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Regio- and Stereoselective Synthesis of Functionalized Vinyl Sulfides Based on Pyridine-2-thiol and Propynoic Acid and Its Derivatives

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Abstract—Regio- and stereoselective methods of synthesis of 3-(pyridin-2-ylsulfanyl)prop-2-enoic acid and alkyl (*Z*)-3-(pyridin-2-ylsulfanyl)prop-2-enoates have been developed on the basis of nucleophilic addition of pyridine-2-thiol to propynoic acid and alkyl propynoates. Reactions of alkyl (*Z*)-3-(pyridin-2-ylsulfanyl)prop-2-enoates with methyl iodide afforded 2-[(3-alkoxy-3-oxoprop-1-enyl)sulfanyl]-1-methylpyridinium iodides.

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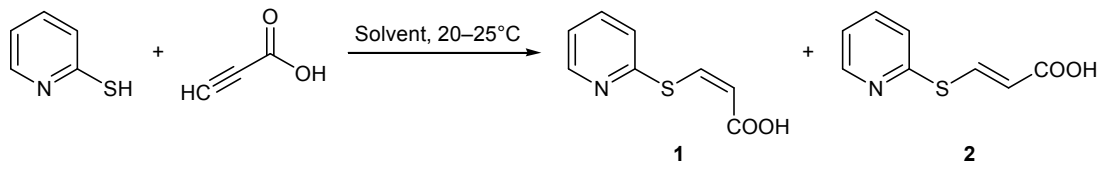
Pyridine ring is considered a pharmacohporic group constituting a structural fragment of many medicinal agents [1, 2]. Pyridine derivatives with sulfur-containing substituents were found to exhibit antiviral, antitumor, anti-inflammatory, and anti-HIV activities [3–6], whereas some pyridinium salts showed a high antimicrobial activity [7, 8].

Vinyl chalcogenides are widely used as building blocks and intermediate products in modern organic synthesis [9–13]. They are generally prepared by nucleophilic addition of organylchalcogenolate or chalcogenide anions to alkynes [9–18]. Of particular importance are vinyl sulfides capable of being involved in radical addition, polymerization, and copolymerization reactions [10–13]. The addition of pyridine-2-thiol to acetylene, phenylacetylene, and diacetylene was reported to produce the corresponding 2-(ethenylsulfanyl)pyridine derivatives [12]. The synthesis of new potentially biologically active vinyl sulfides containing pharmacophoric groups seems to be important.

We previously developed efficient methods for the synthesis of vinyl chalcogenides by nucleophilic addition of chalcogen-centered anions to alkynes [13–18]. In this work we studied how the reaction conditions (solvent, temperature, base) and substituent at the triple bond affect the yield and product ratio in the reactions of pyridine-2-thiol with propynoic acid and alkyl proynoates with the goal of developing efficient procedures for the preparation of functionalized vinyl sulfides that are potential biologically active compounds.

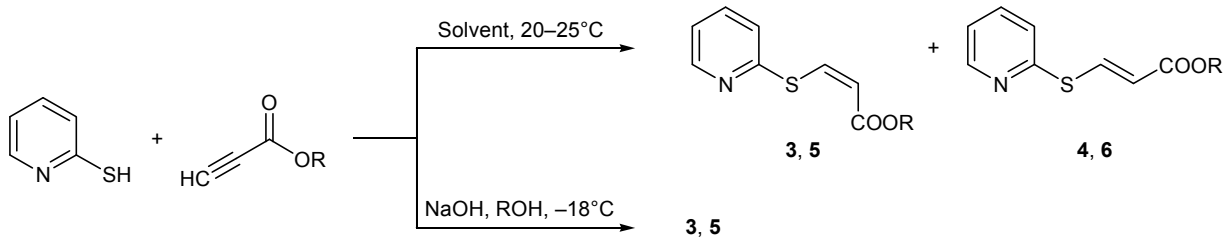
The reaction of pyridine-2-thiol with propynoic acid at room temperature in methylene chloride or acetonitrile quantitatively afforded 3-(pyridin-2-ylsulfanyl)prop-2-enoic acid as a mixture of *Z* and *E* isomers **1** and **2** at a ratio of ~1:3 (CH_2Cl_2) or ~5:6 (MeCN); i.e., in both solvents, *E* isomer **2** was formed

Scheme 1.



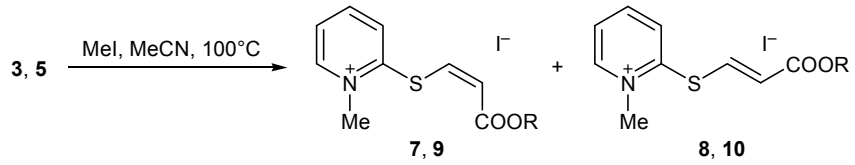
Solvent: CH_2Cl_2, MeCN, MeOH.

Scheme 2.



3, **4**, R = Me; **5**, **6**, R = Et; solvent: CH_2Cl_2, MeCN, MeOH.

Scheme 3.



3, **7**, **8**, R = Me; **5**, **9**, **10**, R = Et.

as the major product (Scheme 1). When the reaction was carried out in methanol, the major product was *Z* isomer **1** (ratio **1**/**2** ~4:3).

Likewise, pyridine-2-thiol reacted with methyl and ethyl propynoate at room temperature in methylene chloride, acetonitrile, methanol, or ethanol to give the corresponding alkyl 3-(pyridin-2-ylsulfanyl)prop-2-enoates **3**–**6** in quantitative yield; the products were mixtures of *Z* and *E* isomers, the former prevailing (Scheme 2). For example, the *Z*/*E*-isomer ratio **3**/**4** was 2:1 in methylene chloride and 5:2 in acetonitrile.

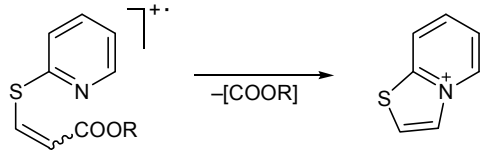
Thus, the reactions of pyridine-2-thiol with alkyl propynoates in the absence of a catalyst are regioselective but not stereoselective. The reaction of pyridine-2-thiol with propynoic acid in methylene chloride and acetonitrile gives preferentially *E* isomer **2** (Scheme 1), whereas its reactions with alkyl propynoates in the same solvents lead to the formation of *Z* isomers **3** and **5** as the major products (Scheme 2).

In order to improve the stereoselectivity of the addition of pyridine-2-thiol to alkyl propynoates, the reactions were carried out on cooling to –15 to –18°C in the presence of a catalytic amount of sodium hydroxide (NaOH, 5 mol %) in methanol (with methyl propynoate) and ethanol (with ethyl propynoate); the reaction mixtures were then stirred at room temperature. As a result, we isolated in high yields (97–98%) the corresponding *Z*-isomeric esters **3** and **5** (Scheme 2).

We also tried to obtain methylpyridinium salts as water-soluble derivatives with potential biological activity. For this purpose, compounds **3** and **5** were reacted with methyl iodide. The formation of pyridinium salts was observed when sulfides **3** and **5** were heated methyl iodide in boiling methanol or acetonitrile; however, under these conditions, the conversion was fairly low, so that it was difficult to isolate the target products. We succeeded in attaining the complete conversion by heating the reactants in a sealed ampule at 100°C (Scheme 3). In this case, 2-[(3-alkoxy-3-oxoprop-1-en-1-yl)sulfanyl]-1-methylpyridinium iodides were isolated in quantitative yield as mixtures of *Z* and *E* isomers; the ratio **7**/**8** was ~5:1, and **9**/**10**, ~4:1. The reactions of propynoic acid derivatives **1** and **2** with methyl iodide under similar conditions led to the formation of mixtures of products which were difficult to separate.

The structure of the isolated compounds was proved by ^{1}H and ^{13}C NMR and mass spectra and elemental analyses. The mass spectra of **1**–**6** contained the molecular ion peaks, but the most abundant was $[PySC_2H_2]^+$ ion with *m*/*z* 136. Analysis of the MS data showed that the main fragmentation pathway of compounds **1**–**6** under electron impact is elimination of the

Scheme 4.



m/*z* 136 (I_{rel} 100%)

1, **2**, R = H; **3**, **4**, R = Me; **5**, **6**, R = Et.

COOH or COOAlk group to form stable [1,3]thiazolo-[3,2-*a*]pyridin-4-ium cation **A** (Scheme 4).

In summary, by studying the effects of the conditions and substituent at the triple bond on the yield and ratio of products of nucleophilic addition of pyridine-2-thiol to propynoic acid and alkyl propynoates, we have developed regio- and stereoselective methods of synthesis of compounds **1**–**10** in up to quantitative yield. The obtained compounds are new promising intermediate products for organic synthesis which also attract interest as potential biologically active substances. The presence of reactive groups (vinylsulfanyl, ester, carboxy, nucleophilic nitrogen atom) in their molecules provides the possibility of their further functionalization and synthesis of new derivatives.

EXPERIMENTAL

The NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400.13 (^{1}H) and 100.61 MHz (^{13}C) using $CDCl_3$ as solvent and hexamethyldisiloxane as reference. The mass spectra (electron impact, 70 eV) were recorded on a Shimadzu GCMS-QP5050A instrument with direct sample admission into the ion source. The elemental analyses were obtained with a Thermo Scientific Flash 2000 CHNS analyzer. The melting points were measured on a Boetius PHMK 05 melting point apparatus (Wagetechnik Rapido). The solvents used were preliminarily dried and distilled.

(*Z*)- and (*E*)-3-(Pyridin-2-ylsulfanyl)prop-2-enoic acids (1/2). A solution of 0.111 g (1 mmol) of pyridine-2-thiol in 2 mL of methylene chloride was added dropwise with stirring to a solution of 0.07 g (1 mmol) of propynoic acid in 2 mL of methylene chloride. The mixture was stirred for 6 h at room temperature, the solvent was distilled off on a rotary evaporator, and the residue was dried under reduced pressure. Yield 0.181 g (quantitative), white powder, mp 33–34°C (from $CHCl_3$); a mixture of *Z* and *E* isomers at a ratio of 1:3. Mass spectrum, *m*/*z* (I_{rel}, %): 181 (20) $[M]^+$, 137 (26) $[PySC_2H_3]^+$, 136 (100) $[PySC_2H_2]^+$, 111 (16) $[PySH]^+$, 79 (67) $[C_5H_5N]^+$, 78 (75) $[C_5H_4N]^+$, 67 (25) $[C_4H_5N]^+$, 51 (53) $[C_4H_3]^+$, 39 (39) $[C_3H_3]^+$. Found, %: C 52.73; H 4.07; N 7.48; S 18.01. $C_8H_7NO_2S$. Calculated, %: C 53.02; H 3.89; N 7.73; S 17.70.

Z Isomer (**1**). ^{1}H NMR spectrum, δ, ppm: 5.87 d (1H, =CHC=O, ^3J = 10.2 Hz), 6.89–6.94 m (1H, Py), 7.08–7.12 m (1H, Py), 7.36–7.40 m (1H, Py), 8.28–8.32 m (1H, Py), 8.26 d (1H, =CHS, ^3J = 10.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 114.20 (=CH), 121.09 (Py), 122.90 (Py), 136.66 (Py), 141.45 (=CHS), 149.12 (Py), 154.92 (NCS), 168.13 (C=O).

E Isomer (**2**). ^{1}H NMR spectrum, δ, ppm: 5.88 d (1H, =CHC=O, ^3J = 15.9 Hz), 6.92–6.97 m (1H, Py), 7.04–7.08 m (1H, Py), 7.40–7.44 (1H, Py), 8.29–8.33 (1H, Py), 8.36 d (1H, =CHS, ^3J = 15.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 117.40 (=CH), 121.28 (Py), 122.85 (Py), 136.81 (Py), 141.66 (=CHS), 149.28 (Py), 153.68 (NCS), 166.62 (C=O).

Methyl (*Z*)-3-(pyridin-2-ylsulfanyl)prop-2-enoate (3). A solution of 0.17 g (2 mmol) of methyl propynoate in 2 mL of methanol was added to a solution of 0.222 g (2 mmol) of pyridine-2-thiol and 0.005 g (0.1 mmol) of 85% NaOH in 8 mL of methanol cooled to –15 to –18°C. The mixture was left to stand for 16 h in a refrigerator (–18°C) and stirred for 6 h at room temperature, 0.006 g (0.11 mmol) of ammonium chloride was added, and the mixture was stirred for 1 h more. The solvent was distilled off on a rotary evaporator, the residue was washed with chloroform (10 mL), and the solution was filtered. The filtrate was evaporated on a rotary evaporator, and the residue was dried under reduced pressure. Yield 0.38 g (97%), white powder, mp 53–54°C (from MeOH). ^{1}H NMR spectrum, δ, ppm: 3.76 s (3H, OCH_3), 6.08 d (1H, =CHC=O, ^3J = 10.2 Hz), 7.06–7.12 m (1H, Py), 7.28–7.32 m (1H, Py), 7.52–7.59 m (1H, Py), 8.47–8.50 m (1H, Py), 8.56 d (1H, =CHS, ^3J = 10.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 51.14 (CH_3), 113.07 (=CH), 121.12 (Py), 122.88 (Py), 136.63 (Py), 141.95 (=CHS), 149.32 (Py), 154.68 (NCS), 166.73 (C=O). Found, %: C 55.63; H 4.82; N 7.36; S 16.13. $C_9H_9NO_2S$. Calculated, %: C 55.37; H 4.65; N 7.17; S 16.42.

Methyl (*E*)-3-(pyridin-2-ylsulfanyl)prop-2-enoate (4). A solution of 0.17 g (2 mmol) of methyl propynoate in 2 mL of methylene chloride was added to a solution of 0.22 g (2 mmol) of pyridine-2-thiol in 8 mL of methylene chloride. The mixture was stirred for 20 h at room temperature, the solvent was distilled off, and the residue was dried under reduced pressure. Yield 0.39 g (quantitative), colorless viscous oily material, a mixture of *Z* and *E* isomers (ratio **3**/**4** ~2:1). Mass spectrum, *m*/*z* (I_{rel}, %): 195 (3) $[M]^+$, 164 (4) $[M - C_2H_5O]^+$, 137 (6) $[PySC_2H_3]^+$, 136 (100) $[PySC_2H_2]^+$, 111 (4) $[PySH]^+$, 79 (11) $[C_5H_5N]^+$, 78 (20) $[C_5H_4N]^+$, 51 (12) $[C_4H_3]^+$, 39 (9) $[C_3H_3]^+$. Found, %: C 55.09; H 4.47; N 6.98; S 16.17. $C_9H_9NO_2S$. Calculated, %: C 55.37; H 4.65; N 7.17; S 16.42.

E Isomer (**4**). ^{1}H NMR spectrum, δ, ppm: 3.73 s (3H, OCH_3), 6.12 d (1H, =CHC=O, ^3J = 15.9 Hz),

7.09–7.14 m (1H, Py), 7.21–7.25 m (1H, Py), 7.55–7.60 m (1H, Py), 8.41–8.44 m (1H, Py), 8.64 d (1H, =CHS, ^3J = 15.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 51.26 (CH_3), 116.31 (=CH), 121.25 (Py), 122.77 (Py), 136.73 (Py), 141.90 (=CHS), 149.74 (Py), 153.60 (NCS), 165.16 (C=O).

Ethyl (*Z*)-3-(pyridin-2-ylsulfanyl)prop-2-enoate (5) was synthesized as described above for compound **3** using ethanol as solvent. Yield 98%, white powder, mp 61–62°C (from EtOH). ^{1}H NMR spectrum, δ, ppm: 1.31 t (3H, CH_3, ^3J = 7.1 Hz), 4.24 q (2H, CH_2, ^3J = 7.1 Hz), 6.08 d (1H, =CHC=O, ^3J = 10.3 Hz), 7.08–7.13 m (1H, Py), 7.28–7.32 m (1H, Py), 7.56–7.61 m (1H, Py), 8.47–8.51 m (1H, Py), 8.51 d (1H, =CHS, ^3J = 10.3 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.21 (CH_3), 60.28 (CH_2), 113.77 (=CH), 121.19 (Py), 123.13 (Py), 136.71 (Py), 141.78 (=CHS), 149.50 (Py), 155.13 (NCS), 166.68 (C=O). Found, %: C 56.63; H 5.11; N 6.92; S 15.61. $C_{10}H_{11}NO_2S$. Calculated, %: C 57.39; H 5.30; N 6.69; S 15.32.

Ethyl (*E*)-3-(pyridin-2-ylsulfanyl)prop-2-enoate (6) was synthesized as described above for compound **4**. Colorless viscous oily material; mixture of *Z* and *E* isomers, ratio **5**/**6** 2:1 (quantitative overall yield). Mass spectrum, *m*/*z* (I_{rel}, %): 209 (3) $[M]^+$, 164 (4) $[M - C_2H_5O]^+$, 137 (6) $[PySC_2H_3]^+$, 136 (100) $[PySC_2H_2]^+$, 111 (4) $[PySH]^+$, 79 (12) $[C_5H_5N]^+$, 78 (21) $[C_5H_4N]^+$, 51 (9) $[C_4H_3]^+$, 39 (7) $[C_3H_3]^+$. Found, %: C 56.11; H 5.13; N 6.48; S 15.59. $C_{10}H_{11}NO_2S$. Calculated, %: C 57.39; H 5.30; N 6.69; S 15.32.

E Isomer (**6**). ^{1}H NMR spectrum, δ, ppm: 1.30 t (3H, CH_3, ^3J = 7.1 Hz), 4.23 q (2H, CH_2, ^3J = 7.1 Hz), 6.12 d (1H, =CHC=O, ^3J = 15.9 Hz), 7.08–7.12 m (1H, Py), 7.26–7.30 m (1H, Py), 7.54–7.59 m (1H, Py), 7.47–8.51 m (1H, Py), 8.54 d (1H, =CHS, ^3J = 15.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 13.80 (CH_3), 62.20 (CH_2), 117.18 (=CH), 121.37 (Py), 123.04 (Py), 136.81 (Py), 141.71 (=CHS), 149.98 (Py), 154.11 (NCS), 165.01 (C=O).

(*Z*)- and (*E*)-2-[(3-Methoxy-3-oxoprop-1-en-1-yl)sulfanyl]-1-methylpyridinium iodides (7/8). A mixture of 0.195 g (1 mmol) of compound **3**, 0.43 g (3 mmol) of methyl iodide, and 1 mL of acetonitrile was heated for 4 h at 100°C in a sealed ampule. The solvent and excess methyl iodide were distilled off ion a rotary evaporator, and the residue was dried under reduced pressure. Yield 0.337 g (quantitative), dark yellow viscous oily material, a mixture of *Z* and *E* isomers at a ratio of 5:1. Found, %: C 35.34; H 3.78; N 3.98; S 9.23. $C_{10}H_{12}INO_2S$. Calculated, %: C 35.62; H 3.59; N 4.15; S 9.51.

Z Isomer (**7**). ^{1}H NMR spectrum, δ, ppm: 3.74 s (3H, OCH_3), 4.47 s (3H, NCH_3), 6.35 d (1H, =CHC=O, ^3J = 9.7 Hz), 7.75 d (1H, =CHS, ^3J = 9.7 Hz), 8.02–8.06 m (1H, Py), 8.33–8.37 m (1H, Py), 8.55–8.60 m (1H, Py), 9.35–9.39 m (1H, Py). ^{13}C NMR spectrum, δ_C, ppm: 47.50 (NCH_3), 51.69 (OCH_3), 119.59 (=CH), 125.31 (Py), 129.59 (Py), 137.55, 144.73, 147.37 (Py, =CHS), 156.06 (NCS), 165.47 (C=O).

E Isomer (**8**). ^{1}H NMR spectrum, δ, ppm: 3.72 s (3H, OCH_3), 4.45 s (3H, NCH_3), 6.44 d (1H, =CHC=O, ^3J = 15.2 Hz), 7.68 d (1H, =CHS, ^3J = 15.2 Hz), 7.96–8.03 m (2H, Py), 8.50–8.54 m (1H, Py), 9.40–9.44 m (1H, Py). ^{13}C NMR spectrum, δ, ppm: 47.26 (NCH_3), 51.83 (OCH_3), 121.60 (=CH), 124.85 (Py), 128.04 (Py), 138.12, 144.38, 147.48 (Py, =CHS), 154.46 (NCS), 163.02 (C=O).

(*Z*)- and (*E*)-2-[(3-Ethoxy-3-oxo-prop-1-en-1-yl)-sulfanyl]-1-methylpyridinium iodides (9/10) were synthesized in a similar way. Yield quantitative, dark yellow viscous oilu material, a mixture of *Z* and *E* isomers at a ratio of 4:1. Found, %: C 37.33; H 3.82; N 4.16; S 8.92. $C_{11}H_{14}INO_2S$. Calculated, %: C 37.62; H 4.02; N 3.99; S 9.13.

Z Isomer (**9**). ^{1}H NMR spectrum, δ, ppm: 1.21 t (3H, CH_3, ^3J = 7.1 Hz), 4.12 q (2H, OCH_2, ^3J = 7.1 Hz), 4.42 s (3H, NCH_3), 6.27 d (1H, =CHC=O, ^3J = 9.7 Hz), 6.59 d (1H, =CHS, ^3J = 9.7 Hz), 7.97–8.01 m (1H, Py), 8.28–8.33 m (1H, Py), 8.45–8.50 m (1H, Py), 9.30–9.35 m (1H, Py). ^{13}C NMR spectrum, δ_C, ppm: 13.97 (CH_3), 48.15 (NCH_3), 61.09 (CH_2), 120.01 (=CH), 125.96 (Py), 130.47 (Py), 138.32, 145.35, 147.78 (Py, =CHS), 155.99 (NCS), 165.61 (C=O).

E Isomer (**10**). ^{1}H NMR spectrum, δ, ppm: 1.19 t (3H, CH_3, ^3J = 7.1 Hz), 4.15 q (2H, OCH_2, ^3J = 7.1 Hz), 4.40 s (3H, NCH_3), 6.36 d (1H, =CHC=O, ^3J = 15.2 Hz), 6.69 d (1H, =CHS, ^3J = 15.2 Hz), 7.90–7.97 m (2H, Py), 8.50–8.55 m (1H, Py), 9.35–9.40 m (1H, Py). ^{13}C NMR spectrum, δ_C, ppm: 13.88 (CH_3), 48.02 (NCH_3), 61.29 (CH_2), 120.11 (=CH), 125.58 (Py), 130.40 (Py), 138.12, 145.15, 147.97 (Py, =CHS), 154.61 (NCS), 163.03 (C=O).

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