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Intramolecular 1,5-Hydride Transfer in Rearrangements of Ammonium Salts Containing Alkoxycarbonylmethyl and 4-Penten-2-ynyl Groups

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Abstract — The Stevens 3,2 rearrangement of dialkylammonium salts containing, along with 4-penten-2-ynyl, an alkoxycarbonylmethyl group, gives mostly hydrogenated products, with simultaneous dealkylation and aldehyde formation. On acid treatment enamine amino esters give keto esters or their further transformation product, 4-ethyl3-hydroxy-5-methyltetrahydrofuran-2-one.

Earlier we showed that dimethyl- and pentamethylene(methoxycarbonylmethyl)(4-penten-2-ynyl)-ammonium bromide under the action of ethereal suspension of sodium alkoxide undergo Stevens 3,2 rearrangement to provide allenic α-dialkylamino esters whose allene–diene rearrangement under the reaction conditions produces dienic amino esters [1, 2]. It is interesting that a diethyl analog **Ia** of the above salts under the same conditions give hydrogenated rearrangement products, with simultaneous elimination of an ethyl group as acetaldehyde [2].

It is known that tertiary amines having an α-hydrogen atom are potential donors of hydride ions and are applied for reduction of electrophilic multiple bonds [3, 4]. The key stage of these reactions is imonium salt formation either via direct hydride transfer from the amine to the multiple bond or via other routes, the most probable of which involves charge-transfer complexes. Intramolecular hydride transfers have also been reported, viz. in isomerization of carbonium ions [5–7], as well as transannular 1,3- and 1,5-hydride transfers [8, 9] and 1,5-hydride transfers in acyclic compounds [10, 11].

In the present work we performed a detailed study of hydride-transfer reactions in the products of the Stevens 3,2 rearrangement of ammonium salts incorporating both 4-penten-2-ynyl and alkoxycarbonylmethyl groups, with various alkyl groups both at the ammonium nitrogen and in the ester group (salts **Ia**–**Ie**) under the action of ethereal suspension of sodium alkoxide (Table 1).

Hydrogenated reaction products are probably formed by a scheme, according to which rearrangement products **IIa**–**IIe** undergo 1,5-hydride transfer, yielding mainly imonium salts **IIIa**–**IIIe**. Under the reaction conditions, the latter convert to enamines **IVa**–**IVe** mixed with amino esters **Va**–**Ve**. Treatment of the reaction mixture immediately after formation of **IIIa**–**IIIe** with water gives rise to hydrolysis products, amino esters **VIa**–**VIe** and **VIIa**–**VIIe**, which can also be formed by aqueous and acid hydrolysis of

Table 1. Hydrogenated products of the Stevens rearrangement of salts **Ia**–**Ie**

Starting salt	Amino ester	Yield, %	bp, °C (*p*, mm)	n_D^{20}	Found, %			Formula	Calculated, %		
					C	H	N		C	H	N
Ia	**VIa**	26	70–72 (3)	1.4800	64.92	9.35	7.95	$C_{10}H_{17}NO_2$	65.97	9.24	7.65
Ib	**VIb**	11	98–101 (4)	1.4762	66.67	9.29	6.92	$C_{11}H_{19}NO_2$	67.00	9.67	7.11
Ic	**VIc**	14	97–98 (2)	1.4700	68.47	9.59	6.31	$C_{12}H_{21}NO_2$	68.24	9.95	6.63
Id	**VId**	12	98–99 (7)	1.4718	66.35	9.37	6.85	$C_{11}H_{19}NO_2$	67.04	9.64	7.11
Ie	**VIe**[a]	10	112–118 (2)	–	64.85	9.33	6.65	$C_{12}H_{21}NO_3$ (88%) + $C_{11}H_{19}N$ (12%)	65.43	9.52	6.45

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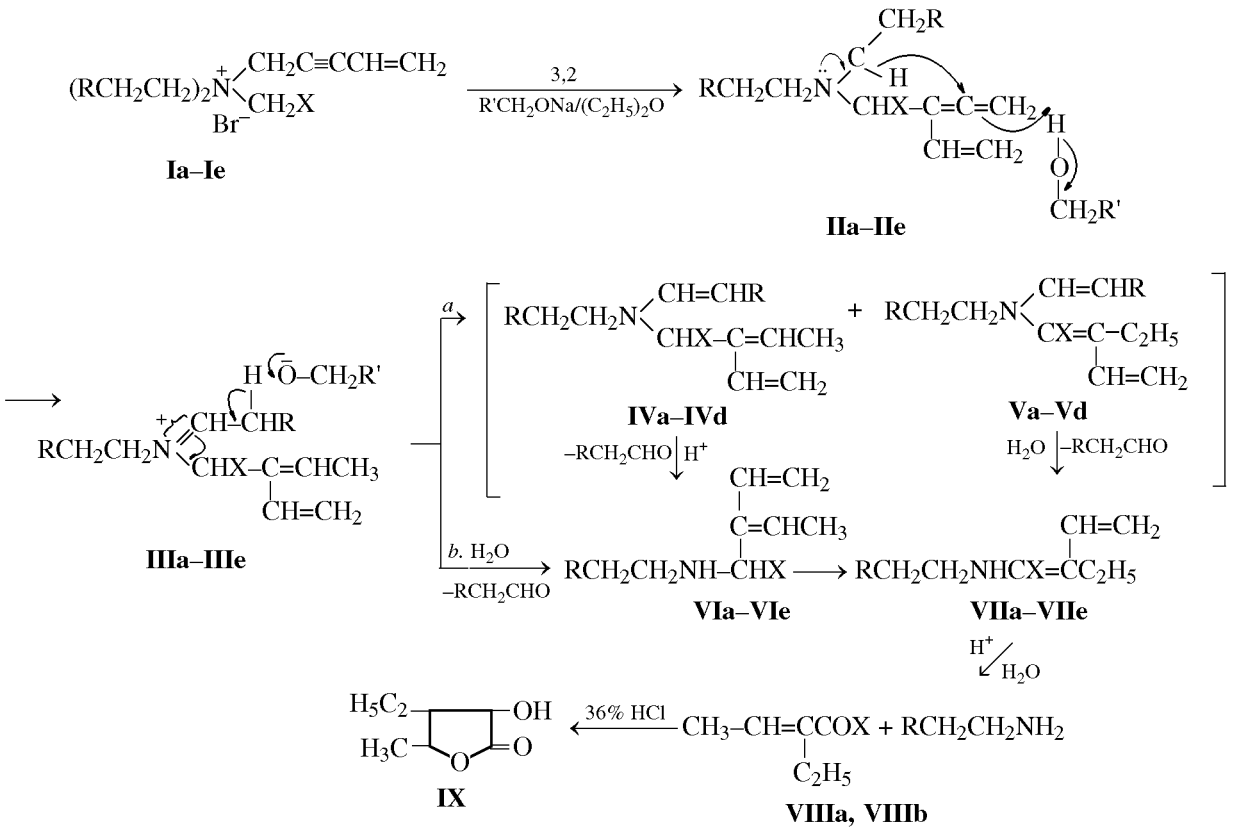
Table 1. (Contd.)

Starting salt	Keto ester	Yield, %	mp, °C (*p*, mm)	n_D^{20}	Found, %		Formula	Calculated, %	
					C	H		C	H
Ia	**VIIIa**	17	87–89 (7)	1.4610	61.38	8.0	$C_8H_{12}O_3$	61.53	7.70
Ib	**VIIIa**	26	83–85 (6)	1.4614	–	–	–	–	–
Ic	**VIIIa**	27	80–82 (4)	1.4609	–	–	–	–	–
Id	**VIIIb**[b]	19	85–89 (3)	–	63.91	8.5	$C_9H_{14}O_3$	63.53	8.2
Ie	**VIIIb**[b]	14	84–89 (3)	–	63.94	8.4	$C_9H_{14}O_3$	63.53	8.2

[a] Contains 12% of (4-penten-2-ynyl)dipropylamine. [b] Contain 15 and 20% of 2-oxo-3-vinyl-3-pentenoic acid, respectively.

mixtures of **IVa**–**IVe** and **Va**–**Ve**. Treatment of mixtures of **VIa**–**VIe** and **VIIa**–**VIIe** with dilute hydrochloric acid results in formation of alkylamines and keto esters **VIIIa, VIIIb**. When treated with concentrated hydrochloric acid, the latter convert to 4-ethyl-3-hydroxy-5-methyltetrahydrofuran-2-one (**IX**).



I–**VII**, R = R' = H (**a**); R = CH_3, R' = H (**b**); R = C_2H_5, R' = H (**c**); R = H, R' = CH_3 (**d**); R = R' = CH_3 (**e**); **VIII**, R = H (**a**), CH_3 (**b**); X = $COOCH_2R'$.

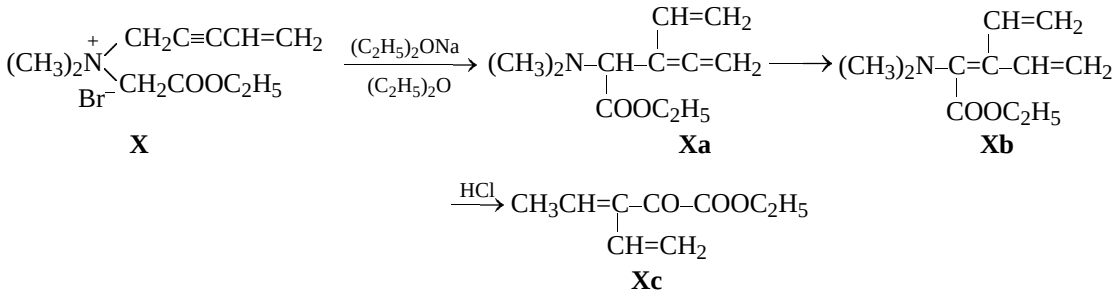
A detailed study of the reaction on an example of salt **Ia** showed that the ether extract after the rearrangement has been complete contains 87% of amine products (titrimetric data). The major component of this mixture is enamine **IVa** (~73%). The fractions of the other components, amino ester **Va** and diethyl(4-penten-2-ynyl)amine, are ~8 and 5%, respectively (GLC data). Note that the fraction of amine **Va** in the mixture increases as the reaction temperature and time, as well as the amount of the base are increased.

Comparison of published [3, 4] and our results led us to conclude that the hydride transfer in the rearrangement products actually gives imonium salts **III** which further transform by route *a* (see scheme). This conclusion follows from the fact that treatment with water of ethereal extract of the reaction mixture with compound **I** sharply decreases the fraction of compound **IVa** in the mixture and increases the fraction of compound **VIa** to 65% (GLC data).

However, route *b* should also not be ruled out, since treatment of anhydrous reaction residue (after decantation of the solvent) with water, followed by extraction with ether, too, gave compounds **VIa** and **VIIa**, the former much prevailing. Moreover, acetaldehyde could be detected as 2,4-dinitrophenylhydrazone.

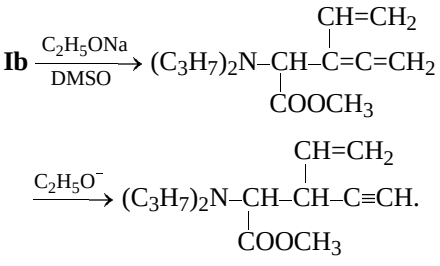
With ethoxycarbonylmethylammonium salts **Id, Ie**, the yields of hydride transfer are rather low, and the reaction mixture contains nonhydrogenated 3,2-rearrangement products. The latter under the reaction conditions undergo prototropic isomerization followed by hydrolysis to convert to ester **Xc**. The fractions of keto ester **VIIIb** in the reaction mixtures are 15 and 20%, respectively (GLC data).

The structure of keto ester **Xc** was proved by spectral methods and, in addition, by the independent synthesis by rearrangement of (ethoxycarbonylmetyl)-dimethyl(4-penten-2-ynyl)ammonium bromide (**X**) followed by hydrolysis of the isomerized rearrangement product **Xb**.



X **Xa** **Xb** **Xc**

We also studied the effect of solvent nature and temperature on the yields of hydrogenated reaction products (with salt **Ib** as an example; Table 2). It was found that the overall yield of the hydrogenated products **VIb, VIIb** is only slightly affected by solvent, but in the reaction mixture in DMSO we detected acetylene **XIa** (~4%). Its formation can be explained by the allene–acetylene rearrangement of the Stevens rearrangement product under the reaction conditions, which is quite probable in DMSO. Steric factors should also be neglected, since the attack of the base on the hydrogen atom at a terminal allenic carbon atom in this substrate is more favored sterically than on the hydrogen atom at the carbon atom neighboring to nitrogen.



XIa

Table 2. Effect of temperature and solvent nature on the yield of hydrogenated rearrangement products of salt **Ib**

Solvent	Temperature, °C	Yield, %	
		VIb	**VIIIa**
Benzene	30–32	12	22
	78–80	10	23
Ether	30–32	14	23
DMSO	30–32	11	22
	40–45	9	20

Two factors are worth mentioning. First, the reaction and the subsequent distillation of the reaction products are accompanied by strong tarring, which affects the yield of the products (Table 1). Second, the reaction yields ~5–12% of amine products of elimination, the starting dialkyl(4-penten-2-ynyl)-amines.

The structures of the products were proved by IR and ^{1}H NMR spectroscopy and mass spectrometry (Table 3), and their purity was controlled by GLC.

Table 3. IR and ^{1}H NMR spectra of compounds **VIa**–**VIe, VIIIa, VIIIb, IX, Xb**, and **Xc**

Comp. no.	IR spectrum, cm^{-1}	^{1}H NMR spectrum (CCl_4), δ, ppm (*J*, Hz)
VIa	920, 1635, 3030, 3090 ($CH=CH_2$), 975 (CH=CH), 1080 (C–N), 1230 (C–O), 1735 (C=O), 3340 (NH)	1.02 t (3H, CH_2CH_3), 1.53 m (1H, NH), 1.75 d (3H, $CH_3C=$), 2.50 q (2H, CH_2CH_3, *J* 7.4), 3.59 s (3H, CH_3O), 4.30 s (1H, NCH), 4.7–5.4 m (2H, $CH_2=$), 5.74 m (1H, $CH_3CH=$, *J* 17.3), 6.10 m (1H, CH=, J_{trans} 12.0, J_{cis} 11.5)
VIb	930, 1610, 1640, 3020, 3090 ($CH=CH_2$), 970 (CH=CH), 1070 (C–N), 1225 (C–O), 1740 (C=O), 3345, 3465 (NH)	0.89 t (3H, $CH_2CH_2CH_3$), 1.09–1.69 m (2H, $CH_2CH_2CH_3$), 1.53 m (1H, NH), 1.78 d (3H, $CH_3C=$), 2.42 q (2H, $CH_2C_2H_5$, *J* 7.4), 3.64 s (3H, CH_3O), 4.24 s (1H, NCH), 4.91 d (1H, $CH_2=CH$, J_{cis} 10.7), 5.36 d (1H, $CH_2=CH$, J_{trans} 17.3), 5.80 m (1H, $CH_3CH=$, *J* 21.3), 6.16 m (1H, $CH_2=CH$)
VIc	920, 990, 1615, 1645, 3090 ($CH=CH_2$), 1080 (C–N), 1740 (C=O), 2130 (C≡C), 3340, 3470 (NH)	0.60–1.00 m (3H, CH_2CH_3), 1.01–1.53 m (4H, $CH_2CH_2CH_3$), 1.46 s (1H, NH), 1.69 d (3H, $CH_3C=$, *J* 8.0), 2.16–2.51 m (2H, $CH_2C_3H_7$), 3.55 s (3H, CH_3O), 4.17 s (1H, NCH), 4.75 d (1H, $CH_2=CH$, J_{cis} 11.3), 5.17 d (1H, $CH_2=CH$, J_{trans} 17.3), 5.65 m (1H, $CH_3CH=$, *J* 22.6), 6.06 m (1H, $CH_2=CH$)
VId	790, 850, 1660 (C=CH), 910, 1620, 3085 ($CH=CH_2$), 1100 (C–N), 1220 (C–O), 1745, 1755 (C=O), 1490, 3375, 3490 (NH)	1.07 t (3H, CH_2CH_3, *J* 8.0), 1.20 t (3H, OCH_2CH_3, *J* 8.0), 1.69 s (1H, NH), 1.87 d (3H, $CH_3C=$, *J* 6.6), 2.60 q (2H, CH_2CH_3), 4.36 s (1H, NCH), 4.98 d (1H, $CH_2=CH$, J_{cis} 12.0), 5.41 d (1H, $CH_2=CH$, J_{trans} 16.6), 5.88 q (1H, $CH_3CH=$, *J* 22.0), 6.22 q (1H, $CH_2=CH$)
VIe	980, 1685 (CH=CH), 1640, 3095 ($CH=CH_2$), 1730, 1740 (C=O), 1275 (C–O), 1470, 3355, 3470 (NH)	0.95 m (3H, CH_2CH_3), 1.08–1.66 m (3H, CH_2CH_3 and NH), 1.17 t (3H, OCH_2CH_3 *J* 8.0), 1.75 d (3H, $CH_3C=$, *J* 8.0), 2.40 m (2H, $CH_2C_2H_5$), 4.17 q (2H, OCH_2CH_3), 4.28 s (1H, NH), 4.92 d (1H, $CH_2=CH$, J_{cis} 12.0), 5.35 d (1H, $CH_2=CH$, J_{trans} 17.0), 5.90 m (1H, $CH_3CH=$, *J* 22.6), 6.26 m (1H, $CH_2=CH$)
VIIIa	820, 1635, 3060, 1675 (C=O), 1125, 1245, 1730 (COO)	0.94 t (3H, CH_2CH_3), 1.92 d (3H, $CH_3C=$, *J* 8.0), 2.28 q (2H, CH_2CH_3, *J* 7.8), 3.77 s (3H, OCH_3), 6.58 m (1H, CH=, *J* 6.7)
VIIIb	820, 1635, 3060, 1670 (C=O), 1125, 1240, 1730 (COO)	0.93 t and 0.95 t (3H, CH_2CH_3), 1.92 d (3H, $CH_3C=$, *J* 8.0), 2.29 q (2H, CH_2CH_3 *J* 7.8), 4.01 q (2H, OCH_2), 6.60 m (1H, CH=, *J* 6.7)
IX	1250 (C–O), 1620 (C=C), 1730 (C=O), 3100–3500 (OH)	1.17 t (3H, CH_2CH_3, *J* 8.0), 1.44 d (3H, $CHCH_3$, *J* 6.7), 1.82–2.80 m (2H, CH_2CH_3), 4.99 m (1H, $CHCH_3$), 7.58 s (1H, OH)
Xb	920, 975, 1575, 1605, 3030, 3090 1070, 1240, 1715 (COO)	0.95 t (3H, CH_2CH_3), 2.37 s (6H, NCH_3), 4.08 q (2H, OCH_2, *J* 7.35), 4.8–5.6 m (4H, $CH_2=$), 6.6–7.5 m (2H, CH=, J_{trans} 18.0, J_{cis} 11.5)
Xc	820, 920, 975, 1630, 1635, 3030, 3090, 1675 (C=O), 1120, 1240, 1735 (COO)	0.97 t (3H, CH_2CH_3), 1.96 d (3H, $CH_3C=$, *J* 8.0), 4.09 q (2H, OCH_2, *J* 7.35), 5.0–5.8 m (2H, $CH_2=$), 6.0–6.95 m (2H, CH=)

EXPERIMENTAL

The IR spectra were obtained in thin film on UR-20 and Specord IR-75 spectrometers. The ^{1}H NMR spectra were measured on a Perkin-Elmer R-12B spectrometer at 60 MHz (in CCl_4, reference TMS). The mass spectra were obtained on an MKh-1320 spectrometer with direct inlet, ionizing energy 70 eV. Gas chromatography was performed on an LKhM-8MD instrument, detector katharometer (column 2000 × 3 mm, 5% OV-17 on Chromaton N-Super, carrier gas helium, rate 60 ml/min, 175–200°C).

General procedure of the rearrangement. *a*. To a suspension of 0.03 mol of salt **Ia**–**Ie** in 20 ml of absolute ether we added sodium alkoxide obtained from 0.06 mol of sodium. After heat evolution had been complete, the mixture was refluxed for an additional 20 min, and ether and water were then added. The organic layer was separated, and the aqueous layer was extracted with ether (3 × 10 ml). The combined extract was acidified with 2.5 N HCl. Nonamine reaction products were extracted with ether (3 × 10 ml), dried with magnesium sulfate, and the solvent was removed. In the ether extract, aldehyde was identified as 2,4-dinitrophenylhydrazone. Vacuum distillation gave keto esters **VIII**. The reaction residue was treated with potash (0–5°C), and amine reaction products were extracted with ether (3 × 10 ml), dried with magnesium sulfate, and the solvent was removed. Amino esters **VI** were isolated by vacuum distillation. The results are listed in Table 1. In the ether extracts by GLC we identified dialkyl(4-penten-2-ynyl)amines. The ^{1}H NMR and IR spectra of compounds **VI** and **VIII** are given in Table 3.

b. To a suspension of 0.03 mol of salt **Ia**–**Ie** in 20 ml of a solvent we added sodium alkoxide obtained from 0.06 mol of sodium. After heat release had been complete, the mixture was kept at a specified temperature for 20–30 min, cooled, the organic layer was separated (except for DMSO which was fully removed by distillation), and the solid residue was washed with absolute ether (3 × 10 ml). The solvent was removed in a vacuum, first at 40–50 mm and then at 4 mm. The residue was examined by GLC and ^{1}H NMR and IR spectroscopy, after which it was acidified with 2.5 N HCl. Further treatment was performed as described in procedure *a*. The solid reaction residue was treated with water, extracted with ether (3 × 10 ml), dried with magnesium sulfate, and the solvent was removed in a vacuum first at 40–50 mm and then at 4 mm. The residue was identified by GLC, using as reference the sample obtained by procedure *a*.

Reaction of dibutyl(methoxycarbonylmethyl)-(4-penten-2-ynyl)ammonium bromide (Ic) with sodium methoxide. According to procedure *a*, from 11.8 g of salt **Ic**, 1.6 g of sodium, and 20 ml of ether we obtained (1) 0.45 g of butyraldehyde 2,4-dinitrophenylhydrazone as needle-like crystals, mp 120–122°C (ethanol), mass spectrum, *m*/*z*: $[M]^+$ 252; (2) 2.7 g of crude methyl 3-ethyl-2-oxo-3-pentenoate (**VIIIa**) contaminated with ~3% of methyl 2-oxo-3-vinyl-3-pentenoate; identified by GLC using authentic samples [2]; distillation gave 1.4 g (26.4%) of keto ester **VIIIa**; and (3) 2.4 g of crude methyl 2-butylamino-3-vinyl-3-pentenoate (**VIc**).

Repeated distillation gave 1.2 g (14%) of amino ester **VIc** which, according to GLC and IR spectral data, contained ~3% of the allene–acetylene rearrangement product **XIb** (weak absorption at 2130 cm^{-1}), 5% of dibutyl(4-penten-2-ynyl)amine (M^+ 211), and 19% of butylamine.

Reaction of (methoxycarbonylmethyl)(4-penten-2-ynyl)dipropylammonium bromide (Ib) with sodium methoxide. According to procedure *b*, from 11.3 g of salt **Ib**, 1.6 g of sodium, and 20 ml of ether we obtained 7.24 g of a mixture of four compounds, the major being **IVb** (85%). Hydrolysis with hydrochloric acid gave (1) 0.48 g (7%) of propionaldehyde 2,4-dinitrophenylhydrazone, mp 140–141°C (ethanol), mass spectrum, *m*/*z*: $[M]^+$ 238; (2) 1.3 g of keto ester **VIIIa**; 2,4-dinitrophenylhydrazone, mp 95–96°C (ethanol), mass spectrum, *m*/*z*: $[M]^+$ 336; (3) 0.75 g (11%) of methyl 2-propylamino-3-vinyl-3-pentenoate (**VIb**), mass spectrum, *m*/*z*: $[M]^+$ 197; as well as 9% of (4-penten2-ynyl)dipropylamine and 13% of propylamine (GLC). From the solid reaction residue, 0.39 g of a mixture was isolated, containing four major components, amino ester **VIb** prevailing (~70%).

Reaction of (ethoxycarbonylmethyl)diethyl(4-penten-2-ynyl)ammonium chloride (Id) with sodium ethoxide. To a suspension of 31.3 g of salt **Id** in 50 ml of ether we added sodium ethoxide obtained from 5.5 g of sodium. The organic layer contained four compounds, prevailing being compound **IVd** (62%) whose hydrolysis yielded ethyl 2-ethylamino-3-vinyl-3-pentenoate (**VId**). Further treatment according to procedure *a* gave (1) 5.1 g (21%) of amino ester **VId**; (2) 2.8 g of a mixture of keto esters **VIIId** and **Xc** (85:15), bp 85–89°C (3 mm), 11% of diethyl(4-penten-2-ynyl)amine, and 16% of ethylamine (GLC).

Reaction of (methoxycarbonylmethyl)(4-penten-2-ynyl)dipropylammonium bromide (Ib) with sodium methoxide in benzene. A mixture of 10.2 g of salt **Ib**, sodium methoxide (obtained from 1.5 g of sodium), and 20 ml of absolute benzene was refluxed for 30 min. Further treatment according to procedure *b* gave (1) 0.65 g (10%) of methyl 2-propylamino-3-vinyl-3-pentenoate (**VIb**); (2) 1.2 g (23%) of 3-ethyl-2-oxo-3-pentenoate (**VIIIa**); and (3) 0.34 g (4%) of propionaldehyde 2,4-dinitrophenylhydrazone, mp 140–141°C (ethanol). By GLC, 7% of (4-penten-2-ynyl)dipropylamine and 16% of propylamine were also found.

Rearrangement of (ethoxycarbonylmethyl)dimethyl(4-penten-2-ynyl)ammonium bromide (X). To 12.4 g of salt **X** in 20 ml of absolute ether we added sodium ethoxide obtained from 2.1 g of sodium. After heat release had been complete, the mixture was refluxed for 20 min, and then ether and water were added. The organic layer was separated, and the residue was extracted with ether. The ether extracts were dried with magnesium sulfate and distilled to obtain 4.35 g (67%) of ethyl 2-dimethylamino-3-vinyl-2,3-pentadienoate (**Xb**), bp 93–94°C (5 mm), n_D^{20} 1.5193. Found, %: C 68.18; H 8.97; N 6.88. $C_{11}H_{17}\cdot NO_2$. Calculated, %: C 67.77; H 8.72; N 7.18.

Acid hydrolysis of ethyl 2-dimethylamino-3-vinyl-2,4-pentadienoate (Xb). To 3.9 g of amino ester **Xb** in 12 ml of absolute ether we added equimolar amount of 1.5 N HCl. The mixture was stirred for 45 min. The ether layer was separated, and the aqueous layer was extracted with two portions of ether. The ether extracts were dried with magnesium sulfate and distilled to obtain 2.4 g (70%) of ethyl 2-oxo-3-vinyl-3-pentenoate (**Xc**), bp 98–100°C (8 mm), n_D^{20} 1.4760. Found, %: C 64.67; H 7.43. $C_9H_{12}O_3$. Calculated, %: C 64.28; H 7.14.

4-Ethyl-3-hydroxy-5-methyltetrahydrofuran-2-one (IX). To 3.2 g of methyl 3-ethyl-2-oxo-3-pentenoate (**VIIIa**) was added with stirring 2.2 ml of 36% HCl. The mixture was heated for 2.5 h at 50–60°C

and then extracted with three portions of ether. The ether extracts were dried with magnesium sulfate and distilled to obtain 1.6 g (56%) of 4-ethyl-3-hydroxy-5-methyltetrahydrofuran-2-one (**IX**), bp 129–130°C (10 mm), n_D^{20} 1.4898 (Table 3). In a similar way, from 3.4 g of ethyl 3-ethyl-2-oxo-3-pentenoate (**VIIIb**) and 2.2 ml of 36% HCl we obtained 1.4 g (49%) of butenolide **IX**.

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