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CONDENSATION OF 2-ALKOXYPROPENALS WITH N,N- AND N,O-1,2-BINUCLEOPHILES. A ROUTE TO 2-(1'-ALKOXYVINYL)IMIDAZO-LIDINES AND -OXAZOLIDINES*

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Condensation of 2-ethoxypropenal with diaminoethylene in different solvents ($CHCl_3$, MeCN, H_2O, DMSO) at room temperature gives an equilibrium mixture (1:1-1.5) of tautomeric 2-(1'-ethoxyvinyl)-1,3-imidazolidine and 2-aminoethylimine of 2-ethoxypropenal as well as 1,2-bis(2'-ethoxypropenylideneamino)ethylene. The latter is readily prepared in quantitative yield using a twofold excess of the aldehyde. ^{1}H NMR was used to demonstrate the effect of heating on the dynamics of the ring-chain tautomeric equilibrium. Reaction of the 2-alkoxypropenals with N-methyl- and N,N'-diphenyl-1,2-diaminoethylenes and with N-phenylaminoethanol gives only the corresponding substituted imidazolidines in 43-95% yield.

Keywords: 2-alkoxypropenals, 1,2-diaminoethylenes, imidazolidines, oxazolidines, condensation, tautomerism.

The reactions of α,β-unsaturated aldehydes with amines occur by various routes. It is known that the reaction of secondary amines with a series of 2-halo-2-alkenals occurs *via ipso* substitution of the halogen atom [1]. Along with this route there are possible 1,4-addition reactions with subsequent condensation to give 1,2-diaminoethanes and 1,3-bis(amino)-2-haloolefines [2]. Piperidine and morpholine add to 2-ethoxypropenal at positions 1, 2 or 1, 4 with subsequent condensation of the product with another molecule of the amine. Mixtures of the isomers of 3,3- or 1,3-diamino-2-ethoxy-1-propenes are formed in the ratio 1:6 in the first case or 3:2 in the second [3]. Evidently the reaction course depends markedly on the nature of the amine.

The reaction of N-acetyl-2-aminopropenal with N,N'-disubstituted diaminoethylenes followed by hydrogenation with $NaBH_4$ forms 1,4-hexahydrodiazepines [4].

Primary amines form Schiff bases with 2-ethoxypropenal [5, 6] although it is noted that in the reaction of acrolein with nucleosides the amino groups of cytosine and thymine can add at the 1,4 positions [7]. β-Amino thiols and β-amino alcohols form a mixture of tautomeric thiazolidines and imino thiols [8] or oxazolidines and imino alcohols [9] with 2-alkoxypropenals.

* Dedicated to Academician of the Russian Academy of Sciences B. A. Trofimov in his 70th jubilee.

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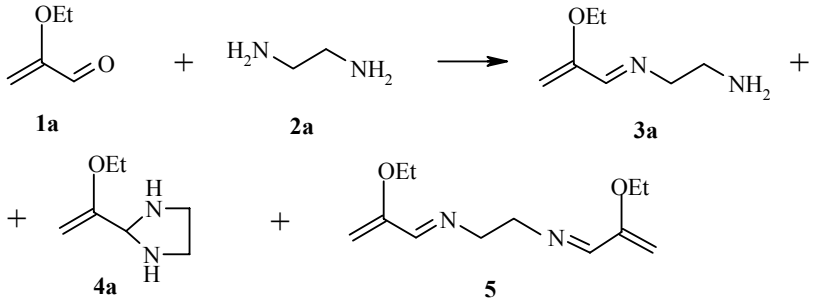
Thus the direction of attack on the C=C bond or the CHO group of the α,β-unsaturated aldehyde depends markedly on the electronic effect of the substituent in the substrate and in the nucleophilic reagent as well as the reaction conditions.

In continuing our work on the chemistry of α-functionally substituted α,β-unsaturated aldehydes and, especially the mutual electronic and steric effect of the captodative substituents in a *gem* position to the double bond we have now studied the regioselectivity of the addition to 2-alkoxypropenals **1** of bifunctional nitrogen nucleophiles **2**, i.e. ethylenediamine, its N- and N,N'-substituted derivatives and also of 2-(N-phenyl-amino)ethanol.

The realization of a possible synthesis of novel 1,3-imidazolidines based on the indicated reaction is connected with the marked interest in the chemistry and the biochemical reactions of this class of compounds at this time [10-17].

We have shown that the reaction of 2-ethoxypropenal (**1a**) with an equimolar amount of 1,2-ethylene-diamine (**2a**) occurs exothermically in the absence of a solvent. Full conversion of aldehyde **1** is seen after several minutes to give three compounds: 3-(2'-aminoethylimino)-2-ethoxypropene (**3a**), 2-(1'-ethoxyvinyl)-imidazolidine (**4a**), and 1,2-bis(2'-ethoxypropylideneamino)ethane (**5**) in the ratio 1:2:1.



1a **2a** **3a** **4a** **5**

This process occurs more slowly in solvents ($CHCl_3$, CH_2Cl_2, MeCN, H_2O). 100% Conversion of aldehyde **1a** occurs over 1 h at 24ºC. It should be noted that the isomeric compounds **3a** and **4a** are found in an equilibrium ratio (1:1-1.5) in all cases and the starting diamine **2a** always remains partially unused. The content of the diimine **5** is 7-17% in the reaction mixture. When the mixture is stored (20ºC) this amount increases somewhat. It is evidently related to the ready reversibility of the condensation of aldehydes with primary amines. Enough traces of moisture in the reaction mixture (or in the air) lead to hydrolysis of compound **4a**. The propenal **1a** so formed then attacks the free amino group of imine **3a** resulting in the thermodynamically stable diimine **5**. The water formed in the latter condensation again hydrolyzes compound **4a** etc. We have, in fact, shown that reaction of diamine **2a** with a twofold excess of aldehyde **1a** gives only the diimine **5** in 100% yield (^{1}H NMR).

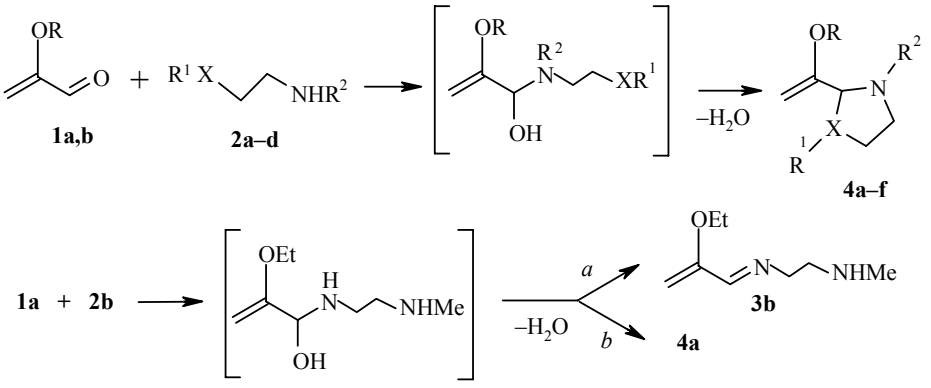
It was not possible to form principally one of the primary condensation products **3a** or **4a** when carrying out the reaction in $CHCl_3$, even with a 2-3-fold excess of diamine **2a**. However, the use of a more polar solvent (DMSO) gave a **3a:4a** ratio of 1:1.7 (without combining with water of condensation).

Since the ring-chain tautomerism of 5-6-membered 1,3-heterocycles affects their reactivity (and hence determines their synthetic use) and is also important for understanding and predicting biochemical processes [10, 16, 17] we have tried to show that compounds **3a** and **4a** are not isomeric (one of which is a labile intermediate) but are actually tautomers. With this in mind we have studied the change in their ratio upon heating the reaction mixture in $CDCl_3$ solution in the NMR spectrometer ampule. After mixing an equimolar amount of reagents over 1.5 h at room temperature a steady (equilibrium) mixture of **3a:4a** (0.9:1) is observed (three control measurements). Heating the ampule for 1 min to 60ºC changes this ratio to 1.38:1 and after 5 min at 60ºC to 1.5:1. Cooling of the ampule to 27ºC (for 10 min) caused the **3a:4a** ratio to return to the starting

equilibrium value (0.9:1). Hence the rate of mutual thermal conversion of compounds **3a, 4a** amounts to minutes and is readily monitored by NMR. Meanwhile, examples are known in which the ratio of tautomers is reached over 3.7 h for oxazolidine–imino alcohols [18] or 7.5 h for thiazolidine-iminothiols [19].

The fraction of diimine **5** does not change upon heating at 60ºC for 6 min in the NMR ampule. Even at higher temperature (80ºC, 1 h, acetonitrile) the reaction of reagents **1a** and **2a** with subsequent cooling leads to the standard ratio **3a:4a** 0.7:1 with an 8% content of diimine **5** in the mixture. However, it was not possible to separate the individual compounds **3a** and **4a**. Upon distillation (block temperature 136ºC) the main reaction product (60-80%) becomes the diimine **5** which was separated in up to 52% yield. Compound **5** is the only reaction product seen when the reaction mixture was analyzed by GLC-MS.

N-Methylated diamines almost always show a higher tendency to cyclization than their analogs unsubstituted on the nitrogen atom [20]. In fact, such a dependence was shown in the reaction of 2-ethoxypropenal **1a** with N-methyldiaminoethylene (**2b**) (20ºC, 1 h) giving a 100% yield (^{1}H NMR) of 2-(1'-ethoxyvinyl)-1-methylimidazolidine (**4b**). Since the starting diamine **2b** has a strongly basic secondary amino group an initial attack in two directions (*a* and *b*) might be expected.



1 a R = Et, **b** R = Me; **2 a** X = NH, $R^1 = R^2$ = H, **b** X = NH, R^1 = H, R^2 = Me, **c** X = NH, $R^1 = R^2$ = Ph, **d** X = O, R^1 = H, R^2 = Ph; **4 a** X = N, R = Et, $R^1 = R^2$ = H, **b** X = N, R = Et, R^1 = H, R^2 = Me, **c** X = N, R = Et, $R^1 = R^2$ = Ph, **d** X = N, R = Me, $R^1 = R^2$ = Ph, **e** X = O, R = Et, R^1 –, R^2 = Ph, **f** X = O, R = Me, R^1 –, R^2 = Ph

However, we never observed the linear isomer **3b** at reasonable concentration although it can be readily identified from the CH=N group proton signal (7.5 ppm for aliphatic systems) [21].

As a result of the lowering of the basicity of the nitrogen atoms *via* the acceptor effects of the phenyl groups in N,N'-diphenyl-1,2-diaminoethane (**2c**) the reaction with 2-ethoxy- and 2-methoxypropenals **1a** and **1b** is much slower than for the diaminoethylenes **2a** and **2b**. At 24ºC the reaction in $CHCl_3$ only occurs to the extent of 30% after 1 h and 80% after 24 h. The 2-(1'-alkoxyvinyl)-1,3-diphenyl-1,3-imidazolidines **4c** and **4d** respectively are stable, crystalline substances.

The reaction of the N-phenylaminoethanol (**2d**) with the 2-alkoxypropenals **1a** and **1b** occurs just slowly and in the same direction. According to ^{1}H NMR data the yield of 2-[1'-ethoxyvinyl]-3-phenyl-oxazolidine (**4e**) is 40% after 1 h and 92% after 24 h. Microwave irradiation markedly speeds up this reaction. Thus, the targeted product **4e** yield is 80% after 4 min, i.e. the rate of the reaction increases by 30 times and the rate of formation of compound **4f** increases by 24 times.

The marked lowering of the basicity of the nitrogen atoms due to the electronegative effects of the substituents in N,N'-diacetyl-N,N'-diaminoethylene results in this diamine not taking part in a reaction with the 2-ethoxypropenal **1a** under comparable conditions (20ºC, 1-25 h).

Hence we have discussed the influence of the electron effects of several substituents at the reactive centers of nitrogen binucleophiles on their regioselectivity and reaction rate with 2-alkoxypropenals. In the conditions chosen the existence of ring-chain tautomerism for the novel 1,3-imidazolidine **4a** containing a vinyloxy group at position 2 has been shown. This fact confirms and develops the recent publication dependence showing that only those N-unsubstituted 1,3-imidazolidines in which the position 2 has a 2-iso-propyl group or two alkyl groups take part in the ring-chain equilibrium while imidazolidines with linear alkyl substituents in position 2 (Me, Et, Pr) exist in $CDCl_3$ exclusively as the cyclic tautomers. For the 2-phenyl- substituted imidazolidine the tautomeric equilibrium is fully shifted to the open isomer side [10]. The introduction of an alkoxyvinyl group at position 2 of the imidazolidines opens up the possibility of their further functionalization.

EXPERIMENTAL

^1H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer (400 and 100 MHz respectively) using $CDCl_3$ with HMDS as internal standard. IR spectra were obtained on a Specord IR-75 spectrometer. Chromato-mass spectrometric analysis was performed on a Hewlett-Packard 5971A instrument (EI, 70 eV, mass selective detector), HP-5890 chromatograph, Ultra-2 column (5% phenylmethylsilicone), evaporator temperature 250ºC, column thermostat temperature 70-280ºC, rate of temperature increase 20 deg./min. An LG MS-1904H 700 watt microwave oven was used for the microwave irradiation with a C-20.1 pyrometer (technoa, from -18ºC to +500ºC).

1,2-Bis(2'-ethoxypropylideneamino)ethane (5). The diamine **2a** (1.12 g, 18.7 mmol) was added to a solution of the propenal **1a** (1.87 g, 18.87 mmol) in $CHCl_3$ (2 ml). After several minutes the ^1H NMR spectrum of the reaction mixture showed 3-(2'-aminoethylimino)-2-ethoxypropene (**3a**) and 2-(1'-ethoxyvinyl)imidazolidine (**4a**), ratio **3a:4a** = 1:1.3. ^1H NMR spectrum of compound **3a**, δ, ppm (*J*, Hz): 1.21 (3H, t, *J* = 6.9, CH_3); 3.00 (2H, m, CH_2); 3.56 (2H, m, CH_2); 3.88 (2H, q, *J* = 6.9, OCH_2); 4.54 (1H, d, *J* = 2.0, $=CH_2$); 4.57 (1H, d, *J* = 2.0, $=CH_2$); 7.58 (1H, s, N=CH). ^1H NMR spectrum of compound **4a**, δ, ppm (*J*, Hz): 1.32 (3H, t, *J* = 7.0, CH_3); 2.87 (2H, dd, *J* = 10.2, *J* = 11.5, CH_2 ring); 3.04 (2H, dd, *J* = 10.2, *J* = 11.5, CH_2 ring); 3.77 (2H, q, *J* = 7.0, OCH_2); 3.99 (1H, d, *J* = 2.0, $=CH_2$); 4.25 (1H, d, *J* = 2.0, $=CH_2$); 4.18 (1H, s, CH). The reaction mixture was dried over $MgSO_4$ and solvent was removed at reduced pressure. Distillation *in vacuo* of the residue gave the product **5** (2.19 g, 52.3%) with bp 127ºC (1 mm Hg). ^1H NMR spectrum of compound **5**, δ, ppm (*J*, Hz): 1.39 (6H, t, *J* = 7.0, CH_3); 3.80 (4H, s, CH_2); 3.87 (4H, q, *J* = 7.0, $2OCH_2$); 4.50 (2H, d, *J* = 2.0, $=CH_2$); 4.56 (2H, d, *J* = 2.0, $=CH_2$); 7.61 (1H, s, N=CH). ^{13}C NMR spectrum, δ, ppm: 14.46 (CH_3); 61.17 (NCH_2); 63.59 (OCH_2); 93.99 ($=CH_2$); 157.76 (=CH–); 159.78 (–CH=). GLC-MS, *m/z* (I_{rel}, %): 223 $[M-1]^+$ (1), 195 (15), 181 (13), 127 (32), 98 (100), 84 (56), 68 (14), 55 (38), 41 (22), 29 (20). Found, %: C 63.92; H 8.92; N 12.39. $C_{12}H_{20}N_2O_2$. Calculated, %: C 64.28; H 8.93; N 12.5.

2-(1'-Ethoxyvinyl)-1-methyl-1,3-imidazolidine (4b). The propenal **1a** (0.95 g, 9.5 mmol) was added to a solution of the N-methyldiaminoethylene **2b** (0.714 g, 9.5 mmol) in $CHCl_3$ (2 ml). The reaction mixture was held for 1 h at 24ºC and the solvent was evaporated *in vacuo* to give compound **4b** (0.604 g, 43%) with bp 45ºC (1 mm Hg). IR spectrum (thin film), ν, cm^{-1}: 1610 and 1650 (C=C), 3100 (sharp), 3320 (broad) (NH). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.33 (3H, t, *J* = 7.0, CH_3); 2.25 (1H, m, CH_2 ring); 2.30 (3H, s, NCH_3); 2.98 (1H, m, CH_2); 3.19 (2H, m, CH_2); 3.30 (1H, s, NH); 3.79 (2H, two q, *J* = 7.0, OCH_2); 4.07 (H, d, *J* = 2.0, $=CH_2$); 4.18 (1H, d, *J* = 2.0, $=CH_2$). GLC-MS, *m/z* (I_{rel}, %): 156 $[M]^+$ (10), 127 (6), 113 (5), 98 (3), 85 $[M-CH_2=COEt]^+$ (100), 84 $[M-CH_2=COEt-H]^+$ (24), 68 (2), 56 (5), 44 (31), 28 (5). Found, %: C 61.31; H 10.22; N 17.88. $C_8H_{16}N_2O$. Calculated, %: C 61.54; H 10.26; N 17.95.

2-(1'-Ethoxyvinyl)-1,3-diphenyl-1,3-imidazolidine (4c). Propenal **1a** (1.08 g, 10.8 mmol) was added to a solution of the diamine **2c** (2.3 g, 10.8 mmol) in EtOH (3 ml). The reaction mixture was held for about

16 h at room temperature. The crystals produced were filtered off and recrystallized twice from ethanol to give compound **4c** (2.74 g, 86.32%) with mp 68.2ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.11 (3H, t, *J* = 7.0, CH_3); 3.53 (2H, q, *J* = 7.0, OCH_2); 3.67 (2H, m, CH_2); 3.73 (2H, m, CH_2); 4.13 (1H, d, *J* = 2.0, =CH_2); 4.3 (1H, d, *J* = 2.0, =CH_2); 5.45 (1H, s, CH); 6.72 (6H, m, *o*-, *p*-H Ph); 7.18 (4H, m, *m*-H Ph). ^{13}C NMR spectrum, δ, ppm: 14.41 (CH_3); 45.79 ((CH_2)$_2$); 62.83 (OCH_2); 75.83 (CH); 86.11 (=CH_2); 113.39 (C_m); 117.46 (C_p); 128.77 (C_o); 144.81 (C_{ipso}); 159.95 (=C–O). Found, %: C 77.73; H 7.62; N 9.47. $C_{19}H_{22}N_2O$. Calculated, %: C 77.51; H 7.53; N 9.52.

2-(1'-Methoxyvinyl)-1,3-diphenyl-1,3-imidazolidine (4d). Propenal **1b** (1.25 g, 6 mmol) was added to a solution of diamine **2c** (0.5 g, 6 mmol) in $CHCl_3$ (2 ml). The reaction mixture was held for about 16 h at room temperature. The crystals produced were filtered and recrystallized twice from $CHCl_3$ to give compound **4d** (1.61 g, 95.8%) with mp 94.8ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.37 (3H, s, OCH_3); 3.66 (2H, m, CH_2); 3.74 (2H, m, CH_2); 4.16 (1H, d, *J* = 2.6, =CH_2); 4.33 (1H, d, *J* = 2.6, =CH_2); 5.45 (1H, d, CH); 6.71 (6H, m, *o*-, *p*-H Ph); 7.18 (4H, m, *m*-H Ph). ^{13}C NMR spectrum, δ, ppm: 45.26 (CH_2); 4.25 (CH_3); 75.32 (CH); 85.33 ((CH_2)$_2$); 112.81 (C_m); 117.01 (C_p); 128.32 (C_o); 144.81 (arom C_{ipso}); 159.95 (=C–O). Found, %: C 77.34; H 7.22; N 10.02. $C_{18}H_{20}N_2O$. Calculated, %: C 77.14; H 7.14; N 10.0.

2-(1'-Ethoxyvinyl)-3-phenyl-1,3-oxazolidine (4e). Propenal **1a** (1.5 g, 15 mmol) was added to a solution of N-phenyl-2-aminoethanol (**2d**) (2.05 g, 15 mmol) in $CHCl_3$ (2 ml). The reaction mixture was held at 24ºC for 24 h. Solvent was evaporated under reduced pressure and the residue was distilled *in vacuo* to give compound **4e** (1.65 g, 66.1%) with bp 120-125ºC (1 mm Hg). With the use of microwave irradiation (1 min×4, 420 watts, 48.6ºC) the yield of the product **4e** was 80%. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.32 (3H, t, *J* = 7.0, CH_3); 3.42 and 3.54 (2H, two m, OCH_2); 3.76 (2H, m, CH_2N); 4.09 and 4.29 (2H, two d, *J* = 2.0, =CH_2); 4.18 (2H, m, CH_2O ring); 5.21 (1H, s, CH); 6.55 (2H, d, *J* = 8.0, *o*-H Ph); 6.72 (1H, t, *J* = 7.6, *p*-H Ph); 7.15 (2H, dd, *J* = 8.0, *J* = 7.6, *m*-H Ph). ^{13}C NMR spectrum, δ, ppm: 14.11 (CH_3); 46.64 (NCH_2); 63.02 (OCH_2); 65.10 (OCH_2 ring); 83.93 (=CH_2); 89.74 (CH); 112.48 (C_m); 117.30 (C_p); 128.64 (C_o); 145.11 (C_{ipso}); 159.04 (=C–O). Found, %: C 71.42; H 7.90; N 6.41. $C_{13}H_{17}NO_2$. Calculated, %: C 71.23; H 7.76; N 6.39.

3-(1'-Methoxyvinyl)-3-phenyl-1,3-oxazolidine (4f). Propenal **1b** (1.217 g, 14 mmol) was added to a solution of aminoethanol **2c** (1.937 g, 14 mmol) in $CHCl_3$ (2 ml). The reaction mixture was held for 24 h at room temperature. Solvent was evaporated under reduced pressure and the residue was distilled *in vacuo* to give compound **4f** (1.794 g, 62.5%) with bp 137ºC (1 mm Hg). Carrying out the reaction in a microwave oven (1 min × 5, 420 watts, 47.6ºC) increased the reaction rate 24 times. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.57 (2H, m, CH_2N); 3.61 (3H, s, OCH_3); 4.18 (3H, m, CH_2O and 1H from =CH_2); 4.34 (1H, d, *J* = 2.3, =CH_2); 5.26 (1H, s, CH); 6.59 (2H, d, *J* = 7.9, *o*-H Ph); 6.70 (1H, t, *J* = 7.3, *p*-H Ph); 7.16 (2H, dd, *J* = 7.3, *J* = 7.9, *m*-H Ph). ^{13}C NMR spectrum, δ, ppm: 47.29 (NCH_2); 55.65 (OCH_3); 65.78 (OCH_2); 84.40 (=CH_2); 90.22 (CH); 113.07 (C_m); 117.92 (C_p); 129.30 (C_o); 145.76 (C_{ipso}); 160.46 (=C–O). Found, %: C 69.96; H 7.42; N 7.05. $C_{12}H_{15}NO_2$. Calculated, %: C 70.24; H 7.32; N 6.83.

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