

Synthesis and Some Transformations of 2-Alkenyl-4-alkoxymethylbutanolides

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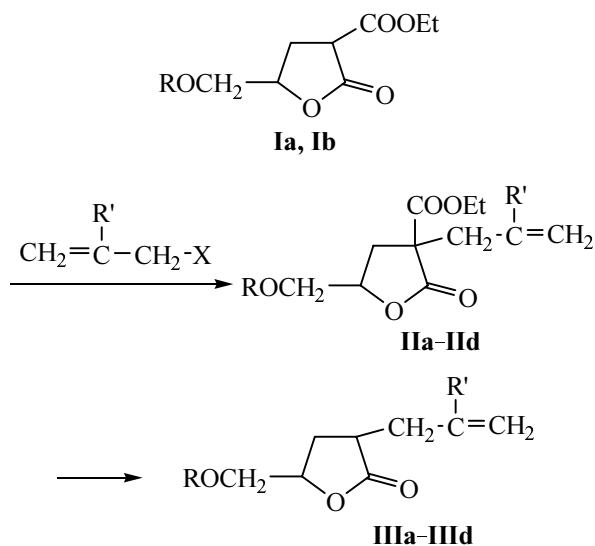
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Abstract—The alkylation of 4-alkoxymethyl-2-ethoxycarbonylbutanolides led to the formation of 2-alkenyl-4-alkoxymethyl-2-ethoxycarbonylbutanolides which at alkaline hydrolysis provided new 2-alkenyl-4-alkoxymethylbutanolides. By oxidation of the latter with a mixture of hydrogen peroxide and formic acid 4-alkoxymethyl-2-(2-R-2,3-dihydroxy)propylbutanolides were obtained. The diolactones were shown to suffer under the conditions of the pinacol-pinacolone rearrangement a conversion into previously unknown formyl- and epoxy lactones.

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We formerly demonstrated that 4-alkoxymethyl-2-ethoxycarbonylbutanolides **Ia** and **Ib** were active CH-acids, and they were well suitable for application to the fine organic synthesis for preparation of various lactone-containing compounds [1–4]. In extension of research in the field of saturated γ -lactones and aiming at the preparation of new functionally-substituted butanolides we carried out an alkylation of reagents **Ia** and **Ib** with alkenyl halides in the presence of an equimolar amount of sodium ethylate in the anhydrous ethanol solution. As a result 2-alkenyl-4-alkoxymethyl-2-ethoxycarbonylbutanolides **IIa–IIId**

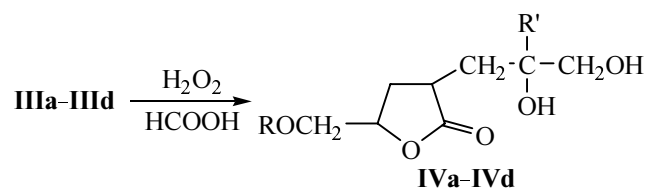


I, R = Pr (**a**), Am (**b**); R' = Me, X = Cl; R' = H, X = Br; **II**, **III**, R = Pr, R' = H (**a**), Me (**c**); R = Am, R' = H (**b**), Me (**d**).

IIId were obtained in a high yield (75–90%). The alkaline hydrolysis and decarboxylation of the latter led to the formation of 2-alkenyl-4-alkoxy-methylbutanolides **IIIa–IIIId**.

The chemistry of α -glycols is sufficiently well understood, but the studies dedicated to heterocyclic α -glycols, in particular, to lactone-containing diols are scarce [5–9].

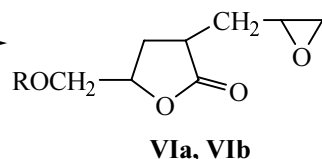
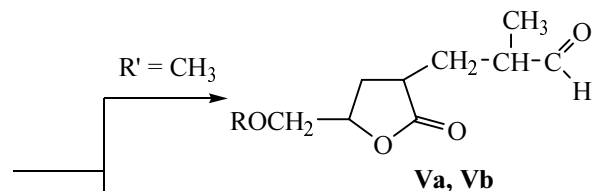
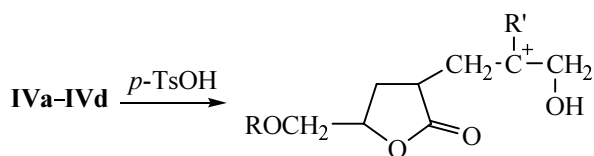
To synthesize diolactones we attempted an oxidation of reagents **IIIa–IIIId** with 30% hydrogen peroxide in the presence of 85% formic acid. Thus we obtained 4-alkoxymethyl-2-(2-R-2,3-dihydroxy)propylbutanolides **IVa–IVd**.



IV, R = Pr, R' = H (**a**), Me (**c**); R = Am, R' = H (**b**), Me (**d**).

Some reaction of the latter were performed which, on the one hand, confirm the structure of compounds **IVa–IVd**, and on the other hand, produce new classes of lactone-containing compounds previously unmentioned in the literature.

We investigated the behavior of compounds **IVa–IVd** under the conditions of the pinacol-pinacolone rearrangement. It was established that in the presence of the

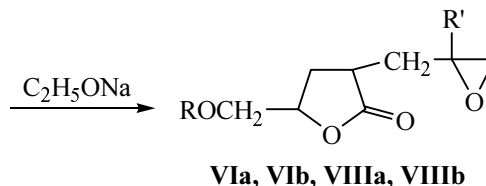
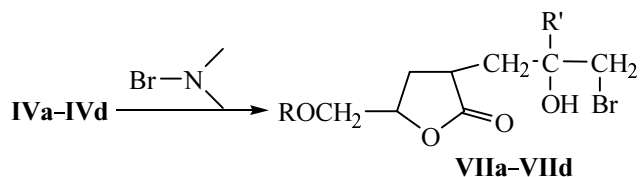


V, VI, R = Pr (a), Am (b).

p-toluenesulfonic acid the primary-tertiary diololactones **IVc** and **IVd** readily underwent the pinacol-pinacolone rearrangement to give the corresponding aldehydo-lactones. The reaction occurred regioselectively leading to the formation in a high yield of 2-alkoxymethyl-2-(2-methyl-2-formyl)ethylbutanolides **Va** and **Vb**. The reaction of the primary-secondary diololactones **IVa** and **IVb** resulted in the formation of epoxy-lactones, 4-alkoxymethyl-2-(2,3-epoxy)propylbutanolides **VIa** and **VIb**.

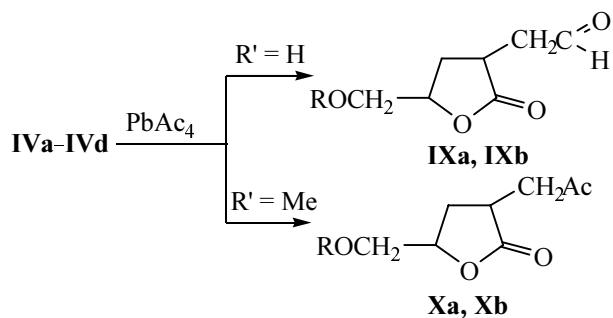
The different paths taken by the reaction may be ascribed to the difference in the stability of the carbocation site generated by the action of the *p*-toluenesulfonic acid. Presumably in the case of the less stable and consequently more reactive secondary carbocation the probability of a hydride shift sharply decreases and as sharply increases the possibility of an attack by the unshared electron pair of the oxygen in the neighboring hydroxy group; as a result form epoxy-lactones **VIa** and **VIb**. The use of a stronger acid, like sulfuric acid, caused the tarring of the final products.

In order to develop a general procedure for epoxy-lactones preparation and to prove the structure of compounds **VIa** and **VIb** the diololactones **IVa-IVd** were converted into the corresponding bromohydrines by treating with N-bromosuccinimide. The reaction should be carried out in aqueous-acetone medium for 4 h. Under these conditions 2-alkoxymethyl-2-(3-bromo-2-hydroxy)propylbutanolides **VIIa-VIIId** were obtained in high yields. The debromination of these bromohydrines with sodium alcoholate resulted in the target epoxides **VIa, VIb** and **VIIIa, VIIIb**.



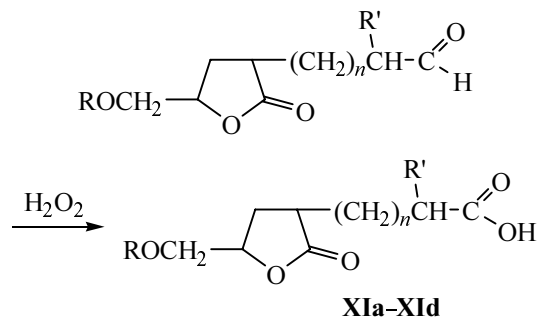
VII, R = Pr, R' = Me (a), H (c); R = Am, R' = Me (b), H (d);
VIII, R = Pr, R' = Me (a); R = Am, R' = Me (b).

Reagents **IVa-IVd** were easily oxidized with lead tetraacetate giving the corresponding carbonyl compounds. At R' = H formyl-lactones obtained, 4-alkoxymethyl-2-formylmethylbutanolides **IXa** and **IXb**, were prepared for the first time, whereas at R' = Me the oxidation led to the formation of 4-alkoxymethyl-2-acetylbutanolides **Xa** and **Xb** identical to the ketolactones described in [10].



IX, X: R = Pr (a), Am (b).

Carboxyl-lactones are known to be good synthons for preparation of lactone-containing thioureas and various classes of heterocycles [11-13]. Planning to obtain carb-



XI, R = Pr, R' = Me (a), H (c); R = Am, R' = Me (b), H (d);
 $n = 1$ (a, b), 0 (c, d).

oxylactones of new structure and at the same time to prove the structure of compounds **Va**, **Vb** and **IXa**, **IXb** the latter were subjected to oxidation under conditions of the Baeyer–Villiger reaction. The reaction was performed with the use of a mixture of 30% aqueous hydrogen peroxide and 85% formic acid. The 4-alkoxymethyl-2-carboxyalkylbutanolides **XIa–XIId** were obtained in high yields. The compounds were quantitatively titrated with 1 equiv of 0.1 N NaOH in the cold and with 2 equiv at heating.

EXPERIMENTAL

IR spectra of compounds **II–XI** were recorded from thin films on a spectrophotometer Nicolet FTIR Nexus. TLC was carried out on Silufol UV-254 plates, eluent ethanol–benzene–hexane, 3:3:10, for compounds **IVa–IVd**, 2:1:1, development in iodine vapor.

Initial 4-alkoxymethyl-2-ethoxycarbonylbutanolides were prepared as described in [14].

2-Alkenyl-4-alkoxymethyl-2-ethoxycarbonylbutanolides IIa–IIId. To a solution obtained by dissolving 2.3 g (0.1 mol) of metal sodium in 20 ml of anhydrous ethanol was added dropwise 0.1 mol of an appropriate 4-alkoxymethyl-2-ethoxycarbonylbutanolide. The mixture was stirred for 15 min and then 0.11 mol of alkenyl halide was added dropwise. The reaction mixture was stirred for 2 h without heating and 4 h at 75–80°C. On distilling off the ethanol the residue was cooled, and water was added acidified with HCl to pH 2–3. The products were extracted into ether, the extracts were washed with water and dried with anhydrous magnesium sulfate. On removing the solvent the residue was subjected to distillation.

2-Allyl-4-propoxymethyl-2-ethoxycarbonylbutanolide (IIa). Yield 78%, bp 121–122°C (1 mm Hg), R_f 0.49, n_D^{20} 1.4585, d_4^{20} 1.0639. Found, %: C 62.40; H 8.00. $C_{14}H_{22}O_5$. Calculated, %: C 62.22; H 8.15.

2-Allyl-4-amyloxymethyl-2-ethoxycarbonylbutanolide (IIb). Yield 90%, bp 133–134°C (1 mm Hg), R_f 0.54, n_D^{20} 1.4590, d_4^{20} 1.0357. Found, %: C 64.55; H 8.60. $C_{16}H_{26}O_5$. Calculated, %: C 64.43; H 8.72.

2-Methyl-2-propenyl-4-propoxymethyl-2-ethoxycarbonylbutanolide (IIc). Yield 81%, bp 133°C (2 mm Hg), R_f 0.46, n_D^{20} 1.4600, d_4^{20} 1.0644. Found, %: C 63.20; H 8.60. $C_{15}H_{24}O_5$. Calculated, %: C 63.38; H 8.45.

2-Methyl-2-propenyl-4-amyloxymethyl-2-ethoxycarbonylbutanolide (IIId). Yield 75%, bp 150°C (2 mm Hg), R_f 0.50, n_D^{20} 1.4595, d_4^{20} 1.0314. Found, %: C 65.25; H 9.00. $C_{17}H_{28}O_5$. Calculated, %: C 65.38; H 8.97.

In the IR spectra of compounds **IIa–IIId** the following absorption bands were observed (cm^{-1}): 1760 (C=O lactone), 1725 (C=O ester), 1640 (C=C), 1190, 1210 (COC), 3080 ($=CH_2$).

2-Alkenyl-4-alkoxymethylbutanolides IIIa–IIIId. To a solution in 16 ml of water of 7 g (0.175 mol) of sodium hydroxide and 0.5 ml of Katamine AB was added dropwise 0.05 mol of reagent **IIa–IIId**, the mixture was stirred for 1 h at 20–25°C and 2 h at 55–60°C. On cooling the mixture was acidified with concn. HCl to pH 2–3, extracted with ether, the extracts were washed with water and dried with anhydrous magnesium sulfate. On removing the solvent the residue was subjected to distillation.

2-Allyl-4-propoxymethylbutanolide (IIIa). Yield 87%, bp 92–93°C (2 mm Hg), R_f 0.47, n_D^{20} 1.4533, d_4^{20} 1.0022. Found, %: C 66.80; H 8.95. $C_{11}H_{18}O_3$. Calculated, %: C 66.67; H 9.09.

2-Allyl-4-amyloxymethylbutanolide (IIIb). Yield 92%, bp 110–112°C (2 mm Hg), R_f 0.49, n_D^{20} 1.4580, d_4^{20} 0.9782. Found, %: C 68.95; H 9.85. $C_{13}H_{22}O_3$. Calculated, %: C 69.03; H 9.74.

2-Methyl-2-propenyl-4-propoxymethylbutanolide (IIIc). Yield 85%, bp 100°C (2 mm Hg), R_f 0.44, n_D^{20} 1.4590, d_4^{20} 0.9976. Found, %: C 68.05; H 9.30. $C_{12}H_{20}O_3$. Calculated, %: C 67.92; H 9.43.

2-Methyl-2-propenyl-4-amyloxymethylbutanolide (IIId). Yield 90%, bp 111–112°C (1 mm Hg), R_f 0.48, n_D^{20} 1.4585, d_4^{20} 0.9744. Found, %: C 70.15; H 9.90. $C_{14}H_{24}O_3$. Calculated, %: C 70.00; H 10.00.

In the IR spectra of compounds **IIIa–IIIId** the following absorption bands were observed (cm^{-1}): 1765 (C=O lactone), 1640 (C=C), 1190, 1210 (COC), 3080 ($=CH_2$).

4-Alkoxymethyl-2-(2-R-2,3-dihydroxy)propylbutanolides IVa–IVd. A mixture of 0.53 mol of reagent **IIIa–IIIId**, 2.9 ml (0.063 mol) of 85% formic acid, and 8.7 ml of 30% hydrogen peroxide was stirred for 3 h at 20–25°C and 17 h at 55–60°C. Water and formic acid were removed at a reduced pressure (15–20 mm Hg), and the residue was distilled in a vacuum.

2-(2,3-Dihydroxy)propyl-4-propoxymethylbutanolide (IVa). Yield 77%, bp 195–196°C (2 mm Hg), R_f 0.48, n_D^{20} 1.4760, d_4^{20} 1.1489. Found, %: C 56.55; H 8.75. $C_{11}H_{20}O_5$. Calculated, %: C 56.69; H 8.62.

4-Amyloxymethyl-2-(2,3-dihydroxy)propylbutanolide (IVb). Yield 83%, bp 211–212°C (2 mm Hg), R_f 0.50, n_D^{20} 1.4750, d_4^{20} 1.1091. Found, %: C 60.15; H 9.10. $C_{13}H_{24}O_5$. Calculated, %: C 60.00; H 9.23.

2-(2-Methyl-2,3-dihydroxy)propyl-4-propoxymethylbutanolide (IVc). Yield 72%, bp 200–201°C (2 mm Hg), R_f 0.51, n_D^{20} 1.4765, d_4^{20} 1.1182. Found, %: C 58.65; H 8.80. $C_{12}H_{22}O_5$. Calculated, %: C 58.54; H 8.94.

4-Amyloxymethyl-2-(2-methyl-2,3-dihydroxy)propylbutanolide (IVd). Yield 74%, bp 199–200°C (1 mm Hg), R_f 0.53, n_D^{20} 1.4710, d_4^{20} 1.0825. Found, %: C 60.30; H 9.50. $C_{14}H_{26}O_5$. Calculated, %: C 60.43; H 9.35.

In the IR spectra of compounds **IV** the following absorption bands were observed (cm^{-1}): 1760 (C=O lactone), 1145, 1190 (COC), 3415 (OH associated).

2-(2-Methyl-2-formyl)ethyl-4-propoxymethylbutanolide (Va). A mixture of 12.3 g (0.05 mol) of compound **IVc**, 0.5 g of *p*-toluenesulfonic acid, and 100 ml of benzene was boiled with a Dean–Stark trap till the end of water liberation (4 h), then the mixture was cooled, washed with water, and dried with anhydrous magnesium sulfate. On removing the solvent the residue was subjected to distillation. Yield 8.3 g (73%), bp 141°C (1 mm Hg), R_f 0.48, n_D^{20} 1.4610, d_4^{20} 1.0597. Found, %: C 63.25; H 8.65. $C_{12}H_{20}O_4$. Calculated, %: C 63.16; H 8.77. Thiosemicarbazone: mp 101–102°C.

Likewise was obtained **4-amyloxymethyl-2-(2-methyl-2-formyl)ethylbutanolide (Vb)**. Yield 78%, bp 160–161°C (1 mm Hg), R_f 0.45, n_D^{20} 1.4615, d_4^{20} 1.0319. Found, %: C 65.75; H 9.15. $C_{14}H_{24}O_4$. Calculated, %: C 63.63; H 8.77. Thiosemicarbazone: mp 112–113°C.

In the IR spectra of compounds **Va** and **Vb** the following absorption bands were observed (cm^{-1}): 1770 (C=O lactone), 1725 (C=O aldehyde), 2735 (C–H aldehyde), 1130, 1180 (COC).

4-Propoxymethyl-2-(2,3-epoxypropyl)butanolide (VIa) was similarly prepared from 9.3 g (0.04 mol) 4-propoxymethyl-2-(2,3-dihydroxy)propylbutanolide, 0.4 g of *p*-toluenesulfonic acid, and 80 ml of benzene. Yield 6.3 g (69%), bp 139–140°C (1 mm Hg), R_f 0.49, n_D^{20} 1.4600, d_4^{20} 1.0589. Found, %: C 65.75; H 9.15. $C_{12}H_{20}O_4$. Calculated, %: C 63.16; H 8.77.

Likewise was obtained **4-amyloxymethyl-2-(2,3-epoxypropyl)butanolide (VIb)**. Yield 69%, bp 139–140°C (1 mm Hg), R_f 0.49, n_D^{20} 1.4600, d_4^{20} 1.0589. Found,

%: C 65.75; H 9.15. $C_{12}H_{20}O_4$. Calculated, %: C 63.16; H 8.77.

4-Alkoxymethyl-2-(2-R-2-hydroxy-3-bromo)propylbutanolides VIIa–VIIId. A mixture of 0.05 mol of an appropriate 4-alkoxymethyl-2-substituted butanolide, 13.4 g (0.075 mol) of *N*-bromosuccinimide, 10 ml of acetone, and 15 ml of water was stirred for 1 h at 20–25°C and 4 h at 55–60°C. Acetone was distilled off, the residue was cooled, extracted with ether, the extracts were washed with water and dried with anhydrous magnesium sulfate. On removing the solvent the residue was subjected to distillation.

2-(3-Bromo-2-hydroxy-2-methylpropyl)-4-propoxymethylbutanolide (VIIa). Yield 68%, bp 163–165°C (1 mm Hg), R_f 0.51, n_D^{20} 1.4920, d_4^{20} 1.3222. Found, %: C 46.70; H 6.70; Br 26.00. $C_{12}H_{21}O_4Br$. Calculated, %: C 46.60; H 6.80; Br 25.89.

4-Amyloxymethyl-2-(3-bromo-2-hydroxy-2-methylpropyl)butanolide (VIIb). Yield 65%, bp 175–176°C (1 mm Hg), R_f 0.50, n_D^{20} 1.4935, d_4^{20} 1.2734. Found, %: C 50.00; H 7.30; Br 23.65. $C_{14}H_{25}O_4Br$. Calculated, %: C 49.85; H 7.42; Br 23.74.

2-(3-Bromo-2-hydroxypropyl)-4-propoxymethylbutanolide (VIIc). Yield 70%, bp 154–155°C (1 mm Hg), R_f 0.52, n_D^{20} 1.4910, d_4^{20} 1.3468. Found, %: C 44.85; H 6.30; Br 27.00. $C_{11}H_{19}O_4Br$. Calculated, %: C 44.75; H 6.44; Br 27.12.

4-Amyloxymethyl-2-(3-bromo-2-hydroxypropyl)butanolide (VIIId). Yield 67%, bp 168–169°C (1 mm Hg), R_f 0.49, n_D^{20} 1.4925, d_4^{20} 1.2905. Found, %: C 48.40; H 7.05; Br 24.15. $C_{13}H_{23}O_4Br$. Calculated, %: C 48.30; H 7.12; Br 24.77.

In the IR spectra of compounds **VIIa–VIIId** the following absorption bands were observed (cm^{-1}): 1765 (C=O lactone), 1145, 1190 (COC), 3400 (OH), 920 (C–Br).

4-Alkoxymethyl-2-(2-R-2,3-epoxy)propylbutanolides VIa, VIb, VIIa, and VIIb. To sodium ethylate prepared from 0.5 g (0.02 mol) of metal sodium and 8.5 ml of anhydrous EtOH was added 10 ml of anhydrous benzene and then dropwise 0.02 mol of bromohydrin **VIIa–VIIId** in 50 ml of anhydrous benzene. The mixture was stirred for 10 h at 20–25°C and 1 h at 50–55°C. On cooling water acidified with HCl was added to the reaction mixture, the products were extracted into benzene, the extracts were washed with water and dried with anhydrous magnesium sulfate. On removing the solvent the residue was subjected to distillation.

In the IR spectra of compounds **VI** and **VIII** the following absorption bands were observed (cm^{-1}): 1780 ($\text{C}=\text{O}$ lactone), 1130, 1180 (COC).

2-(2,3-Epoxypropyl)-4-propoxymethylbutanolide (VIa). Yield 69%, bp 135–136°C (1 mm Hg), R_f 0.54, n_D^{20} 1.4760, d_4^{20} 1.1263. Found, %: C 67.20; H 8.55. $\text{C}_{11}\text{H}_{18}\text{O}_4$. Calculated, %: C 67.29; H 8.41.

4-Amyloxymethyl-2-(2,3-epoxypropyl)butanolide (VIb). Yield 73%, bp 147°C (1 mm Hg), R_f 0.53, n_D^{20} 1.4765, d_4^{20} 1.0859. Found, %: C 64.55; H 8.95. $\text{C}_{13}\text{H}_{22}\text{O}_4$. Calculated, %: C 64.46; H 9.09.

2-(2-Methyl-2,3-epoxypropyl)-4-propoxymethylbutanolide (VIIIa). Yield 77%, bp 148–149°C (1 mm Hg), R_f 0.56, n_D^{20} 1.4765, d_4^{20} 1.1102. Found, %: C 63.30; H 8.90. $\text{C}_{12}\text{H}_{20}\text{O}_4$. Calculated, %: C 63.16; H 8.77.

4-Amyloxymethyl-2-(2-methyl-2,3-epoxypropyl)butanolide (VIIIb). Yield 71%, bp 166–167°C (1 mm Hg), R_f 0.55, n_D^{20} 1.4770, d_4^{20} 1.0784. Found, %: C 65.80; H 9.65. $\text{C}_{14}\text{H}_{24}\text{O}_4$. Calculated, %: C 65.63; H 9.38.

4-Propoxymethyl-2-formylmethylbutanolide (IXa). To a solution of 9.3 g (0.04 mol) of compound **IVa** in 100 ml of anhydrous benzene was added by portions 17.7 g (0.04 mol) of lead tetraacetate, the mixture was stirred for 4 h, filtered, and on removal of the solvent the residue was distilled. Yield 5.4 g (67%), bp 129–130°C (1 mm Hg), R_f 0.51, n_D^{20} 1.4595, d_4^{20} 1.1012. Found, %: C 60.10; H 7.90. $\text{C}_{10}\text{H}_{16}\text{O}_4$. Calculated, %: C 60.00; H 8.00. Thiosemicarbazone: mp 85–86°C.

Likewise was obtained **4-amylloxymethyl-2-formylmethylbutanolide (IXb)**. Yield 69%, bp 139–140°C (1 mm Hg), R_f 0.49, n_D^{20} 1.4600, d_4^{20} 1.0589. Found, %: C 65.75; H 9.15. $\text{C}_{12}\text{H}_{20}\text{O}_4$. Calculated, %: C 63.16; H 8.77. Thiosemicarbazone: mp 81–82°C.

In the IR spectra of compounds **IXa** and **IXb** the following absorption bands were observed (cm^{-1}): 1780 ($\text{C}=\text{O}$ lactone), 1730 ($\text{C}=\text{O}$ aldehyde), 2750 ($\text{C}-\text{H}$ aldehyde), 1130, 1180 (COC).

2-Acetyl-4-propoxymethylbutanolide (Xa) was prepared analogously from 11.6 g (0.05 mol) of compound **IVc**, and 22.2 g (0.05 mol) of lead tetraacetate. Yield 8 g (75%), bp 135°C (2 mm Hg), R_f 0.48, n_D^{20} 1.4585 [10].

Likewise was obtained **4-amylloxymethyl-2-acetomethylbutanolide (Xb)**. Yield 82%, bp 129–130°C (1 mm Hg), R_f 0.53, n_D^{20} 1.4565 [10].

3-(5-Alkoxymethyl-2-oxotetrahydro-3-furyl)propanoic acids XIa–XIId. To a mixture of 0.05 mol of aldehydolactone **Va**, **Vb**, or **IXa**, **IXb** and 25 ml of 85%

formic acid was added dropwise at stirring 7.5 ml of 30% hydrogen peroxide solution. The mixture was stirred for 4 h at 20–25°C and 8 h at 50–55°C. Water and formic acid were removed at a reduced pressure (15–20 mm Hg), and the residue was distilled in a vacuum.

2-Methyl-3-(2-oxo-3-propoxymethyltetrahydro-3-furyl)propanoic acid (XIa). Yield 76%, bp 181–182°C (1 mm Hg), n_D^{20} 1.4615, d_4^{20} 1.1103. Found, %: C 59.20; H 8.35. $\text{C}_{12}\text{H}_{20}\text{O}_5$. Calculated, %: C 59.02; H 8.20.

3-(5-Amyloxymethyl-2-oxotetrahydro-3-furyl)-2-methylpropanoic acid (XIb). Yield 72%, bp 195°C (1 mm Hg), n_D^{20} 1.4630, d_4^{20} 1.0793. Found, %: C 61.85; H 9.00. $\text{C}_{14}\text{H}_{24}\text{O}_5$. Calculated, %: C 61.76; H 9.82.

2-(2-Oxo-5-propoxymethyltetrahydro-3-furyl)propanoic acid (XIc). Yield 80%, bp 166°C (1 mm Hg), n_D^{20} 1.4660, d_4^{20} 1.1683. Found, %: C 55.70; H 7.30. $\text{C}_{10}\text{H}_{16}\text{O}_5$. Calculated, %: C 55.55; H 7.41.

2-(5-Amyloxymethyl-2-oxotetrahydro-3-furyl)propanoic acid (XIId). Yield 81%, bp 178°C (1 mm Hg), n_D^{20} 1.4655, d_4^{20} 1.1129. Found, %: C 59.00; H 8.10. $\text{C}_{12}\text{H}_{20}\text{O}_5$. Calculated, %: C 59.02; H 8.20.

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