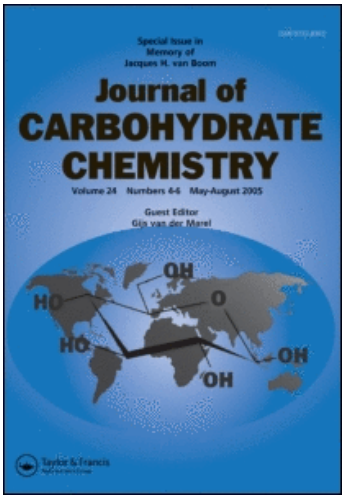

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CONJUGATE ADDITION OF HYDROXYLAMINES TO 4-SUBSTITUTED BUTENOLIDES

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ABSTRACT

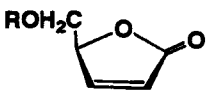
4-Substituted-butenolides treated with free hydroxylamine at pH=5 undergo conjugate addition - rearrangement to afford isoxazolidin-5-ones. This is in contrast to N-substituted hydroxylamines which under the same conditions produce Michael adducts only. In the presence of base, butenolides undergo racemization *via* flat tautomeric forms.

INTRODUCTION

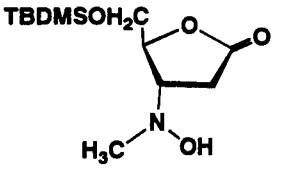
(*S*)-5-Hydroxymethyl-5H-furan-2-one (**1**, 4-hydroxymethylbutenolide) and its *O*-substituted derivatives represent valuable chiral starting materials for the synthesis of a variety of natural products.[1] 4-Substituted-butenolides have been obtained by several methods.[2-4]

Recently two groups have reported on conjugate addition of *N*-methylhydroxylamine to butenolide **2**.[4,5] Reaction proceeded with high stereoselectivity to afford *trans* adduct **3** which could not be rearranged to the corresponding isoxazolidin-5-one **4** even under mild basic conditions.[4] This observation has been found to be in contrast to a similar hydroxylamine addition to α,β-unsaturated δ-lactones **5** which proceeds smoothly to yield isoxazolidin-5-ones **7** *via* the addition - rearrangement pathway (Scheme

1).[6] It should be noted that the formation of isoxazolidin-5-ones, *via* Michael addition of hydroxylamines to unsaturated esters, followed by intramolecular cyclization of the adduct is a known process; the second step requires, however, a basic catalyst and usually heating.[7]



1: R = H
2: R = TBDMS



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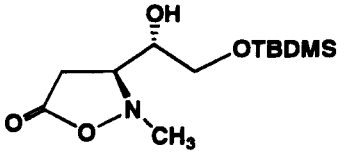
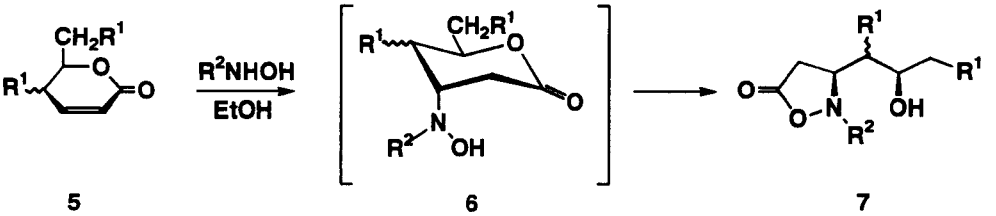


4

For the Michael addition to compound **2**, *N*-methylhydroxylamine hydrochloride - triethylamine in tetrahydrofuran solution has been employed.[5] On the other hand, addition of free and *N*-substituted hydroxylamines to lactones **5** has been performed in methanol or ethanol at pH=5; hydroxylamines were liberated from the respective hydrochlorides by titration with sodium methoxide.[6] Rearrangement of **6** into **7** is probably accelerated by the axial position of the hydroxylamine group in the Michael adduct **6**, which is suitably located for opening of the lactone ring and isoxazolidin-5-one ring formation. Reaction conditions probably play a less decisive role. It should be noted that reaction of **5** with *N*-methylhydroxylamine hydrochloride in the presence of triethylamine in THF afforded **7** as well. Different results from addition of *N*-substituted hydroxylamines to **2** compared to **5** has prompted us to reinvestigate the first reaction. It was particularly interesting to determine whether isoxazolidin-5-ones could be obtained from 4-substituted butenolides of defined absolute configuration.



5 6 7

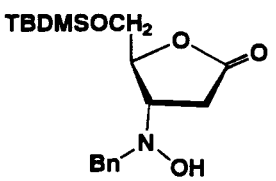
R^1 = H, OAc, OBn, OSilyl; R^2 = Me, Bn, 4-$MeOC_6H_4CH_2$, C_6H_{11}

Scheme 1

RESULTS AND DISCUSSION

Conjugate additions of *N*-methylhydroxylamine and *N*-benzylhydroxylamine hydrochloride to butenolide **2** were performed in tetrahydrofuran solution in the presence of triethylamine and in methanol at pH=5 following our protocol.[6] For the addition reaction to lactone **2**, unsubstituted hydroxylamine was employed as well. Due to the low solubility of hydroxylamine in a THF/Et_3N mixture, reactions were performed in methanol under neutral conditions and in methanol in the presence of triethylamine. In the case of *N*-substituted hydroxylamines, conjugate addition under both mild basic and neutral conditions, afforded hydroxylamines **3** and **8**, respectively. It should be noted that addition at pH=5 provided products having higher values of optical rotations, $[\alpha]_D$ +22.6 versus +21.5° for **3** (lit. Ref. 4, $[\alpha]_D$ +23), and $[\alpha]_D$ +35.0 versus +23.1 for **8**. These testify to a partial racemization of the lactone **2** in the presence of triethylamine. Addition of *N*-methylhydroxylamine took 1 h only, whereas that of *N*-benzylhydroxylamine required overnight reaction, the latter addition causing a higher degree of racemization of **2**. All reactions proceeded with high *trans* stereoselectivity. The *anti* approach of nucleophiles to 4-substituted butenolides is well documented in the literature.[4,5,8]

It should be noted that butenolides may undergo migration of a double bond. Isomerizations of 4-penten-4-olides each having an exocyclic double bond, and their non-conjugated (β,γ) and conjugated (α,β) isomers each having an endocyclic double bond have been studied.[9] Very recently, the base catalyzed γ-epimerization of the terminal butenolide fragment of acetogenins was also reported.[10]



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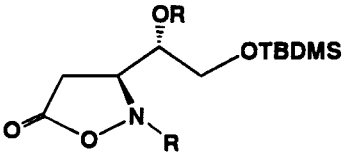


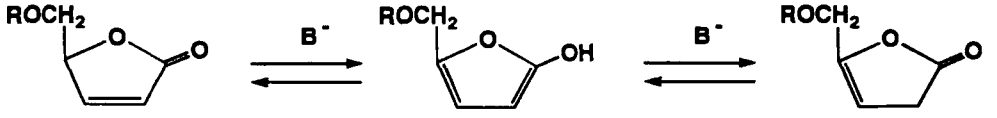
9: R = H
10: R = Ac

11: $R^1 = R^2 = H$
12: $R^1 = R^2 = Ac$
13: $R^1 = H$, $R^2 = Ac$

Addition of hydroxylamine to **2** at pH=5 proceeded similarly to the addition-rearrangement pathway found for six-membered ring lactones to afford isoxazolidin-5-one

9 as the single product. On the other hand, addition of hydroxylamine in triethylamine - methanol solution gave a mixture of two racemic products isoxazolidin-5-one **9** and the lactone **11** in a ratio of 3:2, respectively. In pyridine solution partly racemized **9** was formed.

Racemization of the lactones **1** and **2** proceeds *via* tautomeric form **1b**, **2b** and **1c**, **2c**, respectively. Tautomeric equilibrium is accelerated by the presence of a base (Scheme 2) and depends on solvent. Lactone **2** undergoes complete racemization in ethanol in the presence of 20 molar equivalents of Et_3N during 1 h. Lactone **1** racemizes slower than **2**; under the same condition (20 molar equiv of Et_3N in ethanol, 1 h) racemization reaches 19% ee. In THF solution racemization is slower than in ethanol; **2** in 20% Et_3N in THF after 24 h, reaches 55% ee. In pyridine racemization of **2** is negligible, after 24 h, reaches 95% ee.



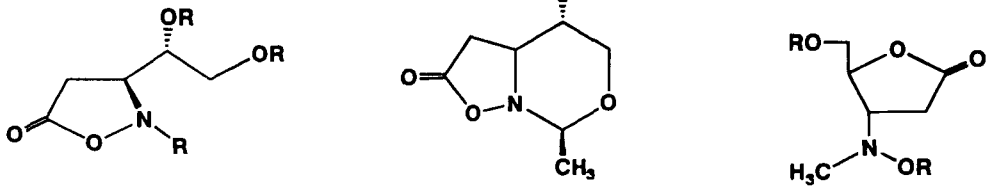
1a: R = H
2a: R = TBDMS

1b
2b

1c
2c

Scheme 2

The addition of hydroxylamine to flat tautomeric forms of the lactone **2** (**2b** or **2c**), catalyzed by amine hydrochloride present in the reaction mixture, led to the lactone **11**.



14: R = H
15: R = Ac

16: R = H
17: R = Ac

18: R = H
19: R = Ac

Hydroxylamine can be added at pH=5 to the 4-hydroxymethylbutenolide **1** to form corresponding isoxazolidin-5-one **14** which was characterized as triacetate **15**. Treatment of the crude **14** with acetaldehyde afforded bicyclic compound **16**, which was

characterized as **17**. The spectral data of **17** corespond well to the respective data of known 2,4,5-trisubstituted 1-aza-3,9-dioxa-8-oxobicyclo[4.3.0]nonanes.[11]

It should be noted that **1** added *N*-methylhydroxylamine in neutral conditions to give the Michael adduct **18** as the single product.

In summary, we showed that the addition-rearrangement reaction leading to isoxazolidin-5-ones can be performed using five-membered ring lactones **1** and **2**, provided unsubstituted hydroxylamine is used. We showed, as well, that commonly used conditions for conjugate addition of hydroxylamines emploing triethylamine as hydrochloric acid scavenger lead, at least, to the partially racemized products. Formation of **9** and **14** in our opinion might lead to further transformations similar to the ones that have been successfully performed by us for compounds **7**.[6,12]

EXPERIMENTAL

^{1}H NMR spectra were recorded with Bruker AM 500 and Varian Gemini 200 spectrometers. IR spectra were obtained on an FT-IR-1600 Perkin-Elmer spectrophotometer. Mass spectra were recorded with an AMD mass spectrometer. Optical rotations were measured with JASCO Dip-360 digital polarimeter. Column chromatography was performed on Merck silica gel 230-400 mesh.

Lactone **1** was purchased from Aldrich or was synthesized following a known procedure.[3] Standard silylation transformed **1** into lactone **2**.[4]

Racemization of lactones 1 and 2. Lactone **1** [0.17 mM; $[\alpha]_D$ - 144 (*c* 1, H_2O)] was dissolved in 96% EtOH (2 mL) and treated with Et_3N (0.5 mL). The mixture was stirred at room temperature for 1 h. Subsequently solvents were evaporated under diminished pressure and the residue was purified by chromatography to give **1** (ee = 19%), $[\alpha]_D$ - 27.0 (*c* 1, H_2O). Lactone **2** {$[\alpha]_D$ - 130.2 (*c* 1.6, CH_2Cl_2), - 138.2 (c 1, $CHCl_3$); lit: Ref. 2b $[\alpha]_D$ - 141 (*c* 0.937, $CHCl_3$), Ref. 2d $[\alpha]_D$ - 136.2 (*c* 1.13, $CHCl_3$), Ref. 4 $[\alpha]_D$ -122 (*c* 1.05, $CHCl_3$)} under the same conditions provides fully racemized compound.

Conjugate addition of *N*-methylhydroxylamine and *N*-benzylhydroxylamine to lactone 2. Procedure A. Lactone **2** (0.05 g, 0.22 mmol) in THF (2 mL) was treated with Et_3N (150 μL, 1.4 mmol) and *N*-substituted hydroxylamine (0.3 mmol). The mixture

was kept at room temperature until disappearance of the substrate. Subsequently, solvents were evaporated and the residue was dissolved in EtOAc. The solution was washed with water, dried, concentrated, and purified by chromatography to afford **3** or **8**.

5-*O*-*tert*-Butyldimethylsilyl-2,3-dideoxy-3-(*N*-methylhydroxylamino)-α-D-*erythro*-pentono-1,4-lactone (3); reaction complete after 1 h, 75%; $[\alpha]_D$ + 21.5 (*c* 1, $CHCl_3$) lit. Ref 4. $[\alpha]_D$ + 23 (*c* 1, $CHCl_3$), spectral data were reported previously.[4]

Anal. Calcd for $C_{12}H_{25}NO_4Si$: C, 52.33; H, 9.15; N, 5.09. Found: C, 52.2; H, 9.2; N, 5.1.

5-*O*-*tert*-Butyldimethylsilyl-3-(*N*-benzylhydroxylamino)-2,3-dideoxy-α-D-*erythro*-pentono-1,4-lactone (8); reaction complete after 18 h; 75%; $[\alpha]_D$ + 23.1 (*c* 0.7, CH_2Cl_2); IR ($CHCl_3$) 3574, 1772 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.73 (bm, 1H, H-4), 3.92 (dd, 1 H, *J* 2.8, 11.3 Hz, H-5), 3.87, 3.78 (2 d, 2 H, *J* 13.0 Hz, Bn), 3.76 (dd, 1 H, *J* 2.4, 11.3 Hz, H-5'), 3.72 (m, 1 H, H-3), 2.79 (bd, 1H, *J* 17.8 Hz, H-2), 2.71 (bdd, 1H, *J* 8.8, 17.8 Hz, H-2'); MS (HR, LSIMS) *m/z* $(M+H)^+$ Calcd for $C_{18}H_{30}NO_4Si$: 352.194412. Found: 352.194225.

Anal. Calcd for $C_{18}H_{29}NO_4Si$: C, 61.50; H, 8.32; N, 3.98. Found: C, 61.7; H, 8.3; N, 3.9

Procedure B. Lactone **2** (0.05 g, 0.22 mmol) in methanol (1 mL) was treated with a previously prepared solution which consisted of *N*-substituted hydroxylamine hydrochloride (0.43 mmol) in methanol (1 mL) titrated with sodium methoxide to pH=5 (~ 0.41 mmol) in methanol (1 mL). Subsequently, standard workup of the reaction mixture following the procedure described above yielded **3** or **8**.

3; reaction complete after 1 h; 80%; $[\alpha]_D$ + 22.6 (*c* 0.7, CH_2Cl_2)

8; reaction complete after 18 h; 76%; mp 72 - 73 °C; $[\alpha]_D$ + 35.4 (*c* 0.3, CH_2Cl_2)

(3*S*,1'*S*) - 2 - Acetyl-3-(1'-acetoxy-2'-*tert*-butyldimethylsilyloxyethyl)isoxazolidin-5-one (10). Addition of hydroxylamine was performed according to procedure A. The crude product **8** was treated with acetic anhydride - pyridine 1 : 1 $^v/_v$ under standard conditions to afford **10**, 73%, $[\alpha]_D$ + 53.4 (*c* 1.4, CH_2Cl_2); IR ($CHCl_3$) 1783 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.26 (bm, 1 H, H-3), 4.59 (m, 1 H, *J* 1.8, 2.1, 2.4 Hz, H-1'), 3.86 (dd, 1 H, *J* 2.4, 11.6 Hz, H-2'a), 3.83 (dd, 1H, *J* 2.1, 11.6 Hz, H-2'b), 2.93 (dd, 1 H, *J* 9.3, 18.1 Hz, H-4a), 2.52 (bdd, 1 H, *J* 1.6, 18.1 Hz, H-4b); MS (HR, LSIMS) *m/z* $(M+H)^+$ Calcd for $C_{15}H_{28}NO_6Si$: 346.168591. Found: 346.170765.

Anal. Calcd for $C_{15}H_{27}NO_6Si$: C, 52.15; H, 7.88; N, 4.05. Found: C, 52.0; H, 7.8; N, 4.1.

(3*S,1'*S**)-2-Acetyl-3-(1'-acetoxy-2'-*tert*-butyldimethylsilyloxyethyl)isoxazolidin-5-one (10) and 4-(*N*-acetoxy-*N*-acetyl-amino)-5-*tert*-butyldimethylsiloxypentanoic-1,4-lactone (12).** Lactone **2** (0.05g, 0.22 mmol) was dissolved in 96% ethanol (2 mL) and treated with hydroxylamine hydrochloride (0.07 g, 1 mmol) and triethylamine (0.5 mL, 3.6 mmol). The reaction mixture was stirred overnight at room temperature. Subsequently solvents were evaporated and the residue was dissolved in water (3 mL) and extracted with AcOEt. The extract was washed, dried, concentrated and treated with acetic anhydride - pyridine 1 : 1 $^{v}/_{v}$ mixture. After standard workup the crude product was purified by chromatography using hexane - EtOAc 7 : 3 $^{v}/_{v}$ as an eluent to afford **12** (0.024 g, 33%) and **10** (0.029 g, 40%).

12: syrup; IR ($CHCl_3$) 1807, 1726 cm^{-1}; ^1H NMR (C_6D_6, 80 °C) δ 3.98 (s, 2 H, CH_2OSi), 2.81 (bt, 2 H, H-2, 2'), 2.53 (t, 2 H, H-3, 3'), 1.99 (s, 3 H, NAc), 1.66 (s, 3 H, OAc), 0.92 (s, 9 H, *t*-Bu); MS (EI, HR) *m/z* $M^{+\cdot}$ Calcd for $C_{15}H_{27}NO_6Si$: 345.160761. Found: 345.161273. During one of the experiments performed under conditions described above the partially acetylated product **13** was isolated in minute amounts. Acetylation of **13** gave **12**.

13: IR ($CHCl_3$) 1798, 1727 cm^{-1}; ^1H NMR ($CDCl_3$) δ 3.72, 3.70 (2 d, 2 H, *J* 10.2 Hz, CH_2OSi), 2.57 (ddd, 1 H, *J* 5.8, 10.1, 17.2 Hz, H-2), 2.43 (ddd, 1H, *J* 5.0, 10.0, 17.2 Hz, H-2'), 2.30 (ddd, 1 H, *J* 5.8, 10.0, 13.3 Hz, H-3), 2.27 (s, 3H, NAc), 2.09 (ddd, 1 H, *J* 5.0, 10.1, 13.3 Hz, H-3'), 0.90 (s, 9 H, *t*-Bu).

Anal. Calcd for $C_{15}H_{27}NO_6Si$: C, 51.46; H, 8.30; N, 4.62. Found: C, 51.6; H, 8.5; N, 4.6.

(3*S*,1'*S*)-2-Acetyl-3-(1',2'-diacetoxyethyl)isoxazolidin-5-one (15). Addition of hydroxylamine to the lactone **1** was performed according to procedure A. The crude product **14** was treated with acetic anhydride - pyridine 1 : 1 $^{v}/_{v}$ mixture. Standard workup and purification on a silica gel column provided **15** (0.182 g, 67%); syrup $[\alpha]_D$ + 56.9 (*c* 0.23, CH_2Cl_2); IR ($CHCl_3$) 1793, 1748, 1686 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.18 (ddd, 1 H, *J* 3.2, 3.9, 9.1 Hz, H-3), 4.75 (dt, 1 H, *J* 3.2, 3.2, 3.8 Hz, H-1'), 4.36 (dd, 1 H, *J* 3.2, 12.4 Hz, H-2'a), 4.22 (dd, 1 H, *J* 3.8, 12.4 Hz, H-2'b), 2.91 (dd, 1 H, *J* 9.1, 17.3 Hz, H-4a),

2.65 (dd, 1 H, *J* 3.9, 17.3 Hz, H-4b), 2.26, 2.10, 2.04 (3 s, 9 H, 3 Ac). MS (LSIMS) *m/z* $(M+H)^+$ 274.

Anal. Calcd for $C_{11}H_{15}NO_7$: C, 48.35; H, 5.49; N, 5.12. Found: C, 48.6; H,5.7; N, 4.9.

(2*S*,5*S*,6*S*) - 5 - Acetoxy - 1-aza-2-methyl-3,9-dioxa-8-oxobicyclo[4.3.0]nonane (17). Crude isoxazolidin-5-one **14**, obtained from lactone **1** (0.114 g, 1 mmol) according to the procedure described above, was treated with acetaldehyde (1.5 mL) and kept at room temperature for 3 h. Subsequently the solution was concentrated and the crude product was acetylated with acetic anhydride - pyridine 1 : 1 mixture. A standard workup gave **17** (0.107 g, 50%); mp 128 -130 °C; $[\alpha]_D$ + 73.0 (*c* 0.5, CH_2Cl_2); IR ($CHCl_3$) 1792, 1750 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.89 (ddd, 1 H, *J* 5.5, 9.6, 10.5 Hz, H-5), 4.46 (q, 1 H, *J* 6.0 Hz, H-2), 4.22 (dd, 1 H, *J* 5.5, 10.9 Hz, H-4), 3.76 (bdd, 1 H, *J* 7.0, 9.6 Hz, H-6), 3.35 (t, 1 H, *J* 10.5, 10.9 Hz, H-4'), 3.05 (dd, 1 H, *J* 7.0, 16.8 Hz, H-7), 2.68 (d, 1 H, *J* 16.8 Hz, H-7'), 2.08 (s, 3 H, Ac), 1.51 (d, 3 H, CH_3); MS (HR, EI) *m/z* $M^{+\cdot}$ Calcd for $C_9H_{13}NO_5$: 215.07937. Found: 215.079191.

3 - (*N*-Acetoxy-*N*-methylamino)-5-*O*-acetyl-2,3-dideoxy-α-D-*erythro*-pentono-1,4-lactone (19). Addition of *N*-methylhydroxylamine to lactone **1** was performed according to procedure A. The crude product **19** was treated with acetic anhydride - pyridine 1 : 1 $^v/_v$ to afford, after column chromatographic purification, **19** (0.20 g, 83%), syrup; $[\alpha]_D$ + 32.6 (*c* 1, CH_2Cl_2); IR ($CHCl_3$) 1786, 1762, 1752 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.75 (ddd, 1 H, *J* 3.1, 3.4, 4.3, Hz, H-4), 4.40 (dd, 1H, *J* 3.1, 12.3 Hz, H-5), 4.19 (dd, 1 H, *J* 4.3, 12.3 Hz, H-5'), 3.66 (ddd, 1 H, *J* 3.4, 6.2, 6.9 Hz, H-3), 2.79 (s, 3 H, NCH_3), 2.77 (m, 2 H, H-2, 2'), 2.10, 2.06 (2 s, 6 H, 2 Ac). MS (LSIMS) *m/z* $(M+H)^+$ 246.

Anal. Calcd for $C_{10}H_{15}NO_6$: C, 48.47; H, 6.12; N, 5.71. Found: C, 48.5; H, 6.2; N, 5.8.

ACKNOWLEDGMENTS

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