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Reactions of Heterocumulenes with Organometallic Reagents: XII*. Reaction of Metallated Vinyl Ethers with Isocyanates: Ingenious Synthesis of Pyruvic Acid Amides

N. A. Nedolya and N. I. Shlyakhtina

Faworsky Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences, Irkutsk, 664033 Russia
e-mail: nina@irioch.irk.ru

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Abstract—Acid hydrolysis of 2-alkoxy-N-substituted acrylamides easily obtained by reacting α-metallated vinyl ethers with isocyanates proceeded regiospecifically leading to the formation in a high yield of virtually undescribed and difficultly available 2-oxo-N-substituted propanamides.

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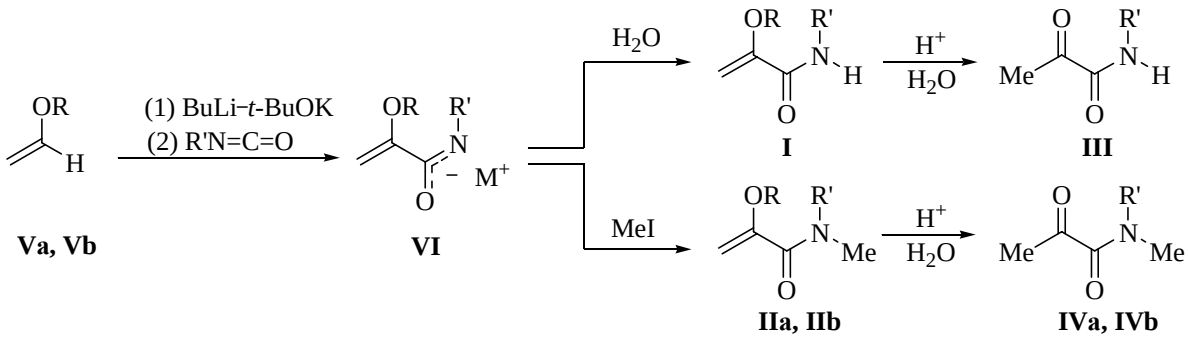
Vinyl ethers find versatile applications [2], in particular, in the synthesis of carbonyl compounds, including industrially important acetaldehyde, oxyaldehydes, 19-norsteroids (promising substances for the hormonal therapy) etc., due to the high ability of the vinyloxy group to undergo protolytic [3, 4]. The acid hydrolysis of the α-metallated vinyl ethers with electrophiles readily provides more complex and functionalized keto-systems which mostly are unavailable by other procedures [5].

Involving into the reaction with α-metallated vinyl ethers {alkoxyethane carbanions easily generated in situ by treatment with the Lochmann–Schlosser base (*n*-BuLi–*t*-BuOK, THF–hexane, –100...–40°C [6])} accessible aliphatic and aromatic isocyanates that we have recently performed [7] ensures a simple and convenient route to a new family of previously unknown and difficultly available N-mono- **I** and N,N-di- **II** substituted acrylamides, promising monomers, synthons, and building blocks for the fine organic synthesis, including a synthesis of ingenious keto-systems.

However, inasmuch as 2-alkoxy-*N*-alkyl- **I** and 2-alkoxy-*N*,*N*-dialkyl- **II** acrylamides [7] are, on the one hand, α-substituted vinyl ethers and on the other hand, amides of α-substituted acrylic acid, i.e., typical representatives of captodative alkenes [8], it is fairly difficult to predict a priori their reactivity, in particular, their behavior under acid hydrolysis conditions. The double carbon-carbon bond in vinyl ethers and acrylamides is known to possess qualitatively different chemical character: electrophilic reactions are characteristic of vinyl ethers and nucleophilic reactions, of acrylamides. Moreover, the structure of compounds **I** and **II** alongside the vinyl group contains a rival reaction center, amide function, and this fact can complicate the reaction (lead to the formation of products mixtures), or totally direct it to another pathway, for the amides are also prone to the hydrolytic cleavage, not only in basic but also in acidic media [9]. At the same time a successful performance of regioselective protolytic cleavage of compounds **I** and **II** at the vinyloxy group opens the way to previously unknown derivatives of pyruvic (2-oxopropanoic) acid that takes a fairly significant part in various biochemical processes [10].

We report here on the investigation of the behavior under acid hydrolysis conditions of certain representatives of α-substituted acrylic acid amides which we have formerly synthesized [7]. The study resulted in demonstrating the retention of the principal properties of vinyl ethers by 2-methoxy-*N*-propyl- (**I**) and 2-alkoxy-*N*-alkyl-*N*-methyl- (**II**) acrylamides, although the presence of the amide fragment in their structure might significantly affect as already mentioned the reactivity of the vinyloxy group. The compounds sufficiently readily underwent a protolytic cleavage giving in high yields previously unknown and inaccessible 2-oxo-*N*-

* For Communication XI, see [1].



M = K, Li; **I, III**, R = Me, R' = *n*-Pr; **II, IV**, R = Me, R' = *n*-Pr (**a**); R = Et, R' = *i*-Pr (**b**); **V**, R = Me (**a**), Et (**b**).

propyl- (**III**) and 2-oxo-*N*-alkyl-*N*-methyl- (**IV**) propanamides (pyruvic acid amides).

The synthesis of acrylamides **I** and **II** was carried out by reaction of α-metallated methyl and ethyl vinyl ethers (**V**) with propyl and isopropyl isocyanates (followed by mild hydrolysis with water solution of ammonium chloride or by N-alkylation with methyl iodide of adduct **VI**) under conditions comprehensively described in [7]. Hydrolysis of compounds **I** and **II** was performed in a mixed water-organic (dioxane, methanol) solvent in the presence of hydrochloric acid taken not in a commonly used catalytic quantity [2–4], but in a super-

Preparation conditions for 2-oxo-*N*-alkyl- **III** and 2-oxo-*N*,*N*-dialkyl- **IV** propanamides by acid-catalyzed hydrolysis of acrylamides **I**, **II**, and **VII** (2 ml of water, 2 ml of dioxane, 1.4 mmol of acrylamide)

Run no.	Reagent no.	Reaction product no.	XR	R'	Temperature, °C	Time, min	Amount of HCl, g[a] (mmol)[b]	Conversion of compounds **I** and **II**, %[c]
1	**Ia**	**III**	OMe	*n*-Pr	~20	10	0.20 (1.8)	~0
2	**Ia**	**III**	OMe	*n*-Pr	~20	10	0.50 (4.5)	~0
3	**Ia**	**III**	OMe	*n*-Pr	30–40	30	0.30 (2.7)	~100
4	**IIa**	**IVa**	OMe	*n*-Pr	~20	10	0.20 (1.8)	~0
5	**IIa**	**IVa**	OMe	*n*-Pr	30–40	40	0.20 (1.8)	~90
6	**IIa**	**IVa**	OMe	*n*-Pr	30–40	135	0.20 (1.8)	~100
7	**IIa**	**IVa**	OMe	*n*-Pr	30–40	60	0.25 (2.3)	~100
8[d]	**IIa**	**IVa**	OMe	*n*-Pr	30–40	60	0.25 (2.3)	~94
9[e]	**IIa**[f]	**IVa**	OMe	*n*-Pr	40–45	60	0.41 (11.3)	~100[g]
10[d]	**IIb**	**IVb**	OEt	*i*-Pr	30–40	60	0.25 (2.3)	~100
11[e]	**IIb**[h]	**IVb**	OEt	*i*-Pr	45	60	0.41 (11.3)	~100[i]
12[d]	**VII**	**IVa**	SEt	*n*-Pr	30–40	180	0.26 (2.4)	~0
13[d]	**VII**	**IVa**	SEt	*n*-Pr	30–40	60	0.50 (4.5)	~70
14[d]	**VII**	**IVa**	SEt	*n*-Pr	30–40	120	0.50 (4.5)	~75[j]

[a] 33% water solution of HCl.
[b] Calculated on dry HCl.
[c] Estimated from the integral intensity of vinyl protons signals in the ^{1}H NMR spectra.
[d] In methanol (2 ml).
[e] In 10 ml of MeOH.
[f] 7.0 mmol.
[g] Preparative yield of compound **IVa** 75%.
[h] 6.0 mmol.
[i] Preparative yield of compound **IVb** 78%.
[j] Reaction product is obviously impure: many foreign signals in the ^{1}H NMR spectrum.

stoichiometric amount (1.3–3.2 mmol of HCl per 1 mmol of substrate), for it had been shown before [11] that the amide moiety efficiently bound the acid catalyst. The investigated reaction conditions and the results obtained are presented in the table.

The reaction progress was monitored by IR [7, 12, 13] and ^{1}H NMR [7, 13, 14] spectra of the reaction mixtures. The intensity of absorption bands ν(C=C) 1580–1630 cm^{-1} and signals at 4.40 d and 5.36 d ppm (CH_2=) were followed in the spectra of compound **I** or signals at 4.21–4.30 d and 4.36–4.46 d ppm (CH_2=) in the spectra of compounds **IIa** and **IIb**, 3.62–3.64 s ppm (OMe) or 1.29 t and 3.77 q ppm (OEt) of alkoxyethene fragment (up till their total disappearance), and simultaneously was observed the appearance of very strong carbonyl absorption bands at 1700 cm^{-1} (MeC=O) and singlets in the ^{1}H NMR spectra [2.37–2.46 ppm (Me)] belonging to the acyl group. The absorption band at 1630 cm^{-1} in the IR spectra of the reaction products corresponds to NC=O vibrations. In the ^{13}C NMR spectrum the acyl carbonyl signal appeared at 194–198 ppm, whereas the amide carbonyl signal was observed at 166–167 ppm. The carbonyl signals of the initial amides **I** and **II** are located in the same region, at 162 and 166 ppm respectively. The lack of vinyl carbon signals [86–89 (CH_2=), 154–157 ppm (C=)] in the ^{13}C NMR spectra of the reaction products is an additional proof of the protolytic cleavage at the vinyloxy group in acrylamides **I** and **II**.

As seen from the table, compounds **I** and **II** are sufficiently easily (from the preparative viewpoint) converted into 2-oxo-*N*-alkyl- (**III**) and 2-oxo-*N*,*N*-dialkyl- (**IV**) propanamides: by short (0.5–1 h) heating of water-dioxane or water-methanol solutions at relatively not high (30–45°C) temperature in the presence of hydrochloric acid. At the same time the reaction did not proceed at room temperature even at the 3.2 molar excess of HCl (see the table, runs nos. *1*, *2*, *4*); these findings showed that compared to the unsubstituted alkyl vinyl ethers whose lower representatives underwent total hydrolytic cleavage in water solutions of low acidity and even neutral [3] the reactivity of alkoxyethenes **I** and **II** containing in the α-position an amide moiety was sharply reduced in the electrophilic addition reactions at the vinyloxy group [15]. The rate of the reaction under study grows naturally with the hydrochloric acid concentration (see the table, runs nos. *6* and *7*). The data available did not reveal any significant effect on the reaction rate of the substituent at the nitrogen atom.

For comparison, the thioanalog of compound **IIa**, *N*-methyl-*N*-propyl-2-(ethylsulfanyl)acrylamide (**VII**) [H_2C=C(SEt)C(=O)N(Me)Pr] prepared analogously from the carbanion of (ethysulfanyl)ethene and propyl-isocyanate [7] in reaction under study proved to be as expected considerably more passive than 2-alkoxy-acrylamides **I** and **II**. Under comparable hydrolysis conditions (see the table, cf. run no. *12* and runs nos. *8* and *10*) compound **VII** showed high stability against hydrolysis and was recovered intact (by the ^{1}H NMR data). Even prolonging the reaction to 3 h (instead of 40–60 min for compound **II**) at HCl concentration 1.7 mmol per 1 mmol of substrate (see the table, run no. *12*) did not initiated the hydrolysis. Only the raising the HCl concentration to 3.2 mmol per 1 mmol of substrate, namely, nearly twofold, resulted in formation of compound **IVa** in ~70% yield (see the table, run no. *13*). However the attempt to raise the conversion of 2-(ethylsulfanyl)acrylamide (**VII**) by increasing the reaction time from 1 to 2 h keeping the same other parameters of reaction from the run no. 13 provided only a target product contaminated with impurities (see the table, run no. *14*).

The electron-donor properties of sulfur atom in vinyl sulfides are known to be less pronounced than those of oxygen atom in the alkoxyethenes (different estimates [4, 16, 17] consider the *p*,π-conjugation in a vinylsulfanyl group to be 2–3 times weaker than in a vinyloxy group). Therefore the π-electron density excessive with respect to ethylene or 3-chloro-1-propene [18] on the β-carbon atom subjected to the electrophile attack in alkyl vinyl sulfides is ~3 times lower (0.06–0.10 of an electron charge) than in the oxygen analogs (0.20–0.25) [16, 17]. The stabilization of the intermediate carbocation by the unshared electron pairs of sulfur is also less efficient. Consequently the α,β-unsaturated sulfides are less active in reactions of electrophilic addition than the corresponding ethers[4, 16, 19]. In particular, the hydrolysis of vinyl sulfides occurs usually 100–1000 times slower than the hydrolysis of the structurally similar vinyl ethers [4, 19, 20].

However a more well-grounded conclusion on the reactivity of 2-(alkylsulfanyl)acrylamides with respect to electrophilic reagents requires additional careful research. At the same time since the hydrolysis of 2-(ethylsulfanyl)acrylamide (**VII**) gives 2-oxo-*N*-propyl-propanamide (**IVa**) that is easily obtained from 2-methoxyacrylamide (**IIa**) (see the table) we consider in the framework of this study to be unnecessary the

optimization of the conditions for protolytic cleavage of compound **VII**.

2-Oxo-*N*-propyl- (**III**) and 2-oxo-*N*,*N*-dialkyl- (**IV**) propanamides are weakly colored fluids, practically odorless. Like the initial alkoxyacrylamides **II**, compounds **IV** exist as isomers mixtures: alongside the characterisic for amides *cis-trans* isomerism [9] caused by hindered rotation around the partially sp^2-hybridized amide bond C(=O)–N also a different reciprocal orientation of the key fragments of the molecule may be manifested, namely, of acyl and amide groups with respect to the C–C bond [Me(O=)C–C(=O)N]. This fact clearly appears in their ^{1}H and ^{13}C NMR spectra: all signals are split (but MeC=O) in the proton spectrum.

The IR spectra of compounds **IVa** and **IVb** contain characteristic absorption bands of C=O stretching vibrations at 1630 and 1700 cm^{-1} belonging respectively to amide and acyl moieties. The absorption of C=C bond commonly observed in the region 1580–1630 cm^{-1} is lacking.

Thus we demonstrated that carbanions generated from available vinyl ethers can be used in reactions with heterocumulenes, in particular, with isocyanates, as efficient synthons of acyl anions providing a possibility to obtain new families and classes of oxocompounds.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Specord 75IR from thin films. NMR spectra were registered on a spectrometer Bruker DPX-400 (operating frequency 400 and 100 MHz for ^{1}H and ^{13}C respectively) from ~5–10% solutions of samples in $CDCl_3$ at room temperature, internal reference HMDS.

Compounds **I** and **II** were prepared in 66–76% yield as described in [7].

2-Oxo-*N*-propylpropanamide (III). To a solution of 0.20 g (1.40 mmol) of compound **I** in 2 ml of dioxane was added a solution of 0.30 g (2.71 mmol) of 33% HCl in 2 ml of water, and the mixture was stirred for 0.5 h at 30–40°C. The reaction product was extracted into hexane and ethyl ether, the extract was dried with K_2CO_3, and the solvents were removed under reduced pressure to obtain a residue of 0.18 g (~100%) of compound **III**. ^{1}H NMR spectrum, δ, ppm: 7.25 br.s (1H, NH), 3.23 m (2H, NCH_2), 2.46 s (3H, MeC=O), 1.55 m (2H, β-CH_2), 0.93 t (3H, Me). Found, %: C 55.65; H 8.70; N 10.55. $C_6H_{11}NO_2$. Calculated, %: C 55.80; H 8.58; N 10.84.

***N*-Methyl-2-oxo-*N*-propylpropanamide (IVa).** To a solution of 1.10 g (7.0 mmol) of compound **IIa** in 10 ml of methanol was added a solution of 1.25 g (11.30 mmol) of 33% HCl in 10 ml of water, and the mixture was stirred for 1 h at 40–45°C. The reaction mixture was treated with water solution of KOH. The reaction product was extracted into ethyl ether, the extract was dried with K_2CO_3, and the solvents were removed under reduced pressure to obtain a residue of 0.75 g (75%) of compound **IVa**, after distillation in a vacuum yield was 0.63 g (~63%), bp 61–64°C (0.5–1 mm Hg), n_D^{18} 1.4590. IR spectrum, cm^{-1}: 580, 750, 890, 980, 1010, 1100, 1190, 1220, 1270, 1310, 1350, 1380, 1400, 1460, 1490, 1630, 1700, 2870, 2920, 2950. ^{1}H NMR spectrum, δ, ppm: 3.33 m, 3.21 m (2H, NCH_2), 2.96 s, 2.93 s (3H, NMe), 2.39 s (3H, MeC=O), 1.61 m (2H, β-CH_2), 0.90 t, 0.84 t (3H, Me, *J* 7.4 Hz). ^{13}C NMR spectrum, δ, ppm: 198.23, 194.21 (Me<u>C</u>=O), 166.68, 166.09 (NC=O), 50.92, 48.39, 44.68, 44.07, 42.55 (NCH_2), 34.60, 31.92, 30.83, 29.32 (NMe), 28.09, 27.88, 27.43, 27.20 (<u>Me</u>C=O), 21.12, 20.76, 19.56 (β-CH_2), 10.72, 10.46 (Me). Found, %: C 58.89; H 9.21; N 9.66. $C_7H_{13}NO_2$. Calculated, %: C 58.72; H 9.15; N 9.78.

***N*-Isopropyl-*N*-methyl-2-oxopropanamide (IVb).** To a solution of 1.03 g (6.0 mmol) of compound **IIb** in 10 ml of methanol was added a solution of 1.25 g (11.30 mmol) of 33% HCl in 10 ml of water, and the mixture was stirred for 1 h at ~45°C. The reaction mixture was treated with water solution of KOH. The reaction product was extracted into ethyl ether, the extract was dried with K_2CO_3, and the solvents were removed under reduced pressure to obtain a residue of 0.67 g (~78%) of compound **IVb**, after distillation in a vacuum yield was 0.60 g (~71%), bp 53–55°C (0.5–1 mm Hg), n_D^{18} 1.4515. IR spectrum, cm^{-1}: 580, 700, 750, 880, 910, 980, 1010, 1100, 1170, 1200, 1260, 1340, 1370, 1390, 1400, 1460, 1630, 1700, 2920, 2970. ^{1}H NMR spectrum, δ, ppm: 4.59 m, 3.77 m (1H, NCH), 2.70 s, 2.69 s (3H, NMe), 2.27 s (3H, MeC=O), 1.10 d, 1.04 d (6H, Me_2C, *J* 6.7 Hz). ^{13}C NMR spectrum, δ, ppm: 198.95, 198.91 (Me<u>C</u>=O), 167.16, 166.38 (C=O), 48.74, 44.22 (NCH), 28.23, 28.18, 27.69, 27.40 (NMe), 24.94, 22.52, 22.20 (<u>Me</u>C=O), 20.27, 18.83 (<u>Me</u>$_2$C). Found, %: C 59.01; H 9.05; N 9.82. $C_7H_{13}NO_2$. Calculated, %: C 58.72; H 9.15; N 9.78.

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