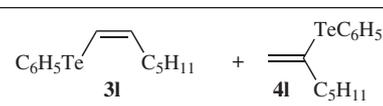
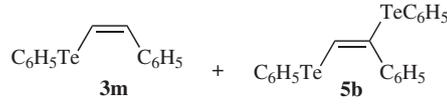
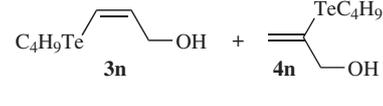
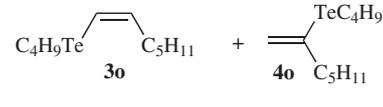
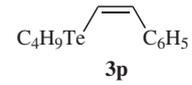
Aiming to extend the scope of this new method, we performed the hydrotelluration of terminal alkynes. Thus, when diphenyl ditelluride **2b** (0.5 mmol) reacted with propargyl alcohol **1a** (2.0 mmol) in the presence of NaBH₄ (1.2 mmol) and [bmim]BF₄ (0.5 mL), the respective vinyl tellurides **3h** and **4h** were obtained in 89% yield after stirring at 60 °C for 6.5 h. In contrast to the observed for the hydroselenation reaction, a large excess of alkyne is not necessary. Thus, when 1.2 mmol of propargyl alcohol **1a** reacted with 0.5 mmol of C₆H₅TeTeC₆H₅ under the same conditions, a mixture of the vinyl tellurides **3h** and **4h** was obtained in 88% yield after 7 h (**3h:4h** ratio = 73:27; Table 1, entry 8). Similarly to the selenium analogs, the reaction works well with other alkynols, giving preferentially the respective anti-Markovnikov adducts **3i-k** in good yields (Table 1, entries 9-11). Equally to the observed for diphenyl diselenide, the reaction with phenylacetylene gave the corresponding product **5b** in 82% yield, but with a ration (*Z*)-**3m:5b** = 5:95 (Table 1, entry 13). When dibutyl ditelluride **2c** was used as

Table 1. Hydrochalcogenation of alkynes using [bmim]BF₄ as recyclable solvent

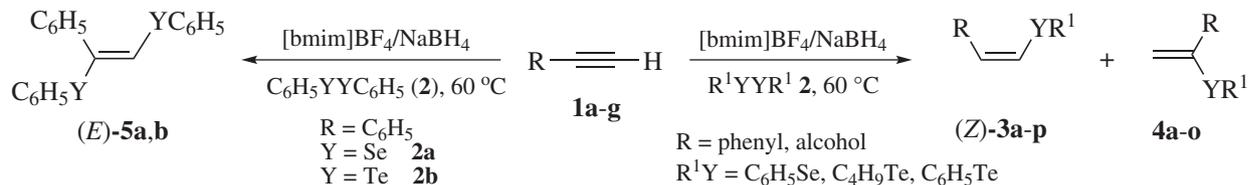
Entry	Alkyne 1	Chalcogenide 2	Products 3 + 4	time (h)	Yield ^a (%)	Ratio ^b (Z)- 3:4
1		$C_6H_5SeSeC_6H_5$ 2a		6	87	72:28
2		2a		10	62	91:9
3		2a		9	90	80:20
4		2a		10	81	>95:5
5		2a		6	74	44:56
6	C_5H_{11} -C≡C-H 1f	2a		9	trace	-
7	C_6H_5 -C≡C-H 1g	2a		3	85	c
8	1a	$C_6H_5TeTeC_6H_5$ 2b		7	88	73:27
9	1b	2b		10	64	90:10
10	1c	2b		8	69	93:07
11	1d	2b		11	67	94:06

^aYields of pure products isolated by column chromatography (hexanes/AcOEt) and identified by ¹H and ¹³C NMR. ^bDetermined by GC of the crude reaction mixture and confirmed after isolation of the individual isomers. ^cObtained as mixture of (Z)-**3g** and (E)-**5a** (ratio 26:74). ^dObtained as mixture of (Z)-**3m** and (E)-**5b** (ratio = 5:95).

Table 1. Continuation

Entry	Alkyne 1	Chalcogenide 2	Products 3 + 4	time (h)	Yield ^a (%)	Ratio ^b (Z)- 3 : 4
12	1f	2b		12	trace	-
13	1g	2b		6	82	d
14	1a	C ₄ H ₉ TeTeC ₄ H ₉ 2c		5	85	15:85
15	1f	2c		12	trace	-
16	1g	2c		6	80	100:0

^aYields of pure products isolated by column chromatography (hexanes/AcOEt) and identified by ¹H and ¹³C NMR. ^bDetermined by GC of the crude reaction mixture and confirmed after isolation of the individual isomers. ^cObtained as mixture of (Z)-**3g** and (E)-**5a** (ratio 26:74). ^dObtained as mixture of (Z)-**3m** and (E)-**5b** (ratio = 5:95).

**Scheme 1.**

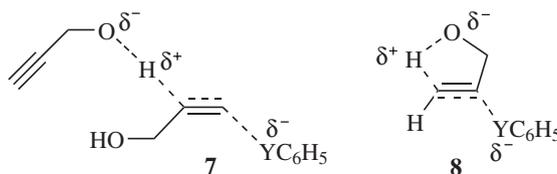
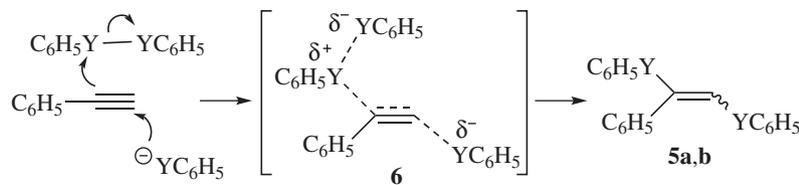
organochalcogenium source, the results were different of those observed with the diphenyl analogs **2a** and **2b**. In the reaction with propargyl alcohol **1a**, a mixture of vinyl tellurides **3n** and **4n** was obtained in 85% yield after 5 h at 60 °C, with the Markovnikov adduct **4n** being predominant (**3n**:**4n** ratio = 15:85; Table 1, entry 14).

When **2c** reacted with phenylacetylene **1g** under the same conditions, only (Z)-(2-butyltelluro-vinyl)-benzene, **3p** was obtained in 80% yield (Table 1, entry 16). Unfortunately, no products were isolated when 1-heptyne **1f** was used as starting alkyne, with only trace amount being detected by GC (Table 1, entries 6, 12 and 15).

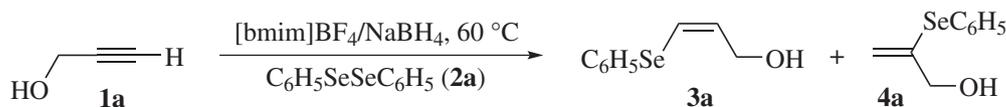
A plausible mechanism for the reaction of phenyl acetylene with diphenyl dichalcogenides using [bmim]BF₄ as solvent for formation of bis- and mono-phenylchalcogen alkenes respectively, is depicted on Scheme 2. The formation of bis-phenylchalcogen alkenes **5a** and **5b** can be attributed probably to the low solubility of the starting

reagents **2a** and **2b** in the ionic liquid compared with **2c**, which makes the reaction behavior like a solvent-free one, with formation of an intermediate analogous to **6** (Scheme 2).³⁴ A free radical chain addition mechanism could also be involved.⁴⁵ For the propargylic alcohols, however, the formation of the respective bis-chalcogen alkenes were not observed. This fact suggests that in this case, the intermediates **7** and **8** could be involved in the formation of **3** and **4**, respectively.⁴⁶

To check the efficacy of this method, a reuse study of the ionic liquid medium [bmim]BF₄ was carried out for the reaction showed in Scheme 3. After completion of the hydroselenation of propargyl alcohol **1a**, the reaction mixture was diluted with ethyl ether and the product was isolated. The ionic liquid was recovered, dried under vacuum and reused for further reactions. The ionic liquid maintained its good level of efficiency even after being reused three times (Table 2).



Scheme 2.



Scheme 3.

Table 2. Reuse of [bmim]BF₄ in hydroselenation of alkyne **1a**

Run	Yield 3a + 4a (%) ^c	Ionic Liquid recovery (%)
1 ^a	87	99
2 ^b	84	99
3 ^b	83	98
4 ^b	79	97

^aReactions were performed using alkyne **1a** (2.0 mmol), diphenyl diselenide **2a** (0.5 mmol) and NaBH₄ (1.2 mmol) in [bmim]BF₄ (0.5 mL) under N₂ atmosphere at 60 °C. ^bRecovered ionic liquid was used. ^cObtained as a mixture of (*Z*)-**3a** and **4a** (ratio = 72:28).

Conclusions

We demonstrated the use of NaBH₄/[bmim]BF₄ as a reducing system for the addition of chalcogenolate anions to alkynyl alcohols and phenylacetylene to give selectively mono- and bis-organochalcogenides alkenes. This improved, simple, fast and clean protocol occurs under mild conditions, with non-aqueous work-up and the ionic liquid can be easily recovered and utilized for further reactions.

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