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Kinetics and mechanism of the pyridine-catalyzed reaction of phenylselenenyl halides and some unsaturated alcohols

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ABSTRACT

The kinetics and mechanism of the reaction of phenylselenenyl halides (PhSeX, X = Cl, Br) and some primary unsaturated alcohols (pent-4-en-1-ol and hex-5-en-1-ol) in tetrachloromethane media have been studied, under the *pseudo*-first order conditions, in the presence and absence of pyridine as catalyst by variable temperature UV–vis spectrophotometry. Under the kinetic conditions, both the slower uncatalyzed and faster catalyzed (by pyridine) paths give the change of absorbance of the reaction mixture at some wavelength. The obtained values for rate constants have shown that the reactions with phenylselenenyl bromide are slower. Also, the reactions of both phenylselenenyl halides with hex-5-en-1-ol are faster than those with pent-4-en-1-ol. The negative values for entropy of activation for all studied reactions confirm the S_N2 mechanism of substitution.

1. Introduction

During last years, cyclic ether units are important synthetic targets in organic and medical chemistry due to their widespread occurrence in many complex natural compounds, exhibiting important biological activities [1]. These units can be found isolated in monocyclic or polycyclic compounds, fused with other cyclic ethers or forming spiro systems [2]. The presence of molecules with oxygenated heterocycles in nature is receiving considerable attention considering their capacity for modification of the transport of metallic cations Na^+, K^+, and Ca^{2+} through the lipid membranes [3,4], this activity being responsible for their antibiotic [3], neurotoxic [5], antiviral [6] and cytotoxic actions [7] and as growth regulators [3,8] or inhibitors of the level of cholesterol in blood [9].

A number of synthetic approaches have been devised in order to construct the cyclic ether moiety, using a carbon–carbon [10] or a carbon–oxygen [11] cyclization step or modifying cyclic precursor [12]. For some time we have been involved in the development and exploration of new methods for cyclofunctionalization of unsaturated alcohols [11h,12g,15–17].

For many reasons selenocyclofunctionalization has the advantage because the introduction of the heteroatom, the manipulation of the obtained product and the removal of the function are facilitated by simple and mild condition required [11–17]. This methodology has been extended to more complex systems having alcohol and double bond functions. The reactions of phenylselenenyl halides and unsaturated alcohols are usually considered to be a two-step mechanism: electrophilic addition of the reagent to the double bond of the alkenols and nucleophilic attack of the hydroxylic oxygen results in the formation of a ring. That is a rate-determining first step leading to the formation of a seleniranium ion followed by a second product-determining step (Fig. 1).

However such a mechanism is not unique. Indeed if the reaction of phenylselenenyl halides and alkenols is regarded as a nucleophilic displacement at bivalent selenium, several variations of this two-step mechanism can be envisioned: the first, analogous to the S_N1 mechanism at a saturated carbon atom, and second, analogous to S_N2 mechanism and finally an addition–elimination mechanism [18].

Recently, we presented an approach to cyclic ethers from tertiary alkenols using PhSeX (X = Cl, Br) in the presence of pyridine [16,17]. Procedure works smoothly resulting in quantitative formation of the cyclic ethers. We were interested in exploring how primary alkenols pent-4-en-1-ol and hex-5-en-1-ol behave in the presence of pyridine and have therefore undertaken a study of the reaction of these alkenols with PhSeX (X = Cl, Br) in the presence of pyridine which is resulting in almost quantitative formation of the cyclic ethers (Fig. 1). This observation may be ascribed to the role of the pyridine. It appears that the presence of pyridine is beneficial to the cyclization process and more likely due to its basic properties. In addition, pyridine could enhance the nucleophilicity of the hydroxyl group of the alkenol and also mediate the stabilization of oxonium ion intermediates by abstracting

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the hydrogen (1b and 2b, Fig. 1). It seems that pyridine could play several roles. Prompted by what we found, we considered it synthetically interesting and profitable for our purposes to do some kinetic measurements to confirm that observation. We have used a conventional kinetic method to determine the values of rate constants and other thermodynamic parameters.

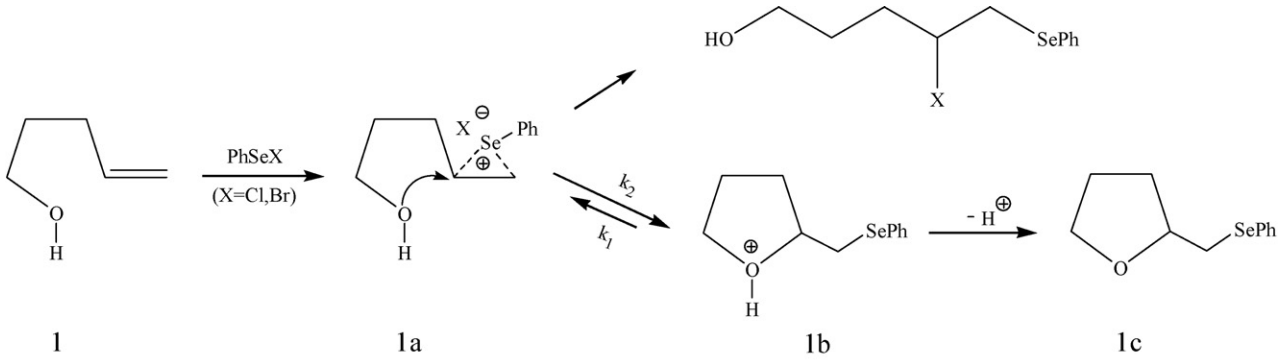
2. Experimental

2.1. Instrumentation

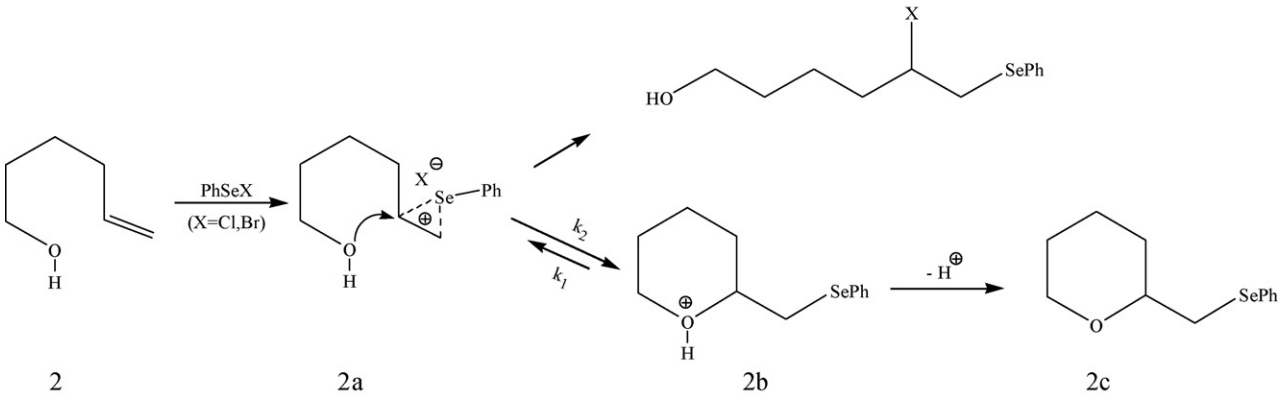
Gas–liquid chromatography (GLC) analysis was performed by a Deni instrument, model 2000 with capillary apolar columns. ^{1}H and ^{13}C NMR spectra were run in $CDCl_3$ on a Varian Gemini 200 MHz NMR spectrometar. IR spectra were obtained with PerkinElmer Model 137B and Nicolet 7000 FT spectrophotometers. Microanalyses were performed in "Dornis and Colbe" laboratory (Germany). Thin-layer chromatography (TLC) was carried out on 0.25 mm E. Merck precoated silica gel plates (60F-254) using UV light for visualization. For column chromatography, E. Merck silica gel (60, particle size 0.063–0.200 mm) was used.

2.2. General procedure

All synthetic reactions (for preparation, isolation and identification of cyclic ethers 1c and 2c, Fig. 1) were carried out on a 1 mmol scale. To a magnetically stirred solution of alkenol (1 mmol) and catalyst (1 mmol) in dry dichloromethane (dried over $CaCl_2$) or tetrachloromethane (the results are almost equal in both solvents, but it is better to use dichloromethane for the reason of easily removing from reaction mixture) dried over $CaCl_2$ (5 cm^3) solid PhSeCl (0.212 g, 1.1 mmol) or PhSeBr (0.260 g, 1.1 mmol) was added at room temperature. The reaction was completed in few minutes. The pale yellow solution was washed with 1 M HCl aqueous solution (5 cm^3), saturated $NaHCO_3$ aqueous solution and water. The organic



Yields of cyclic ether product:	PhSeCl	PhSeCl/py
	69%	≈100%
	PhSeBr	PhSeBr/py
	63%	≈100%



Yields of cyclic ether product:	PhSeCl	PhSeCl/py
	81%	≈100%
	PhSeBr	PhSeBr/py
	75%	≈100%

Fig. 1. Phenylselenoetherification of pent-4-en-1-ol and hex-5-en-1-ol.

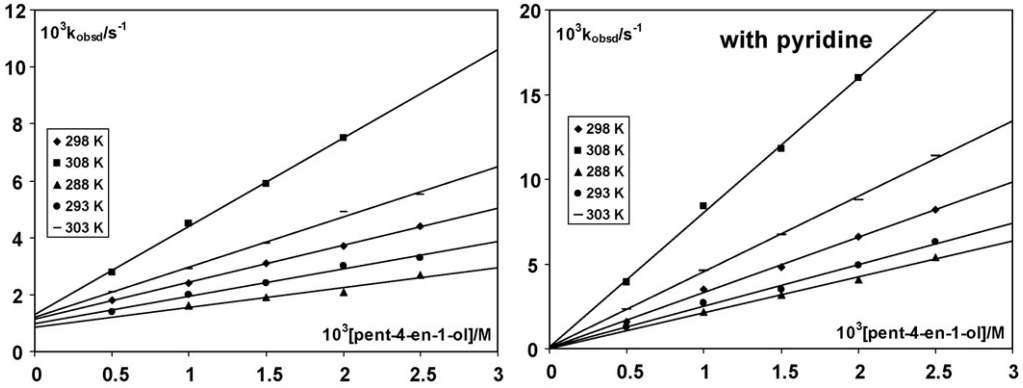


Fig. 2. *Pseudo*-first order rate constants, k_{obsd}, as a function of concentration of alcohol and temperature for the reactions between PhSeCl and pent-4-en-1-ol in CCl_4 in the presence and absence of pyridine.

layer was dried over Na_2SO_4, concentrated and chromatographed. The products (1c and 2c, Fig. 1) were obtained after the eluation of the traces of diphenyl diselenide from a silica gel-dichloromethane column. All the products were characterized and identified on the basis of their spectral data [11h].

2.3. Kinetic measurements

A conventional kinetic method for determination of the values of rate constants and other thermodynamic parameters was used regarded the reaction as a nucleophilic displacement at bivalent selenium.

Substitution reactions between phenylselenenyl halides and primary unsaturated alcohols were studied spectrophotometrically using UV–vis PerkinElmer Lamda 35 spectrophotometer equipped with water thermostated cell. All reactions were followed at five different temperatures (15, 20, 25, 30, and 35 °C). The temperatures of reaction mixtures were controlled throughout all kinetic experiments to ±0.1 °C.

All solutions were prepared by measuring the calculated amounts of substances in tetrachloromethane. Tetrachloromethane is more suitable than dichloromethane for kinetic experiments in terms of the boiling point, but for synthetic purpose it is better to use dichloromethane which is removed easily from reaction mixture and does not change yields. The reactions were initiated by mixing equal volumes of phenylselenenyl halide and alcohol solutions in the quartz cuvette. During all experiments the concentration of halide was constant (1×10^{-4} M), while the concentration

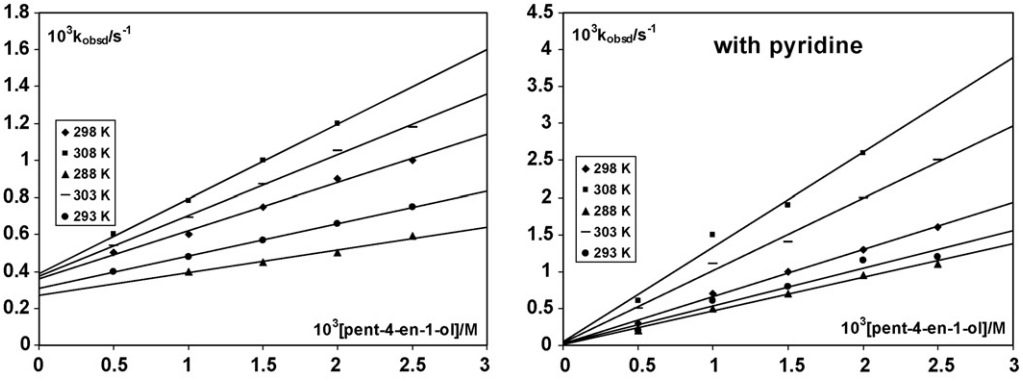


Fig. 3. *Pseudo*-first order rate constants, k_{obsd}, as a function of concentration of alcohol and temperature for the reactions between PhSeBr and pent-4-en-1-ol in CCl_4 in the presence and absence of pyridine.

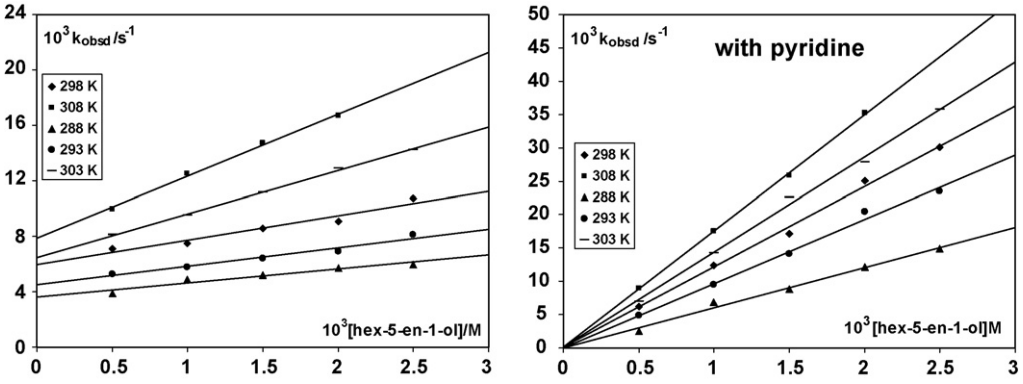


Fig. 4. *Pseudo*-first order rate constants, k_{obsd}, as a function of concentration of alcohol and temperature for the reactions between PhSeCl and hex-5-en-1-ol in CCl_4 in the presence and absence of pyridine.

of alcohol was varied from 5×10^{-4} M to 2.5×10^{-3} M. For the experiments with the presence of pyridine, the concentration of pyridine was equimolar to phenylselenenyl halide concentration.

Spectral changes, resulting from the mixing of phenylselenenyl halide and alcohol solutions, were recorded over the wavelength range 220–600 nm to establish a suitable wavelength at which kinetic experiments could be performed. The *pseudo*-first-order rate constants, k_{obsd}, were determined according to Eq. (1) by fitting all kinetic runs as single exponential function.

$$A_t = A_0 + (A_0 - A_\infty)\exp(-k_{obsd}t) \quad (1)$$

The observed *pseudo*-first order rate constants, k_{obsd}, were calculated as the average value from two to five independent kinetic runs using computational program Microsoft Excel and Origin 6.1. Obtained experimental data are reported in Tables 1S–4S (Supporting Material).

3. Results and discussion

In resent years, we have studied intramolecular cyclization of some Δ^4- and Δ^5-alkenols by means of phenylselenenyl halides [11h], PhSeX (X = Cl, Br). Intramolecular heterocyclization is the main reaction in the case of all investigated primary and secondary alkenols, while tertiary alkenols, under the same experimental conditions, are not converted into cyclic products at all by PhSeBr and in a small amount with PhSeCl. On the other hand in the presence of the pyridine all alkenols (primary, secondary and tertiary) cyclized in excellent yields with both reagents (PhSeCl and PhSeBr) [16,17b].

In this paper the kinetics of the reactions between phenylselenenyl halides (PhSeX, X = Cl, Br) and some unsaturated alcohols (pent-4-en-1-ol and hex-5-en-1-ol) in the presence and absence of pyridine were studied under the *pseudo*-first-order conditions at five different temperatures in tetrachloromethane as a solvent. The calculation of k_{obsd} was explained in Section 2.

The concentration dependence of k_{obsd} for studied reactions is presented by the plots in Figs. 2–5 (data are given in Tables from 1S to 4S in Supporting Material). However, k_{obsd} linearly depends of the concentration of alcohol.

The observed rate constants, k_{obsd}, as a function of total alcohol concentration can be described by the following equation:

$$k_{obsd} = k_1 + k_2[\text{alcohol}] \quad (2)$$

In this equation k_2 presents the second order rate constant for the forward reaction, which depends on alcohol concentration, and k_1 shows the effect of reverse (or parallel) reactions on the substitution process (Fig. 1). The rate constant k_1 is independent of the alcohol concentration. The values for k_2 were calculated from the slopes of the plots k_{obsd} versus of the alcohol concentration [19], while the values for k_1 were determined from the intercept of the observed lines (Figs. 2–5).

The experimental data are summarized in Figs. 2–5 and calculated values for rate constants and activation parameters are given in Table 1.

Data from Table 1 show that the second order rate constants, k_2, are about three to five order of magnitude larger than k_1.

Hex-5-en-1-ol is more reactive than pent-4-en-1-ol for all studied conditions. This confirms the fact that in the process of intramolecular cycliyzation it is much easier to form cyclic ether by nucleophilic attack of OH group staring from hex-5-en-1-ol than from pent-4-en-1-ol, which also gives the more thermodynamic stable products (2c, Fig. 1) than with pent-4-en-1-ol (1c, Fig. 1). So, the type of alkenols has a large influence on the kinetic and thermodynamic characteristics of cyclization products. Also, PhSeCl is more reactive than PhSeBr.

Table 1
Rate constants and activation parameters for the reactions between phenylselenenyl halides and unsaturated primary alcohols in the presence and absence of pyridine in CCl_4

	PhSeCl							PhSeBr					
	T (K)	λ (nm)	k_2 ($M^{-1}s^{-1}$)	k_1 (s^{-1})	E_a (kJ M^{-1})	$\Delta H^{\neq}$ (kJ M^{-1})	$\Delta S^{\neq}$ (J $K^{-1}M^{-1}$)	λ (nm)	k_2 ($M^{-1}s^{-1}$)	k_1 (s^{-1})	E_a (kJ M^{-1})	($H^{\neq}$ (kJ M^{-1})	($S^{\neq}$ (J $K^{-1}M^{-1}$)
pent-4-en-1-ol	288	280	0.70 ± 0.01	(0.85 ± 0.02) × 10^{-3}	53	50 ± 2	−74 ± 3	275	0.12 ± 0.01	(2.7 ± 0.2) × 10^{-4}	44	42 ± 3	−1.19 ± 5
	293		0.96 ± 0.06	(0.98 ± 0.09) × 10^{-3}					0.18 ± 0.02	(3.1 ± 0.4) × 10^{-4}			
	298		1.30 ± 0.02	(1.13 ± 0.03) × 10^{-3}					0.26 ± 0.01	(3.6 ± 0.3) × 10^{-4}			
	303		1.76 ± 0.08	(1.20 ± 0.08) × 10^{-3}					0.33 ± 0.01	(3.4 ± 0.2) × 10^{-4}			
	308		3.10 ± 0.08	(1.3 ± 0.1) × 10^{-3}					0.40 ± 0.01	(3.9 ± 0.2) × 10^{-4}			
pent-4-en-1-ol and pyridine	288	295	2.12 ± 0.09	(2.4 ± 0.2) × 10^{-5}	48	45 ± 3	−83 ± 4	275	0.45 ± 0.03	(1.1 ± 0.5) × 10^{-5}	40	38 ± 2	−122 ± 4
	293		2.46 ± 0.09	(4.0 ± 0.2) × 10^{-5}					0.51 ± 0.06	(2.3 ± 0.2) × 10^{-5}			
	298		3.26 ± 0.10	(5.0 ± 0.2) × 10^{-5}					0.64 ± 0.02	(2.5 ± 0.4) × 10^{-5}			
	303		4.46 ± 0.10	(8.0 ± 0.2) × 10^{-5}					0.98 ± 0.05	(3.0 ± 0.1) × 10^{-5}			
	308		7.94 ± 0.09	(10.0 ± 0.4) × 10^{-5}					1.28 ± 0.1	(5.0 ± 0.2) × 10^{-5}			
hex-5-en-1-ol	288	259	1.0 ± 0.1	(3.46 ± 0.2) × 10^{-3}	56	54 ± 4	−58 ± 2	259	0.76 ± 0.06	(4.2 ± 0.1) × 10^{-3}	44	41 ± 2	−104 ± 3
	293		1.34 ± 0.2	(4.49 ± 0.3) × 10^{-3}					0.86 ± 0.09	(5.2 ± 0.1) × 10^{-3}			
	298		1.76 ± 0.2	(5.96 ± 0.4) × 10^{-3}					0.90 ± 0.04	(6.9 ± 0.1) × 10^{-3}			
	303		3.12 ± 0.1	(6.50 ± 0.2) × 10^{-3}					1.92 ± 0.2	(8.9 ± 0.4) × 10^{-3}			
	308		4.46 ± 0.2	(7.9 ± 0.2) × 10^{-3}					2.28 ± 0.1	(11.3 ± 0.2) × 10^{-3}			
hex-5-en-1-ol and pyridine	288	260	6.02 ± 0.3	(0.2 ± 0.06) × 10^{-4}	37	35 ± 3	−108 ± 4	260	3.64 ± 0.20	(2.0 ± 0.3) × 10^{-5}	40	38 ± 4	−102 ± 3
	293		9.62 ± 0.5	(0.5 ± 0.08) × 10^{-4}					4.68 ± 0.08	(4.0 ± 0.2) × 10^{-5}			
	298		12.1 ± 0.5	(0.7 ± 0.09) × 10^{-4}					5.64 ± 0.06	(6.0 ± 0.1) × 10^{-5}			
	303		14.2 ± 0.5	(0.9 ± 0.07) × 10^{-4}					8.10 ± 0.20	(8.0 ± 0.3) × 10^{-5}			
	308		17.4 ± 0.3	(1.0 ± 0.2) × 10^{-4}					10.9 ± 0.02	(10 ± 3) × 10^{-5}			

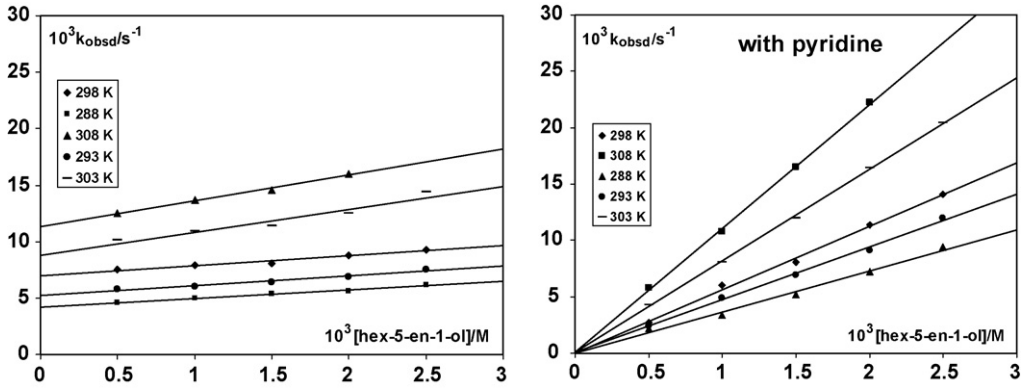


Fig. 5. *Pseudo*-first order rate constants, k_{obsd}, as a function of concentration of alcohol and temperature for the reactions between PhSeBr and hex-5-en-1-ol in CCl_4 in the presence and absence of pyridine.

The catalytic function of pyridine has also been described by the second order rate constants, presented in Table 1. The reactions with pyridine are faster. The catalytic effect is slightly different depending on the type of alcohol and phenylselenenyl halides that are used. Taking into account the results from Table 1 at 298 K pyridine increases the rate of reaction of pent-4-en-1-ol about 2.5 times (with PhSeCl and PhSeBr) and in the case of hex-5-en-1-ol the increase is about 6.7 times with PhSeCl and 6.3 with PhSeBr.

From Figs. 2–5 some differences between the reactions with and without pyridine are remarkable. All lines in the reactions with pyridine start almost from the origin of the graph. This means that these reactions have no reverse or parallel runs, which is in agreement with the synthetically obtained yields (≈100%) for cyclization products in the reactions with pyridine as the catalyst (Fig. 1).

According to Eyring equation (Eq. (3)) the enthalpy and entropy of activation were determined.

$$\ln\left(\frac{k_2}{T}\right) = -\frac{\Delta H^{\neq}}{RT} + \left[\ln\left(\frac{k_t}{h}\right) + \frac{\Delta S^{\neq}}{R}\right] \quad (3)$$

The negative values for entropy of activation indicate that the reactions of PhSeX (X = Cl, Br) with pent-4-en-1-ol and hex-5-en-1-ol in the presence and in the absence of pyridine follow the mechanism of bimolecular nucleophile substitution S_N2 pathway.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molcata.2008.03.014.

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