

## Vinyl Ethers with Isothiocyanate Group: XIX. Reaction with Dicarboxylic Acids: a Simple Route to *N,N'*-Bis[(2-vinyloxy)ethyl]amides

S. A. Nedolya, S. V. Tolmachev, V. P. Zinov'yeva, and G. I. Sarapulova

Faworsky Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences, Irkutsk, 664033, Russia

Received June 14, 2001

**Abstract**—Reactions of 2-(vinyloxy)ethyl isothiocyanate with dicarboxylic acids (malonic, succinic, glutaric, adipic, pimelic, sebacic acids) under nucleophilic conditions (10–15 wt% of Et<sub>3</sub>N, 55–80°C, 5–50 min) yield quantitatively *N,N'*-bis[(2-vinyloxy)ethyl]amides.

A catalyzed by bases reaction of isothiocyanates with carboxylic acids was described among methods of preparation of *N*-substituted amides [2–6]. The process usually requires relatively high temperature (160–170°C, 15–40 min). The unique significance of amide function both in nature and chemical science is well known [7, 8], and from this viewpoint the development of convenient synthesis of new amide representatives, especially functionally substituted, is important.

2-(Vinyloxy)ethyl isothiocyanate (**I**) as bifunctional monomer and reagent extensively applied to structurally selective modification of different compounds [9, 10], including simultaneous synthesis and functionalization of pyrroles and 2,3-dihydropyridines [11–15], is an appropriate starting compound for the preparation of a new family of functional amides via carboxylic acids. We were the first to study reactions between 2-(vinyloxy)ethyl isothiocyanate and monocarboxylic acids of various structures [9, 10, 16–20]. It was found that in the presence of 10–15 wt% of Et<sub>3</sub>N acetic, propionic, 2-methylpropanoic, 3-methylbutanoic, pentanoic, octanoic, and 2-(3-indolyl)ethanoic acids cleanly and as a rule quantitatively added across the N=C bond affording previously unknown *N*-[(2-vinyloxy)ethyl]amides of the respective acids [19, 20]. The reaction was carried out predominantly without solvent at stoichiometric reagents ratio and 40–90°C within 0.5–4 h. Yet the halocarboxylic acids (2-chloro-, 2-bromo-, trifluoroacetic acids) under the same conditions unexpectedly added exclusively to the vinyloxy group of isothiocyanate **I** providing 1-(2-isothiocyanatoethoxy)ethyl

2-haloacetates [19, 20]. The same products were also obtained under electrophilic conditions, which was more natural [16–18] in keeping with the chemical character of the vinyloxy group. Here, the sufficiently strong halocarboxylic acids obviously completely bind the amine catalyst into stable salts and, thus, remove it from the active zone of the reaction. The increase in Et<sub>3</sub>N concentration (up to equimolar) usually resulted only in strong tarring of the reaction mixture but did not affect the direction of the reaction.

The reactions of isothiocyanates with dicarboxylic acids apparently are not described.

In the present study aiming at preparation of previously unavailable divinyl ethers of diamidodiols, promising polyfunctional monomers, synthons, and modifiers of polymeric materials, and also hoping to acquire new data on the reactivity and synthetic potential of 2-(vinyloxy)ethyl isothiocyanate we investigated its reactions with a number of dicarboxylic acids (malonic, succinic, glutaric, adipic, pimelic, sebacic, maleic, and phthalic) under nucleophilic conditions. Same as with monocarboxylic acids, the result cannot be predicted beforehand, for the process may be complicated by a concurrent (or selective) addition of the acid to vinyloxy group and by transformation of the arising amide into 2-methyl-1,3-oxazolanone (via intramolecular addition of the NH group to vinyloxy group) [9, 19, 20].

It was established that, in the presence of catalytic amounts of triethylamine (10–15 wt%), 2-(vinyloxy)ethyl isothiocyanate as expected reacted with alkanedicarboxylic acids with sufficiently high regioselectivity affording in virtually quantitative yield

\* For communication XVIII, see [1].

**Table 1.** Reaction conditions of 2-(vinyloxy)ethyl isothiocyanate **I** with dicarboxylic acids, R(COOH)<sub>2</sub> [molar ratio isothiocyanate **I**:R(COOH)<sub>2</sub> 2:1, 0.01–0.02 mol]<sup>a</sup>

Run no.	R	Amount of Et <sub>3</sub> N, wt%	Temperature, °C <sup>b</sup>	Time, h	no. of bis-amide	Yield bis-amide, % <sup>c</sup>
1	CH <sub>2</sub>	15	55–60 (32)	5	<b>III</b>	~40–50 <sup>d</sup>
2 <sup>e</sup>	CH <sub>2</sub>	15	80–85	25	<b>III</b>	~50–60 <sup>d</sup>
3 <sup>f</sup>	CH <sub>2</sub>	15	90–143	13	<b>III</b>	~100 <sup>d,g</sup>
4	CH <sub>2</sub>	15	100–120 (32)	4	<b>III</b>	~100 <sup>d,g,h</sup>
5 <sup>i</sup>	CH <sub>2</sub>	15	~110 (34)	~5–7 min	<b>III</b>	~100 <sup>j</sup>
6 <sup>k</sup>	(CH <sub>2</sub> ) <sub>2</sub>	10	70–80 (27)	4	<b>IV</b>	~50–60 <sup>d,l</sup>
7 <sup>k</sup>	(CH <sub>2</sub> ) <sub>2</sub>	15	70–80 (29)	~50 min	<b>IV</b>	47.3 (~100) <sup>j</sup>
8	(CH <sub>2</sub> ) <sub>3</sub>	15	~60 (31)	~5–7 min <sup>m</sup>	<b>V</b>	56.7 (~100) <sup>j</sup>
9	(CH <sub>2</sub> ) <sub>4</sub>	15	55–60 (28)	~10 min	<b>VI</b>	84.9 (~100) <sup>j</sup>
10	(CH <sub>2</sub> ) <sub>4</sub>	10	55–60	1	<b>VI</b>	63.3 (~100) <sup>j</sup>
11	(CH <sub>2</sub> ) <sub>4</sub>	5	75–77 (22.5)	~15 min <sup>m</sup>	<b>VI</b>	71.1 (~100) <sup>j</sup>
12 <sup>k</sup>	(CH <sub>2</sub> ) <sub>4</sub>	1	55–60 <sup>n</sup>	6	<b>VI</b>	~50 <sup>d,l,o</sup>
13	(CH <sub>2</sub> ) <sub>5</sub>	15	~60 (26)	~3–5 min <sup>m</sup>	<b>VII</b>	67.1 (~100) <sup>j</sup>
14	(CH <sub>2</sub> ) <sub>8</sub>	10	45–60 (22.5)	~5–7 min <sup>m</sup>	<b>VIII</b>	57.7 (~100) <sup>j</sup>
15	CH=CH	15	55–60 (39)	5	<b>IX</b>	~40–50 <sup>d,o</sup>
16 <sup>e</sup>	CH=CH	15	80–85	25	<b>IX</b>	~100 <sup>d,p</sup>
17 <sup>f</sup>	CH=CH	15	138–143	3	<b>IX</b>	~100 <sup>d,g</sup>
18	CH=CH	15	~100 (36) <sup>q</sup>	~3–5 min	<b>IX</b>	~100 <sup>d,p</sup>
19	CH=CH	15	85–90	~3–5 min	<b>IX</b>	~100 <sup>d,p,r</sup>
20	<i>n</i> -C <sub>6</sub> H <sub>4</sub>	10	55–60 (22) <sup>s</sup>	1		
			70–80	3		
			~80 <sup>t</sup>	3	<b>X</b>	~85–90 <sup>d,l,o,u</sup>
21 <sup>e</sup>	<i>n</i> -C <sub>6</sub> H <sub>4</sub>	15	70–80 (31)	13	<b>X</b>	~90–95 <sup>d,l,o,v</sup>
22 <sup>i,k</sup>	<i>n</i> -C <sub>6</sub> H <sub>4</sub>	15	95–105 (37)	2	<b>X</b>	<sup>w</sup>

<sup>a</sup> The acid was added to a solution of catalyst in isothiocyanate **I**.

<sup>b</sup> The temperature of self-heating after reagents and catalyst mixing at 21°C is given in parentheses.

<sup>c</sup> Preparative yield with respect to reagents amount taken for the process.

<sup>d</sup> Conversion of isothiocyanate **I** estimated from the IR spectra of reaction mixture [from relative intensity of the band at 2100–2200 cm<sup>-1</sup> (N=C=S)].

<sup>e</sup> 5 ml of benzene.

<sup>f</sup> 5 ml of xylene.

<sup>g</sup> IR spectrum: weak bands of NH and CH<sub>2</sub>=CHO, band of N=C=S is lacking.

<sup>h</sup> Sulfur content ~7.5%.

<sup>i</sup> Molar ratio acid:isothiocyanate 1:3.

<sup>j</sup> Yield of crude product.

<sup>k</sup> The catalyst was added to a mixture of acid and isothiocyanate **I**.

<sup>l</sup> Concurrent reaction at vinyloxy group (IR spectrum).

<sup>m</sup> The reaction mixture was additionally kept for 45–60 min at room temperature.

<sup>n</sup> The reaction mixture was heated to 98–100°C, then the temperature was decreased to 55–60°C within ~10 min.

<sup>o</sup> Tarring of the reaction mixture.

<sup>p</sup> Polymer.

<sup>q</sup> Self-heating from 100 to 195°C.

<sup>r</sup> Sulfur content ~14%.

<sup>s</sup> Heating to ~85°C, then the temperature was decreased to 55–60°C within ~10 min.

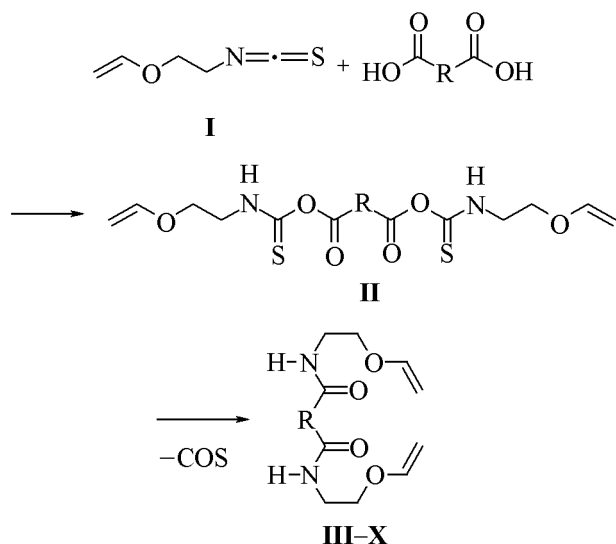
<sup>t</sup> Benzene (8 ml) was added and heating to boiling was continued for 3 h more.

<sup>u</sup> Sulfur content 5.8%.

<sup>v</sup> In <sup>1</sup>MNMR spectrum signals of amide **X** were registered.

<sup>w</sup> Strongly tarred crystals.

previously unknown *N,N'*-bis[(2-vinyloxy)ethyl]-amides **III–VIII**.



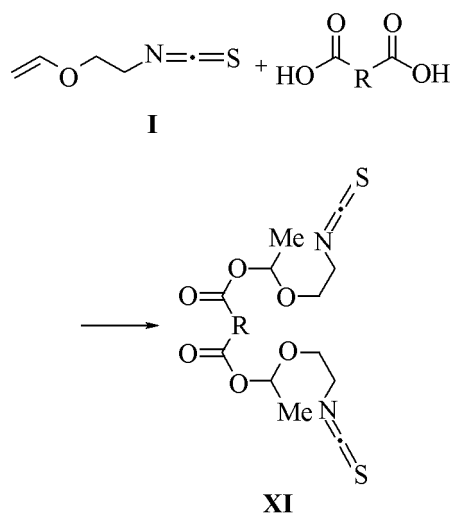
R = (CH<sub>2</sub>)<sub>n</sub>, n = 1 (**III**), 2 (**IV**), 3 (**V**), 4 (**VI**), 5 (**VII**), 8 (**VIII**); CH=CH (**IX**); *o*-C<sub>6</sub>H<sub>4</sub> (**X**).

The reaction progress was monitored by IR spectra of the reaction mixtures: The intensity decrease in absorption bands of N=C=S at 2199–2299 cm<sup>-1</sup> was followed till complete disappearance at retention of the main absorption bands of vinyloxy group at 820–840, 960, 1200, 1320, 1620–1640 cm<sup>-1</sup>.

The reaction conditions and amides yield are summarized in Table 1. The data presented in Table 1 show that same as with the monocarboxylic acids [19, 20] the process in the majority of cases occurs under relatively mild conditions: without solvent, at short heating (for several minutes, mostly to 55–60°C) of stoichiometric amounts of reagents. The formation of an intermediate **II** is accompanied by simultaneous ready and as a rule complete elimination of carbonyl sulfide as indicates gas evolution in the course of the reaction, elemental analysis (lack of sulfur), and IR spectra (disappearance of usual strong C=O absorption band at ~1700 cm<sup>-1</sup> [19]) of reaction products. Only bis-amide **III** even after purification contained ~2% of sulfur (Table 2). The qualitative indication of the completion of reaction was the stopped gas evolution.

The effect of catalyst amount on the duration of the process and on bis-amide yield was investigated by an example of reaction between isothiocyanate **I** with adipic acid (Table 1, runs nos. 9–12). On decreasing the Et<sub>3</sub>N concentration from 15 to 1 wt% the time of

reaction grew from ~10 min to 6 h, and a trend to the alternative reaction path, addition of the acid to vinyloxy group to furnish bis-acetal-acylal (**XI**) [R = (CH<sub>2</sub>)<sub>4</sub>] was more pronounced [1]. The concurrent reaction at vinyloxy group occurred also with succinic and phthalic acids in the presence of 10 wt% of Et<sub>3</sub>N (Table 1, runs nos. 6 and 20). Since the carboxylic acids are known [21] to react with vinyl ethers at heating also without acid catalyst, the decreasing of triethylamine concentration is obviously unfeasible. Besides at low concentrations of triethylamine the elimination of carbonyl sulfide is retarded, and therefore the target products contain impurities of intermediate **II** or its derivatives.



The result of reaction is also somewhat affected by the order of reagents and catalyst addition. To suppress the unwanted reaction at the vinyloxy group it is feasible to add the acid to the solution of the catalyst in isothiocyanate. No advantages provided the addition of solvents (benzene, xylene), and as a rule it decelerated the process. (Table 1, runs nos. 2, 3, 16, 21).

In the presence of 10–15 wt% of Et<sub>3</sub>N glutaric, adipic, pimelic, and sebacic acids cleanly and quantitatively add to isothiocyanate **I** at 45–60°C within ~5–10 min (Table 1, runs nos. 8, 9, 13, 14) yielding the corresponding amides. To attain better results with succinic acid a higher temperature and longer reaction time was required (Table 1, run no. 7). Even stronger among the acids under study was malonic acid, and the process therewith succeeded only at 50% excess of isothiocyanate **I** and ~110°C (Table 1, run no. 5). At stoichiometric reagents ratio the reaction with malonic acid at 55–60°C proceeded very

**Table 2.** Melting points and elemental analyses of *N,N'*-bis[(2-vinyloxy)ethyl]amides **III–VIII**, **X**

Compd. no.	mp, °C	Found, %			Formula	Calculated, %		
		C	H	N		C	H	N
<b>III</b> <sup>a</sup>	~115	52.99	7.23	11.49	C <sub>11</sub> H <sub>18</sub> N <sub>2</sub> O <sub>6</sub>	54.53	7.49	11.56
<b>IV</b>	111–113	54.68	9.49	10.99	C <sub>12</sub> H <sub>20</sub> N <sub>2</sub> O <sub>4</sub>	56.23	7.87	10.93
<b>V</b>	120–121	57.15	9.90	11.03	C <sub>13</sub> H <sub>22</sub> N <sub>2</sub> O <sub>4</sub>	57.76	8.20	10.36
<b>VI</b>	125–126	58.35	8.36	9.77	C <sub>14</sub> H <sub>24</sub> N <sub>2</sub> O <sub>4</sub>	59.13	8.51	9.85
<b>VII</b>	~100	57.96	8.77	8.12	C <sub>15</sub> H <sub>26</sub> N <sub>2</sub> O <sub>4</sub>	60.38	8.78	9.39
<b>VIII</b>	~125	61.38	9.41	7.44	C <sub>18</sub> H <sub>32</sub> N <sub>2</sub> O <sub>4</sub>	63.50	9.47	8.23
<b>X</b> <sup>b</sup>	85–92	61.11	5.52	7.62	C <sub>16</sub> H <sub>20</sub> N <sub>2</sub> O <sub>4</sub>	63.14	6.62	9.20

<sup>a</sup> Sulfur content ~2%. <sup>b</sup> Sulfur content 4.10–4.85%.

**Table 3.** IR spectra of *N,N'*-bis[(2-vinyloxy)ethyl]amides **III–VIII**, **X** (in CCl<sub>4</sub>, cm<sup>-1</sup>)

Compd. no.	ν(N–H)		ν(C=O)		ν[C(O)NH]		ν(C=C–O)	
<b>III</b>	3320	3440	1645	1675	1510	1545	1620	1640
<b>IV</b>		3440	1660 br	1680 sh	1515		1618	1638
<b>V</b>		3450	1665	1680 sh	1515		1618	1638
<b>VI</b>		3440	1665	1680 sh	1520		1620	1640
<b>VII</b>		3440	1665	1680 sh	1515		1618	1638
<b>VIII</b>		3450	1665	1680 sh	1510		1620	1640
<b>X</b>	3422	3472	1710	1720	1525	1550	1618	1638

**Table 4.** <sup>1</sup>H and <sup>13</sup>C NMR spectra of *N,N'*-bis[(2-vinyloxy)ethyl]amides **III–VIII**, **X**

Compd. no.	R	<sup>1</sup> H NMR spectrum, CDCl <sub>3</sub> , δ, ppm				
		NH, s	CH <sub>2</sub> =CHO		OCH <sub>2</sub> , NCH <sub>2</sub>	R
<b>III</b> <sup>a</sup>	CH <sub>2</sub>	8.14	6.49 q, 4.19 d.d, 3.98 d.d	3.31 m	3.67 m	
<b>IV</b>	(CH <sub>2</sub> ) <sub>2</sub>	6.39	6.44 q, 4.18 d.d, 4.02 d.d	3.73 t, 3.51 q	2.53 s	
<b>V</b>	(CH <sub>2</sub> ) <sub>3</sub>	6.16	6.44 q, 4.21 d.d, 4.05 d.d	3.76 t, 3.53 q	2.24 t, 1.97 m	
<b>VI</b>	(CH <sub>2</sub> ) <sub>4</sub>	5.99	6.45 q, 4.19 d.d, 4.03 d.d	3.75 t, 3.54 q	2.21 m, 1.66 m	
<b>VII</b>	(CH <sub>2</sub> ) <sub>5</sub>	5.98	6.44 q, 4.20 d.d, 4.03 d.d	3.74 t, 3.52 q	2.19 t, 1.61 m, 1.32 m	
<b>VIII</b>	(CH <sub>2</sub> ) <sub>8</sub>	5.85	6.44 q, 4.19 d.d, 4.03 d.d	3.74 t, 3.53 q	2.16 t, 1.61 m, 1.2 m	
<b>X</b>	<i>o</i> -C <sub>6</sub> H <sub>4</sub>		6.41 q, 4.47 d.d.d, 4.16 d.d	3.99 m, 3.95 m	7.84 m, 7.71 m	
<b>X</b> <sup>a</sup>	<i>o</i> -C <sub>6</sub> H <sub>4</sub>		6.45 q, 4.22 d.d, 3.97 m	3.97 m	7.87 m	

Compd. no.	<sup>13</sup> C NMR spectrum, CDCl <sub>3</sub> , δ, ppm					
	C=O	OCH=	CH <sub>2</sub> =	OCH <sub>2</sub>	NCH <sub>2</sub>	R
<b>III</b> <sup>a</sup>						
<b>IV</b>	172.36	151.43	87.30	66.64	38.89	31.66
<b>V</b>	172.72	151.32	87.56	66.74	38.80	35.25, 21.88
<b>VI</b>	172.89	151.38	87.31	66.72	38.74	36.18, 25.01
<b>VII</b>	173.20	151.42	87.37	66.83	38.76	36.32, 28.67, 25.18
<b>VIII</b>	173.33	151.39	87.32	66.84	38.69	29.18, 25.66
<b>X</b>	168.19	151.30	87.19	64.62	37.11	134.08 ( <i>o</i> -), 132.11 ( <i>p</i> -), 123.40 ( <i>m</i> -)
<b>X</b> <sup>a</sup>	168.02	151.75	87.69	64.79	37.10	134.82 ( <i>o</i> -), 132.84 ( <i>p</i> -), 123.42 ( <i>m</i> -)

<sup>a</sup> In DMSO-*d*<sub>6</sub>.

slowly, and in 5 h the isothiocyanate conversion did not exceed ~40–50% (Table 1, run no. 1). Although the 100% isothiocyanate conversion was attained by increasing temperature to 120–143°C (Table 1, runs nos. 3 and 4), the product therewith virtually completely transformed presumably into the corresponding 2-methyl-1,3-oxazolanone [1]. Its IR spectrum lacked not only absorption bands of isothiocyanate group but also those of vinyloxy and NH groups. A considerable content of sulfur indicated by elemental analysis revealed the presence of derivatives of adduct **II**. It is quite regular taking into account that the catalyst might be partially or completely bound by the acid. We showed formerly [19, 20] that triethylamine accelerated both addition of carboxylic acids to isothiocyanate **I** and decomposition of the arising mixed anhydride with elimination of COS to yield amides.

Thus it is obvious that with stronger acids the reaction occurs with more difficulty quite in keeping with the electrophilic character of the isothiocyanate group. Presumably this was the reason why we failed to synthesize the corresponding amide **IX** from isothiocyanate **I** and maleic acid (Table 1, runs nos. 15–19). In this case the process was additionally complicated by strong tarring and polymerization of reaction products and perhaps by their cyclization (weak bands of NH and  $\text{CH}_2=\text{CHO}$  in the IR spectra). Same as with malonic acid the conversion at 55–60°C did not exceed ~40–50% (Table 1, run no. 15) at obvious tarring of the product, and at higher temperature (Table 1, runs nos. 16–19) although the isothiocyanate conversion attained 100%, the reaction products were rubber-like polymers. As with malonic acid (Table 1, run no. 4), the elemental analysis showed a large content of sulfur (>14%) in the reaction products. The presence of intermediate **II** (or its derivatives) in the reaction products is also revealed by a relatively strong absorption band in the IR spectra at  $1700\text{ cm}^{-1}$  ( $\text{C}=\text{O}$ ) [19].

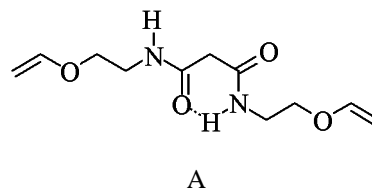
With phthalic acid we succeeded to obtain amide **X** in a low yield (not estimated) at 95–105°C (Table 1, run no. 22), but the main reaction products were resinous substances. In the other runs (Table 1, runs nos. 20, 21) the  $^1\text{H}$  NMR spectra revealed the presence of amide **X** in the products, but its isolation was hampered by strong tarring of the reaction mixture.

Reaction products **III–VIII**, **X** are colorless powders, soluble in the most organic solvents, purified by reprecipitation from acetone with hexane. Their composition and structure were confirmed by

elemental analyses (Table 2), IR (Table 3), and  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra (Table 4). The assignment of the absorption bands in the IR spectra was based on the data from [22–25].

The amide molecules are known to be strongly associated in the solid state due to formation of strong hydrogen bonds. Therefore to facilitate the identification of absorption bands the IR spectra of compounds were registered from diluted solutions (in  $\text{CHCl}_3$ ) where the hydrogen bonds as a rule were partially or completely dissociated (depending on the associate type and the solution concentration).

The frequency values of the absorption bands from the main structural fragments of amides **III–VIII**, **X** confirm their structure and indicate that in diluted solutions amides **IV–VIII** are present mainly or exclusively as a single isomer, *Z*-(*trans*), in conformity with the published data. It is well known that compounds of amide type possess *Z,E*-isomerism with respect to C–N bond [24], and the more stable is commonly the *Z*-isomer to which belongs the high-frequency component of the N–H bond vibrations. The appearance of the second absorption band corresponding to the N–H bond (at  $3320\text{ cm}^{-1}$ ) and strongly reduced value of the low-frequency component in the carbonyl doublet of the amide **III** spectrum is probably due to occurrence of a hydrogen bond with formation of a stable six-membered ring of A type.



In the  $^1\text{H}$  NMR spectrum (in acetone- $d_6$ ) of amide **III** also appear two signals from NH group at 7.82 and 7.25 ppm (with integral intensity ratio ~1:1.5–2). Also the other proton signals are split and displaced in contrast to the spectra of the other amides **IV–VIII** where are registered signals from a single isomer.

The more comprehensive analysis of the spectral characteristics of amides synthesized will be published elsewhere.

## EXPERIMENTAL

IR spectra were registered on a double-beam spectrometer Specord 75IR from KBr pellets and solutions in  $\text{CHCl}_3$  of concentration 0.02–0.005 mol  $\text{l}^{-1}$  in cells 0.01 mm and 10 mm thick respectively. The

measuring scale was 100/100 cm<sup>-1</sup>, scanning time 11 min. The resolution of the spectrometer in 1700–1500 and 3500–3200 cm<sup>-1</sup> regions was 1 and 2 cm<sup>-1</sup> respectively. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on spectrometer Bruker DPX-400 (400 and 100 MHz respectively, ~5–10% solution in CDCl<sub>3</sub>) at room temperature, internal reference HMDS.

2-(Vinylloxy)ethyl isothiocyanate (**I**) was synthesized by method [26], the dicarboxylic acids used were commercial products purified by recrystallization.

**N<sup>1</sup>,N<sup>3</sup>-Bis[2-(vinylloxy)ethyl]malonamide (III)**, **N<sup>1</sup>,N<sup>4</sup>-bis[2-(vinylloxy)ethyl]succinamide (IV)**, **N<sup>1</sup>,N<sup>5</sup>-bis[2-(vinylloxy)ethyl]pentanediamide (V)**, **N<sup>1</sup>,N<sup>6</sup>-bis[2-(vinylloxy)ethyl]hexanediamide (VI)**, **N<sup>1</sup>,N<sup>7</sup>-bis[2-(vinylloxy)ethyl]heptanediamide (VII)**, **N<sup>1</sup>,N<sup>10</sup>-bis[2-(vinylloxy)ethyl]decanediamide (VIII)** and **N<sup>1</sup>,N<sup>2</sup>-bis[2-(vinylloxy)ethyl]phthalamide (X)** were prepared under conditions indicated in Table 1. As a rule, a calculated amount of dicarboxylic acid was added to the stirred solution of triethylamine (10–15 wt%) in 2-(vinylloxy)ethyl isothiocyanate (**I**). In the synthesis of amides **III** and **X** was used a 50% excess of isothiocyanate (**I**) over the stoichiometric amount. The reaction products were purified by reprecipitation with hexane from acetone solution.

#### REFERENCES

- Nedolya, N.A., Tolmachev, S.V., and Zinov'eva, V.P., *Zh. Org. Khim.*, 2001, vol. 37, no. 8, pp. 1133–1137.
- Bogemann, M., Petersen, S., Schultz, O.-E., and Soll, H., *Methoden der organischen Chemie (Houben-Weyl)*, Muller, E., Ed., Stuttgart: Georg Thieme Verlag, 1955, vol. 9, pp. 867–884.
- Organic Sulphur Compounds*, Kharasch, N., Ed. Pergamon Oxford: Press, 1961, vol. 1, pp. 326–338.
- Smith, P.A.S., *Open-Chain Nitrogen Compounds*, New York: Benjamin, 1965.
- Ram, R.N., Ashare, R., and Mukerjee, A.K., *Chem. Ind.*, 1983, no. 14, p. 569.
- Drobnica, L., Kristian, P., and Augustin, J., *The Chemistry of Cyanates and Their Thio Derivatives*, Patai, S., Ed., Chichester: John Wiley & Sons, 1977, part 2, pp. 1003–1221.
- Comprehensive Organic Chemistry*, Barton, D. and Ollisa, U., Eds., Oxford: Pergamon Press, 1984.
- Challis, B.C., and Challis, J.A., in *Comprehensive Organic Chemistry*, Sutherland, I.O., Ed., Pergamon Press, Oxford, 1979.
- Nedolya, N.A. and Trofimov, B.A., *Sulfur Reports*, 1994, vol. 15, no. 2, pp. 237–316.
- Nedolya, N.A. and Trofimov, B.A., *Sulfur Reports*, 1994, vol. 15, no. 3, pp. 339–380.
- Nedolya, N.A., Brandsma, L., Zinov'eva V.P., Trofimov, B.A., *Zh. Org. Khim.*, 1997, vol. 33, no. 1, pp. 91–96.
- Nedolya, N.A., Brandsma, L., Zinov'eva, V.P., and Trofimov, B.A., *Zh. Org. Khim.*, 1998, vol. 34, no. 10, pp. 1559–1564.
- Nedolya, N.A., *Novel Chemistry Based on Isothiocyanates and Polar Organometallics*, PhD. Thesis (Utrecht University, The Netherlands), 1999, 144 p.
- Brandsma, L., Nedolya, N.A., Tarasova, O.A., and Trofimov, B.A., *Khim. Geterotsikl. Soed.*, 2000, no. 11, pp. 1443–1463.
- Brandsma, L., Tolmachev, S.V., and Nedolya, N.A. *Abstracts of Papers, Eurasian Conference on Heterocyclic Compounds*, Suzdal, 2000, p. 44.
- Nedolya, N.A., Papsheva, N.P., Afonin, A.V., and Trofimov, B.A., *Zh. Org. Khim.*, 1991, vol. 27, no. 10, pp. 2239–2241.
- Nedolya, N.A., Papsheva, N.P., Afonin, A.V., Trofimov, B.A., Kukhareva, V.A., and Kashik, T.V., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1991, no. 10, pp. 2370–2372.
- Nedolya, N.A., Papsheva, N.P., Afonin, A.V., Trofimov, B.A., Kukhareva, V.A., and Kashik, T.V., *Sulfur Lett.*, 1992, vol. 14, nos. 2–3, pp. 103–116.
- Nedolya, N.A., Gerasimiva, V.V., Papsheva, N.P., and Trofimov, B.A., *Sulfur Lett.*, 1992, vol. 14, nos. 2–3, pp. 117–128.
- Nedolya, N.A. and Gerasimova, V.V., *Zh. Org. Khim.*, 1993, vol. 29, no. 2, pp. 265–271.
- Nedolya, N.A. and Trofimov, B.A., *Zh. Fiz. Khim.*, 1977, vol. 51, no. 2, pp. 345–348.
- Barnes, A.J. and Orville-Thomas, W.J., *Vibrational spectroscopy. Modern Trends*, Elsevier, 1977.
- Durig, J.R. and Gerson, D.J., *J. Mol. Struct.*, 1981, vol. 71, pp. 131–145.
- The Chemistry of Amides*, Zabicky, J., Ed., New York: John Wiley, 1970.
- De Klein, W.J., *Spectrochim. Acta*, 1971, vol. 27A, no. 1, pp. 93–102.
- Trofimov, B.A., Nedolya, N.A., Papsheva, N.P., Gerasimova, V.V., and Voronkov, M.G., *Zh. Org. Khim.*, 1988, vol. 24, no. 8, pp. 1771–1772.