

Synthesis of Enamino Ketones from *N*-(2-Vinyloxyalkyl)ethane-1,2-diamines

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Abstract—Reactions of *N*-(2-vinyloxyalkyl)ethane-1,2-diamines with 1,3-dicarbonyl compounds gave the corresponding enamino ketones in almost quantitative yield (98–99%).

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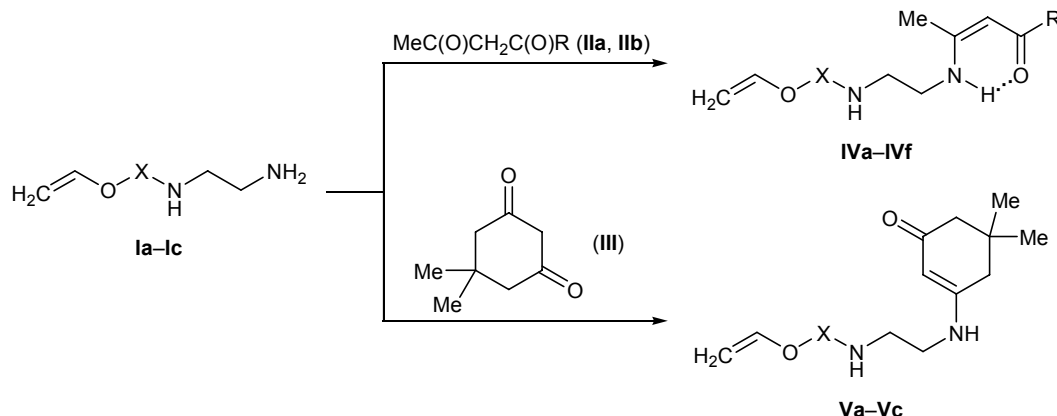
Enaminones are universal and readily accessible synthons for a wide series of heterocyclic compounds; in addition, they exhibit biological activity [1–3]. The reactivity and biological activity of enaminones strongly depend on the nature of the amine fragment. In the present work we were the first to use *N*-(2-vinyloxyalkyl)ethane-1,2-diamines as amine component for the synthesis of enamino ketones. *N*-(2-Vinyloxyalkyl)ethane-1,2-diamines themselves possess broad synthetic potential and diverse biological activity [4], and we anticipated enhancement of these properties in the resulting enaminones.

The condensation of *N*-(2-vinyloxyalkyl)ethane-1,2-diamines **Ia–Ic** with 1,3-dicarbonyl compounds

IIa, IIb, and **III** were performed by heating equimolar mixtures of the reactants in boiling benzene with simultaneous removal of liberated water as azeotrope (Scheme 1). All enaminones **IVa–IVf** and **Va–Vc** were obtained in almost quantitative yield. Enaminones **IVa–IVf** derived from acetylacetone (**IIa**) and ethyl acetoacetate (**IIb**) are viscous oily substances which decompose upon distillation (at a residual pressure of 1–2 mm) but are stable on prolonged storage at room temperature under nitrogen. Enaminones **Va–Vc** derived from 5,5-dimethylcyclohexane-1,3-dione are crystalline substances with sharp melting points.

In the IR spectra of compounds **IVa–IVf** and **Va–Vc** we observed absorption bands in the regions 1500–

Scheme 1.



I, V, X = CH₂CH₂ (**a**), CH₂CH(OH)CH₂ (**b**), CH₂CH₂OCH₂CH(OH)CH₂ (**c**); **II**, R = Me (**a**), OEt (**b**); **IV**, X = CH₂CH₂, R = Me (**a**), OEt (**e**); X = CH₂CH(OH)CH₂, R = Me (**b**), OEt (**d**); X = CH₂CH₂OCH₂CH(OH)CH₂, R = Me (**c**), OEt (**f**).

1585 and 1600–1675 cm^{-1} , which correspond to stretching vibrations of the $\text{N}=\text{C}=\text{CH}-\text{C}=\text{O}$ conjugated bond system; these bands completely overlap those belonging to stretching vibrations of the vinyloxy group (1605–1615 cm^{-1}); stretching vibrations of the $=\text{C}-\text{H}$ bonds appeared in the region 3033–3117 cm^{-1} .

The ^1H NMR spectra of **IVa–IVf** and **Va–Vc** contained only one signal from the olefinic proton in the $\text{NC}=\text{CHC}=\text{O}$ fragment at δ 4.44–4.91 (**IVa–IVf**) and 5.07–5.08 ppm (**Va–Vc**), indicating formation of only one isomer with respect to the double $\text{C}=\text{C}$ bond. Compounds **IVa–IVf** displayed in the ^1H NMR spectra a broadened signal from the $\text{C}=\text{CNH}$ proton involved in intramolecular hydrogen bond (H -chelate ring) at δ 10.81–10.93 (**IVa–IVc**) or 8.62–8.79 ppm (**IVd–IVf**). In the spectra of dimedone derivatives **Va–Vc** the corresponding signal was located in a stronger field, at δ 5.14–5.27 ppm, for fixed *trans*-configuration of the double $\text{C}=\text{C}$ bond makes intramolecular hydrogen bonding $\text{NH}\cdots\text{O}=\text{C}$ impossible. The above data are fully consistent with the chemical shifts of protons in structurally related enamino ketones obtained from 1,3-dicarbonyl compounds and other primary amines [3]. The signal from proton on the nitrogen atom in the $\text{C}=\text{CNHCH}_2$ group of enaminones **IVa–IVf** and **Va–Vc** is slightly broadened, and its intensity corresponds to one proton. Protons in the neighboring methylene group give rise to a multiplet (unresolved doublet of triplets) which looks like a quartet with a coupling constant J of 5.8–6.2 Hz. The intensity of the $\text{C}=\text{CNHCH}_2$ signal indicates the lack of proton exchange with the NH and OH groups under the given conditions.

The fact that the spectra of linear enamino ketones **IVa–IVf** contain only one set of signals corresponding most probably to the *cis-s-trans* isomer may be interpreted in terms of both intramolecular hydrogen bonding $\text{NH}\cdots\text{O}=\text{C}$ and known effect of weakly polar chloroform-*d* used as solvent [1].

EXPERIMENTAL

The IR spectra were recorded on a Bruker Vertex-70 spectrometer from samples prepared as thin films or pelleted with KBr. The ^1H and ^{13}C NMR spectra were measured at 26°C on a Bruker DPX-400 instrument at 400 and 100 MHz, respectively, using CDCl_3 as solvent and hexamethyldisiloxane as internal reference.

Initial 1,3-dicarbonyl compounds **IIa**, **IIb**, and **III** were commercial products. *N*-(2-Vinyloxyethyl)ethane-1,2-diamine (**Ia**) was synthesized according to

the procedure described in [5], and *N*-(2-vinyloxy-alkyl)ethane-1,2-diamines **Ib** and **Ic** were prepared as reported in [6] from ethane-1,2-diamine and vinyl or 2-vinyloxyethyl glycidyl ether.

Enamino ketones IVa–IVf and Va–Vc (general procedure). A mixture of 0.02 mol of *N*-(2-vinyloxy-alkyl)ethane-1,2-diamine **Ia–Ic**, 0.02 mol of diketone **IIa**, **IIb**, or **III** in 100 ml of benzene was heated under reflux in a flask equipped with a Dean–Stark trap until water no longer separated. The solvent was distilled off, and the oily residue was dried at room temperature under reduced pressure (1–2 mm) until constant weight. Some products (dimedone derivatives **Va–Vc**) crystallized.

(E)-4-[2-(2-Vinyloxyethylamino)ethylamino]pent-3-en-2-one (IVa). Yield 98%, $n_D^{20} = 1.5248$. IR spectrum, ν , cm^{-1} : 3285, 3105, 3065, 3030, 2915, 2860, 2825, 1620, 1600, 1555, 1500, 1425, 1360, 1340, 1300, 1280, 1180, 1120, 1060, 1000, 960, 800, 725, 630. ^1H NMR spectrum, δ , ppm: 1.64 br.s (1H, CH_2NHCH_2), 1.87 s (3H, NCCH_3), 1.93 s (3H, $\text{O}=\text{CCH}_3$), 2.78 t (2H, $\text{CH}_2\text{CH}_2\text{NHC}=\text{}$, $^3J = 6.1$ Hz), 2.85 t (2H, $\text{OCH}_2\text{CH}_2\text{NH}$, $^3J = 4.9$ Hz), 3.30 m (2H, $\text{CH}_2\text{NHC}=\text{}$), 3.72 t (2H, OCH_2 , $^3J = 4.9$ Hz), 3.93 d.d (1H, *cis*- $\text{CH}=\text{CO}$, $^2J = 1.5$, $^3J_{\text{cis}} = 6.6$ Hz), 4.12 d.d (1H, *trans*- $\text{CH}=\text{CO}$, $^2J = 1.5$, $^3J_{\text{trans}} = 14.4$ Hz), 4.91 s (1H, $\text{NC}=\text{CH}$), 6.40 d.d (1H, $\text{OCH}=\text{C}$, $^3J_{\text{cis}} = 6.6$, $^3J_{\text{trans}} = 14.4$ Hz), 10.81 br.s (1H, $\text{NHC}=\text{}$). ^{13}C NMR spectrum, δ_C , ppm: 18.95 (NHCCH_3), 28.73 (COCH_3), 43.12 ($\text{CH}_2\text{NHC}=\text{}$), 48.42 ($\text{CH}_2\text{CH}_2\text{NHC}=\text{}$), 49.19 ($\text{OCH}_2\text{CH}_2\text{NH}$), 67.20 (OCH_2), 86.69 ($=\text{CH}_2$), 95.45 ($\text{NC}=\text{CH}$), 151.57 ($\text{OCH}=\text{}$), 162.88 ($\text{NHC}=\text{}$), 194.89 ($\text{C}=\text{O}$). Found, %: C 62.44; H 9.72; N 13.82. $\text{C}_{11}\text{H}_{20}\text{N}_2\text{O}_2$. Calculated, %: C 62.23; H 9.50; N 13.20.

(E)-4-[2-(2-Hydroxy-3-vinyloxypropylamino)ethylamino]pent-3-en-2-one (IVb). Yield 98%, $n_D^{20} = 1.5256$. IR spectrum, ν , cm^{-1} : 3305, 3116, 3079, 2924, 2871, 1615, 1568, 1520, 1440, 1378, 1357, 1318, 1296, 1236, 1200, 1151, 1081, 1017, 1005, 965, 949, 818, 744, 653, 612, 546. ^1H NMR spectrum, δ , ppm: 1.89 s (3H, NCCH_3), 1.94 s (3H, $\text{O}=\text{CCH}_3$), 2.62–2.82 m (6H, CH_2NHCH_2 , OH), 3.31 m (2H, $\text{CH}_2\text{NHC}=\text{}$), 3.66–3.90 m (3H, OCH_2CH), 3.97 d.d (1H, *cis*- $\text{CH}=\text{CO}$, $^2J = 1.7$, $^3J_{\text{cis}} = 6.7$ Hz), 4.16 d.d (1H, *trans*- $\text{CH}=\text{CO}$, $^2J = 1.7$, $^3J_{\text{trans}} = 14.2$ Hz), 4.93 s (1H, $\text{NC}=\text{CH}$), 6.43 d.d (1H, $\text{OCH}=\text{C}$, $^3J_{\text{cis}} = 6.7$, $^3J_{\text{trans}} = 14.2$ Hz), 10.93 br.s (1H, $\text{NHC}=\text{}$). ^{13}C NMR spectrum, δ_C , ppm: 19.05 (NHCCH_3), 28.74 (COCH_3), 42.85 ($\text{CH}_2\text{NHC}=\text{}$), 48.96 ($\text{CH}_2\text{CH}_2\text{NHC}=\text{}$), 51.82 (CHCH_2NH), 68.31 (CHOH), 70.47 (OCH_2), 86.85

(=CH₂), 95.57 (NHC=CH), 151.73 (OCH=), 163.11 (NHC=), 194.85 (C=O). Found, %: C 59.17; H 9.14; N 11.21. C₁₂H₂₂N₂O₃. Calculated, %: C 59.48; H 9.15; N 11.56.

(E)-3-{2-[2-Hydroxy-3-(2-vinyloxyethoxy)propylamino]ethylamino}pent-3-en-2-one (IVc). Yield 99%, $n_D^{20} = 1.5212$. IR spectrum, ν , cm⁻¹: 3310, 3117, 3081, 2922, 2873, 1613, 1574, 1557, 1518, 1454, 1439, 1377, 1357, 1320, 1295, 1201, 1128, 1089, 1036, 1021, 976, 935, 881, 818, 743, 653, 629, 546. ¹H NMR spectrum, δ , ppm: 1.92 s (3H, NHCCH₃), 1.96 s (3H, O=CCH₃), 2.58–2.86 m (6H, CH₂NHCH₂, OH), 3.33 m (2H, CH₂NHC=), 3.51–3.90 m (7H, OCH₂CH₂OCH₂CH), 3.98 d.d (1H, *cis*-CH=CO, ²*J* = 1.7, ³*J*_{cis} = 6.7 Hz), 4.17 d.d (1H, *trans*-CH=CO, ²*J* = 1.7, ³*J*_{trans} = 14.3 Hz), 4.95 s (1H, NHC=CH), 6.45 d.d (1H, OCH=C, ³*J*_{cis} = 6.7, ³*J*_{trans} = 14.3 Hz), 10.89 br.s (1H, NHC=). ¹³C NMR spectrum, δ_C , ppm: 18.61 (NHCCH₃), 28.35 (COCH₃), 42.53 (CH₂NHC=), 48.73 (CH₂CH₂NHC=), 51.68 (CHCH₂NH), 66.94 (=CHOCH₂), 68.47 (CHOH), 69.39 (=CHOCH₂CH₂), 73.77 (OCH₂CH), 86.39 (=CH₂), 95.08 (NHC=CH), 151.37 (OCH=), 162.69 (NHC=), 194.23 (C=O). Found, %: C 58.41; H 9.49; N 10.09. C₁₄H₂₆N₂O₄. Calculated, %: C 58.72; H 9.15; N 9.78.

Ethyl (E)-3-[2-(2-vinyloxyethylamino)ethylamino]but-2-enoate (IVd). Yield 99%, $n_D^{20} = 1.5128$. IR spectrum, ν , cm⁻¹: 3291, 3194, 3117, 3078, 3044, 2978, 2932, 2901, 2873, 2843, 1650, 1607, 1503, 1462, 1444, 1384, 1364, 1320, 1267, 1199, 1174, 1143, 1096, 1058, 1022, 978, 965, 818, 784, 704, 600, 557. ¹H NMR spectrum, δ , ppm: 1.23 t (3H, CH₂CH₃, ³*J* = 7.1 Hz), 1.46 br.s (1H, CH₂NHCH₂), 1.92 s (3H, NHCCH₃), 2.82 t (2H, CH₂CH₂NHC=, ³*J* = 6.1 Hz), 2.90 t (2H, OCH₂CH₂NH, ³*J* = 5.1 Hz), 3.32 m (2H, CH₂NHC=), 3.77 t (2H, OCH₂, ³*J* = 5.1 Hz), 4.00 d.d (1H, *cis*-CH=CO, ²*J* = 1.7, ³*J*_{cis} = 6.7 Hz), 4.07 q (2H, OCH₂CH₃, ³*J* = 7.1 Hz), 4.18 d.d (1H, *trans*-CH=CO, ²*J* = 1.7, ³*J*_{trans} = 14.3 Hz), 4.45 s (1H, NHC=CH), 6.46 d.d (1H, OCH=C, ³*J*_{cis} = 6.7, ³*J*_{trans} = 14.3 Hz), 8.62 br.s (1H, NHC=). ¹³C NMR spectrum, δ_C , ppm: 14.58 (OCH₂CH₃), 19.53 (NHCCH₃), 43.00 (CH₂NHC=), 48.36 (CH₂CH₂NHC=), 49.46 (OCH₂CH₂NH), 58.22 (OCH₂CH₃), 67.15 (=CHOCH₂), 82.55 (NHC=CH), 86.68 (=CH₂), 151.60 (OCH=), 161.62 (NHC=), 170.49 (C=O). Found, %: C 59.87; H 9.39; N 11.48. C₁₂H₂₂N₂O₃. Calculated, %: C 59.48; H 9.15; N 11.56.

Ethyl (E)-3-[2-(2-hydroxy-3-vinyloxypropylamino)ethylamino]but-2-enoate (IVe). Yield 99%,

$n_D^{20} = 1.5208$. IR spectrum, ν , cm⁻¹: 3369, 3299, 3193, 3117, 3081, 2978, 2930, 2903, 2871, 1650, 1606, 1547, 1503, 1458, 1444, 1385, 1364, 1320, 1278, 1201, 1175, 1145, 1095, 1059, 1020, 1006, 979, 965, 873, 821, 785, 705, 601, 557. ¹H NMR spectrum, δ , ppm: 1.23 t (3H, CH₂CH₃, ³*J* = 7.1 Hz), 1.92 s (3H, NHCCH₃), 2.67–2.85 m (6H, CH₂NHCH₂, OH), 3.31 m (2H, CH₂NHC=), 3.72–3.94 m (3H, OCH₂, CHOH), 4.02 d.d (1H, *cis*-CH=CO, ²*J* = 1.5, ³*J*_{cis} = 6.6 Hz), 4.08 q (2H, OCH₂CH₃, ³*J* = 7.1 Hz), 4.22 d.d (1H, *trans*-CH=CO, ²*J* = 1.5, ³*J*_{trans} = 14.2 Hz), 4.46 s (1H, NHC=CH), 6.49 d.d (1H, OCH=C, ³*J*_{cis} = 6.6, ³*J*_{trans} = 14.2 Hz), 8.79 br.s (1H, NHC=). ¹³C NMR spectrum, δ_C , ppm: 14.73 (OCH₂CH₃), 19.65 (NHCCH₃), 42.78 (CH₂NHC=), 49.30 (CH₂CH₂NHC=), 51.67 (CHCH₂NH), 58.47 (OCH₂CH₃), 68.49 (CHOH), 70.35 (OCH₂), 82.78 (NHC=CH), 87.05 (=CH₂), 151.76 (OCH=), 161.74 (NHC=), 170.75 (C=O). Found, %: C 57.47; H 8.57; N 10.60. C₁₃H₂₄N₂O₄. Calculated, %: C 57.33; H 8.88; N 10.29.

Ethyl 3-{2-[2-hydroxy-3-(2-vinyloxyethoxy)propylamino]ethylamino}but-2-enoate (IVf). Yield 99%, $n_D^{20} = 1.5139$. IR spectrum, ν , cm⁻¹: 3480, 3299, 3193, 3117, 3081, 2977, 2929, 2900, 2873, 1648, 1606, 1540, 1504, 1454, 1444, 1385, 1363, 1321, 1277, 1202, 1174, 1133, 1096, 1058, 1022, 978, 966, 820, 784, 705, 666, 606, 558. ¹H NMR spectrum, δ , ppm: 1.18 t (3H, OCH₂CH₃, ³*J* = 7.1 Hz), 1.87 s (3H, NHCCH₃), 2.55–2.73 m (4H, CHCH₂NH, OH), 2.76 t (2H, CH₂CH₂NHC=, ³*J* = 6.0 Hz), 3.26 m (2H, CH₂NHC=), 3.46–3.81 m (7H, OCH₂CH₂OCH₂CH), 4.00 d.d (1H, *cis*-CH=CO, ²*J* = 2.0, ³*J*_{cis} = 6.8 Hz), 4.02 q (2H, OCH₂CH₃, ³*J* = 7.1 Hz), 4.14 d.d (1H, *trans*-CH=CO, ²*J* = 2.0, ³*J*_{trans} = 14.3 Hz), 4.40 s (1H, NHC=CH), 6.43 d.d (1H, OCH=C, ³*J*_{cis} = 6.8, ³*J*_{trans} = 14.3 Hz), 8.70 br.s (1H, NHC=). ¹³C NMR spectrum, δ_C , ppm: 14.63 (OCH₂CH₃), 19.51 (NHCCH₃), 42.77 (CH₂NHC=), 49.36 (CH₂CH₂NHC=), 51.82 (CHCH₂NH), 58.25 (OCH₂CH₃), 67.30 (=CHOCH₂), 69.00 (CHOH), 69.83 (=CHOCH₂CH₂), 74.01 (OCH₂CH), 82.56 (NHC=CH), 86.80 (=CH₂), 151.73 (OCH=), 161.63 (NHC=), 170.53 (C=O). Found, %: C 56.41; H 8.59; N 8.49. C₁₅H₂₈N₂O₅. Calculated, %: C 56.94; H 8.92; N 8.85.

5,5-Dimethyl-3-[2-(2-vinyloxyethylamino)ethylamino]cyclohex-2-en-1-one (Va). Yield 99%, mp 54–56°C. IR spectrum, ν , cm⁻¹: 3221, 3021, 2954, 2937, 2890, 2869, 2840, 1627, 1598, 1567, 1542, 1458, 1451, 1428, 1413, 1383, 1368, 1339, 1331, 1320, 1277, 1255, 1194, 1174, 1155, 1127, 1085, 1073, 1035,

979, 970, 939, 918, 892, 870, 828, 808, 763, 708, 665, 611, 553, 521, 506. ^1H NMR spectrum, δ , ppm: 1.05 s (6H, CH_3), 2.17 s (2H, $\text{CH}_2\text{C}=\text{C}$), 2.19 (2H, $\text{CH}_2\text{C}=\text{O}$), 2.86–2.89 m (5H, $\text{CH}_2\text{NHC}=\text{CH}$), 3.13 m (2H, $\text{CH}_2\text{NHC}=\text{CH}$), 3.77 t (2H, OCH_2 , $^3J = 5.0$ Hz), 4.02 d.d (1H, *cis*- $\text{CH}=\text{CO}$, $^2J = 1.9$, $^3J_{\text{cis}} = 6.7$ Hz), 4.20 d.d (1H, *trans*- $\text{CH}=\text{CO}$, $^2J = 1.9$, $^3J_{\text{trans}} = 14.3$ Hz), 5.08 s (1H, $\text{NHC}=\text{CH}$), 5.14 br.s (1H, $\text{NHC}=\text{CH}$), 6.46 d.d (1H, $\text{OCH}=\text{C}$, $^3J_{\text{cis}} = 6.7$, $^3J_{\text{trans}} = 14.3$ Hz). ^{13}C NMR spectrum, δ_{C} , ppm: 28.38 (CH_3), 32.84 (C^5), 41.94 (C^4), 43.53 ($\text{CH}_2\text{NHC}=\text{CH}$), 47.03 ($\text{CH}_2\text{CH}_2\text{NHC}=\text{CH}$), 48.05 ($\text{OCH}_2\text{CH}_2\text{NH}$), 50.38 (C^6), 67.17 (OCH_2), 87.05 ($=\text{CH}_2$), 95.58 (C^2), 151.61 ($\text{OCH}=\text{C}$), 162.99 (C^3), 196.85 (C^1). Found, %: C 67.04; H 10.02; N 11.15. $\text{C}_{14}\text{H}_{24}\text{N}_2\text{O}_2$. Calculated, %: C 66.63; H 9.59; N 11.10.

3-[2-(2-Hydroxy-3-vinyloxypropylamino)ethylamino]-5,5-dimethylcyclohex-2-en-1-one (Vb). Yield 99%, mp 112–114°C. IR spectrum, ν , cm^{-1} : 3402, 3290, 3256, 3121, 3075, 2955, 2927, 2865, 1650, 1615, 1600, 1574, 1533, 1462, 1448, 1425, 1413, 1380, 1362, 1341, 1315, 1270, 1251, 1196, 1173, 1152, 1128, 1096, 1046, 1002, 981, 965, 931, 900, 890, 834, 819, 798, 745, 680, 665, 609, 547, 526. ^1H NMR spectrum, δ , ppm: 1.05 s (6H, CH_3), 2.16 s (2H, $\text{CH}_2\text{C}=\text{C}$), 2.19 s (2H, $\text{CH}_2\text{C}=\text{O}$), 2.66–2.82 m (4H, $\text{NHCH}_2\text{CH}_2\text{NHC}=\text{CH}$, OH), 2.87 t (2H, CHCH_2NH , $^3J = 5.6$ Hz), 3.16 m (2H, $\text{CH}_2\text{NHC}=\text{CH}$), 3.65–3.72 m (2H, OCH_2), 3.97 m (1H, CHOH), 4.03 d.d (1H, *cis*- $\text{CH}=\text{CO}$, $^2J = 1.8$, $^3J_{\text{cis}} = 6.7$ Hz), 4.20 d.d (1H, *trans*- $\text{CH}=\text{CO}$, $^2J = 1.8$, $^3J_{\text{trans}} = 14.2$ Hz), 5.08 s (1H, $\text{NHC}=\text{CH}$), 5.39 br.s (1H, $\text{NHC}=\text{CH}$), 6.47 d.d (1H, $\text{OCH}=\text{C}$, $^3J_{\text{cis}} = 6.7$, $^3J_{\text{trans}} = 14.2$ Hz). ^{13}C NMR spectrum, δ_{C} , ppm: 28.37 (CH_3), 32.85 (C^5), 42.18 (C^4), 43.55 ($\text{CH}_2\text{NHC}=\text{CH}$), 47.33 ($\text{CH}_2\text{CH}_2\text{NHC}=\text{CH}$), 50.33 (C^6), 51.56 (CHCH_2NH), 68.54 (CHOH), 70.35 (OCH_2), 87.24 ($=\text{CH}_2$), 95.59 (C^2), 151.59 ($\text{OCH}=\text{C}$), 163.11 (C^3), 196.95 (C^1). Found, %: C 63.41; H 9.49; N 9.59. $\text{C}_{15}\text{H}_{26}\text{N}_2\text{O}_3$. Calculated, %: C 63.80; H 9.28; N 9.92.

3-{2-[2-Hydroxy-3-(2-vinyloxyethoxy)propylamino]ethylamino}-5,5-dimethylcyclohex-2-en-1-one (Vc). Yield 99%, mp 62–64°C. IR spectrum, ν , cm^{-1} : 3359, 3119, 3080, 2954, 2926, 2867, 1635, 1618,

1599, 1574, 1536, 1465, 1425, 1413, 1379, 1363, 1321, 1271, 1251, 1202, 1174, 1153, 1127, 1041, 966, 949, 933, 891, 872, 834, 816, 786, 682, 608, 548, 530, 475, 440. ^1H NMR spectrum, δ , ppm: 1.04 s (6H, CH_3), 2.16 s (2H, $\text{CH}_2\text{C}=\text{C}$), 2.17 s (2H, $\text{CH}_2\text{C}=\text{O}$), 2.61–2.81 m (4H, $\text{NHCH}_2\text{CH}_2\text{NHC}=\text{CH}$, OH), 2.85 t (2H, CHCH_2NH , $^3J = 5.8$ Hz), 3.13 m (2H, $\text{CH}_2\text{NHC}=\text{CH}$), 3.43–3.56 m (2H, OCH_2CH), 3.70–3.75 m (2H, $=\text{CHOCH}_2\text{CH}_2$), 3.83 t (2H, $=\text{CHOCH}_2$, $^3J = 4.4$ Hz), 3.90 m (1H, CHOH), 4.02 d.d (1H, *cis*- $\text{CH}=\text{CO}$, $^2J = 2.1$, $^3J_{\text{cis}} = 6.8$ Hz), 4.19 d.d (1H, *trans*- $\text{CH}=\text{CO}$, $^2J = 2.1$, $^3J_{\text{trans}} = 14.3$ Hz), 5.07 s (1H, $\text{NHC}=\text{CH}$), 5.27 br.s (1H, $\text{NHC}=\text{CH}$), 6.46 d.d (1H, $\text{OCH}=\text{C}$, $^3J_{\text{cis}} = 6.8$, $^3J_{\text{trans}} = 14.3$ Hz). ^{13}C NMR spectrum, δ_{C} , ppm: 28.33 (CH_3), 32.77 (C^5), 42.13 (C^4), 43.43 ($\text{CH}_2\text{NHC}=\text{CH}$), 47.27 ($\text{CH}_2\text{CH}_2\text{NHC}=\text{CH}$), 50.31 (NHCH_2CH), 51.66 (C^6), 67.30 ($=\text{CHOCH}_2$), 69.02 (CHOH), 69.88 ($=\text{CHOCH}_2\text{CH}_2$), 74.07 (OCH_2CH), 87.03 ($=\text{CH}_2$), 95.43 (C^2), 151.64 ($\text{OCH}=\text{C}$), 163.13 (C^3), 196.79 (C^1). Found, %: C 62.41; H 9.49; N 8.19. $\text{C}_{17}\text{H}_{30}\text{N}_2\text{O}_4$. Calculated, %: C 62.55; H 9.26; N 8.58.

REFERENCES

1. *The Chemistry of Enamines*, Rappoport, Z., Ed., New York: Wiley, 1994, vol. 1.
2. Foster, J.E., Nicholson, J.M., Butcher, R., Stables, J.P., Edafiogho, I.O., Goodwin, A.M., Henson, M.C., Smith, C.A., and Scott, K.R., *Bioorg. Med. Chem.*, 1999, vol. 7, p. 2415.
3. Freimanis, Ya.F., *Khimiya enaminoketonov, enaminoiminov, enaminoionov* (Chemistry of Enamino Ketones, Enamino Imines, and Enamino Thiones), Riga: Zinatne, 1974.
4. Trofimov, B.A., *Geteroatomnye proizvodnye atsetilena* (Heteroatom Acetylene Derivatives), Moscow: Nauka, 1981, p. 38; Kukharev, B.F., Stankevich, V.K., and Klimenko, G.R., *Usp. Khim.*, 1995, vol. 64, p. 562.
5. Kukharev, B.F., Stankevich, V.K., Lobanova, N.A., Stankevich, V.V., and Kukhareva, V.A., *Russ. J. Org. Chem.*, 2007, vol. 43, p. 1880.
6. Kukharev, B.F., Stankevich, V.K., Lobanova, N.A., Klimenko, G.R., and Sadykov, E.Kh., *Russ. J. Org. Chem.*, 2006, vol. 42, p. 1409.