Anal. Calcd for $C_{35}H_{32}O_6$: C, 76.6; H, 5.88. Found: C, 76.2; H, 5.76.

Acknowledgment. We thank the American Cancer Society for financial support.

Registry No. 1, 76231-50-0; 2, 65904-27-0; 3, 133009-70-8; 4, 133098-46-1; 5, 133098-47-2; 6, 133009-71-9; 7, 133009-72-0; 12, 122745-02-2; 12 isoproxy derivative, 133009-73-1; $Pd(OAc)_2$, 3375-31-3; 2-bromopropane, 75-26-3.

Ritter-like Reactions of 1,2-Anhydropyranose Derivatives

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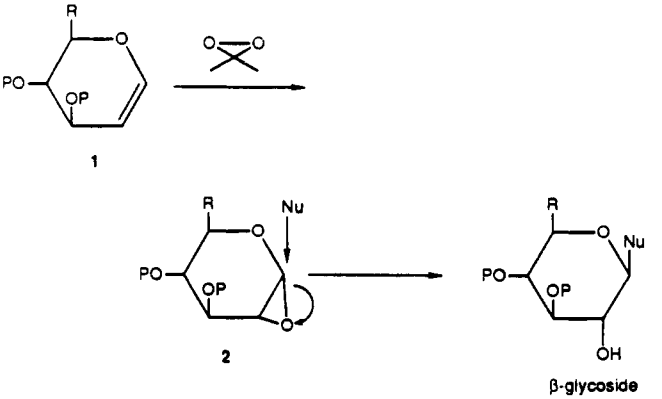
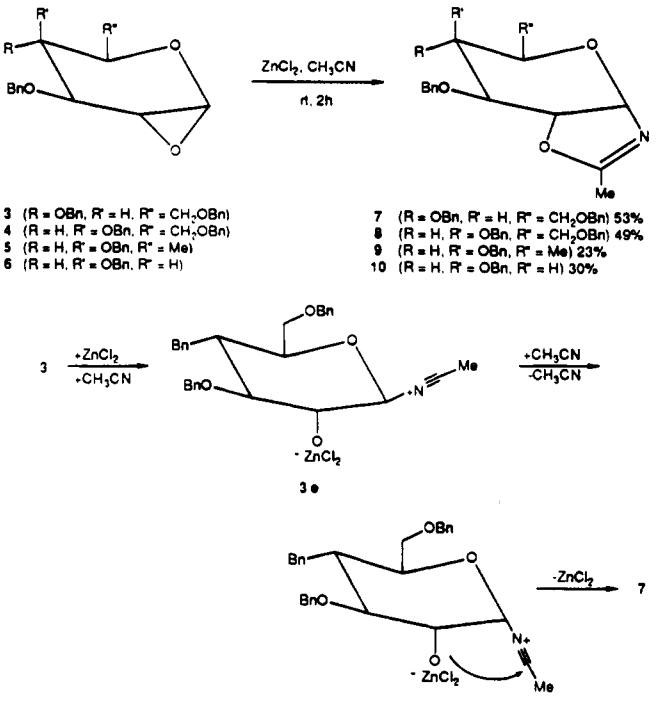
Recently we described a one step synthesis of α-1,2-anhydropyranose systems (cf. 2) from glycals (1).[1,2] If the resident protecting groups (P) lack participatory functionality (cf. benzyl or silyl ethers), nucleophilic attack occurs at the anomeric carbon with a high degree of stereoselectivity favoring inversion. Given the ease of reaching various glycals by total synthesis,[3] or by partial synthesis from other monosaccharides,[4] and given the excellent stereoselectivity which can be realized from the use of 1,2-anhydro sugars as glycosyl donors, the importance of the method is likely to grow. Application of such oxiranes to the synthesis of oligosaccharides and to the synthesis of other glycosyl donors (cf. *n*-pentenyl glycosides,[5] fluorides,[6] and thiophenyl ethers[7]) has recently been reported.[8] A potentially important outcome of the method is that it unveils a free hydroxyl group at C_2,[9] thus differentiating that oxygen from protected oxygens at carbons 3, 4, and 6. In this report we demonstrate an interesting application of this feature of the process in the context of the preparation of the previously uncharacterized C_1-nitrogen linked 1,2-glycooxazoline unit (see compounds 7–10)[10] by a Ritter[11] "type" solvolysis of these epoxides.[12]



Solutions of 1,2-anhydropyranose systems **3–6** in dry acetonitrile were treated with anhydrous zinc chloride to produce oxazolines **7–10** in the yields indicated. While the compounds were fully characterized (IR, ^1H NMR, HRMS, and optical rotation), they proved to be too sensitive for shipment and accurate combustion analysis. A pathway which is presumably general for the series is shown for the transformation of **3** → **7**. It is suggested that **3** undergoes the usual inversion to afford the equatorial anomeric system **3e**. This intermediate suffers inversion to produce axial anomer **3a**,[12] which is captured by the proximal α-hydroxyl function at C_2. Alternatively, **3e** might be produced by a S_Ni type opening of the oxirane.



With oxazoline **7** in hand, we tested the possibility of its intermediacy in the transformation of 2,3,4,6-tetra-*O*-benzyl-D-glucose to **11** and *N*-benzylacetamide under the

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(10) The isomeric 5*H*-pyrano[3,2-*d*]oxazoles are well known. For their use as glycosyl donors, see: (a) Thomas, R. L.; Abbas, S. A.; Matta, K. L. *Carbohydr. Res.* **1988**, *175*, 153. (b) Piskorz, C. F.; Abbas, S. A.; Matta, K. L. *Carbohydr. Res.* **1984**, *131*, 257.

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(12) For recent examples of elaborating an axial anomeric nitrilium intermediate via a Ritter-like solvolysis of anomeric N.P.G. derivatives in acetonitrile, see: (a) Ratcliffe, A. J.; Konradsson, P.; Fraser-Reid, B. *J. Am. Chem. Soc.* **1990**, *112*, 5665. (b) Ratcliffe, A. J.; Fraser-Reid, B. *J. Chem. Soc. Perkin Trans. 1* **1990**, 747. In these cases the C_2-hydroxyl group is protected and no oxazoline is produced.

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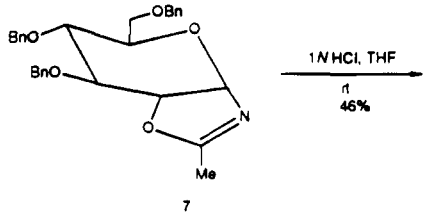
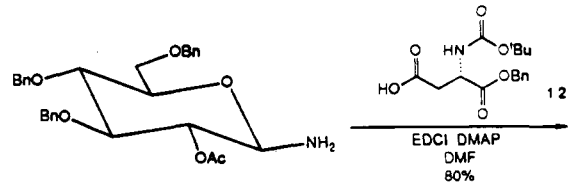
influence of triflic anhydride. Pavia[13] had discovered this reaction and proposed that it passes through 7. For this pathway to be viable, oxazoline 7 would have to have undergone acid-catalyzed hydrolysis to afford the β-anomer 11, which was the isolated carbohydrate product in the Pavia process. In the event, acid-catalyzed hydrolysis of 7 did indeed afford 11 presumably by anomerization of the kinetically generated C_1-α amino grouping.[14] Thus oxazoline 7 is certainly a permissive intermediate in the Pavia reaction.

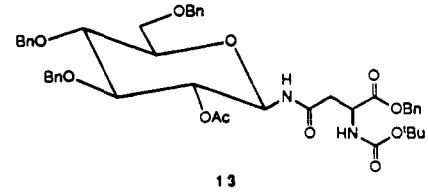
Acylation of 11 with the commercially available aspartic acid derivative 12, mediated by EDCI–DMAP, afforded the β-glucosylasparagine derivative 13. The presence of a β-glucosylasparagine linkage in a glycopeptide has been reported in one instance.[15] Further studies of the application of 1,2-anhydropyranose derivatives to glycoside synthesis are in progress.



7



11



13

Experimental Section

General Procedure for Preparation of Oxazolines (7, 8, 9, and 10). To a solution of 1,2-anhydropyranose[1] (240 μmol) in anhydrous acetonitrile (3 mL) was added 1 M $ZnCl_2$ in diethyl ether (250 μL). After being stirred for 2 h, at room temperature and under N_2, the mixture was quenched by the addition of saturated aqueous $NaHCO_3$ (10 mL) and further diluted with H_2O (10 mL). The resulting mixture was extracted with diethyl ether (3 × 20 mL), and the combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by SiO_2 chromatography (20% ethyl acetate in methylene chloride).

(3a*S*,5*R*,6*R*,7*S*,7a*R*)-6,7-Bis(benzyloxy)-5-[(benzyloxy)methyl]-2-methyl-5*H*-pyrano[2,3-*d*]oxazole (7). 1,2-Anhydro-3,4,6-tri-*O*-benzyl-α-D-glucopyranose 3 (104 mg, 240 μmol) gave 7 (60 mg, 127 μmol, 53%): $[\alpha]^{22}_D$ +53.8° (*c* 2.70, $CHCl_3$); IR ($CHCl_3$) 3050, 3020, 2990, 2900, 2860, 1655, 1600, 1490, 1450, 1385, 1360, 1090 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 7.39–7.18 (m, 15 H, ArH), 5.87 (d, 1 H, *J* = 7.57 Hz, H-1), 4.79–4.43 (m, 7 H, $ArCH_2$ and H-2), 3.81–3.69 (m, 4 H, H-3, H-4, H-6, and H-6′), 3.57–3.54 (m, 1 H, H-5), 2.04 (s, 3 H, CH_3); MS *m/z* 382 ($M^+ - C_7H_7$); HRMS calcd for $C_{29}H_{32}NO_5$ $(M + H)^+$ 474.2282, found 474.2268.

(3a*S*,5*R*,6*S*,7*S*,7a*R*)-6,7-Bis(benzyloxy)-5-[(benzyloxy)methyl]-2-methyl-5*H*-pyrano[2,3-*d*]oxazole (8). 1,2-Anhydro-3,4,6-tri-*O*-benzyl-α-D-galactopyranose 4 (104 mg, 240 μmol) gave 8 (55.3 mg, 117 μmol, 49%): $[\alpha]^{22}_D$ +33.7° (*c* 2.70, $CHCl_3$); IR ($CHCl_3$) 3080, 3060, 3020, 3000, 2920, 2860, 1655, 1600, 1490, 1450, 1390, 1360, 1310, 1270, 1090 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 7.40–7.27 (m, 15 H, ArH), 5.75 (d, 1 H, *J* = 6.55 Hz, H-1), 4.94 (d, 1 H, *J* = 11.49 Hz, $^1/_2$ AB q, $ArCH_2$), 4.79–4.60 (m, 4 H, $ArCH_2$ and H-2), 4.53 (d, 1 H, *J* = 11.82 Hz, $^1/_2$ AB q, $ArCH_2$), 4.46 (d, 1 H, *J* = 11.79 Hz, $^1/_2$ AB q, $ArCH_2$), 4.11–4.09 (m, 1 H, H-4), 4.03–4.00 (m, 1 H, H-5) 3.77–3.60 (m, 3 H, H-3, H-6, and H-6′), 2.05 (s, 3 H, CH_3); HRMS calcd for $C_{29}H_{32}NO_5$ $(M + H)^+$ 474.2282, found 474.2308.

(3a*S*,5*R*,6*S*,7*S*,7a*R*)-6,7-Bis(benzyloxy)-2,5-dimethyl-5*H*-pyrano[2,3-*d*]oxazole (9). 1,2-Anhydro-3,4-di-*O*-benzyl-α-D-fucopyranose 5 (78.3 mg, 240 μmol) gave 9 (20.3 mg, 55 μmol, 23%): $[\alpha]^{22}_D$ +47.4° (*c* 1.00, $CHCl_3$); IR ($CHCl_3$) 3070, 3040, 3015, 3000, 2920, 2860, 1655, 1600, 1490, 1450, 1370, 1350, 1300, 1140, 1070, 1020 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 7.39–7.25 (m, 10 H, ArH), 5.72 (d, 1 H, *J* = 6.66 Hz, H-1), 4.92 (d, 1 H, *J* = 11.72 Hz, $^1/_2$ AB q, $ArCH_2$), 4.78–4.55 (m, 4 H, $ArCH_2$ and H-2), 3.90 (dq, 1 H, *J* = 6.55 and 3.07 Hz, H-5), 3.72 (app t, 1 H, *J* = 2.95 Hz, H-4), 3.59 (dd, 1 H, *J* = 6.98 and 2.18 Hz, H-3), 2.00 (s, 3 H, CH_3), 1.22 (d, 3 H, *J* = 6.57 Hz, CH_3); HRMS calcd for $C_{22}H_{26}NO_4$ $(M + H)^+$ 368.1863, found 368.1859.

(3a*S*,6*S*,7*S*,7a*R*)-6,7-Bis(benzyloxy)-2-methyl-5*H*-pyrano[2,3-*d*]oxazole (10). 1,2-Anhydro-3,4-di-*O*-benzyl-β-L-arabinopyranose 6 (75 mg, 240 μmol) gave 10 (25 mg, 71 μmol, 30%): $[\alpha]^{22}_D$ +46.8° (*c* 1.25, $CHCl_3$); IR ($CHCl_3$); 3040, 3020, 2990, 2900, 2850, 1655, 1600, 1490, 1450, 1390, 1345, 1300, 1100, 1050, 1030 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 7.38–7.26 (m, 10 H, ArH), 5.57 (d, 1 H, *J* = 6.02 Hz, H-1), 4.76–4.57 (m, 5 H, $ArCH_2$ and H-2), 3.98–3.86 (m, 3 H, H-4, H-5, and H-5′), 3.74 (dd, 1 H, *J* = 7.03 and 1.81 Hz, H-3), 2.06 (s, 3 H, CH_3); HRMS calcd for $C_{21}H_{24}NO_4$ $(M + H)^+$ 354.1706, found 354.1727.

2-*O*-Acetyl-3,4,6-tri-*O*-benzyl-β-D-glucopyranosylamine (11). To a solution of oxazoline 7 (44.6 mg, 94 μmol) in 1:1 tetrahydrofuran–water (2 mL) was added 1 N hydrochloric acid (10 μL). After being stirred at ambient temperature for 30 min the mixture was quenched by the addition of 1 N sodium hydroxide (10 mL) and further diluted with water (10 mL). The resulting solution was extracted with ethyl acetate (3 × 25 mL), and the combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by SiO_2 chromatography (20% ethyl acetate in methylene chloride) to give 11 (21.3 mg, 43.3 μmol, 46%): $[\alpha]^{22}_D$ +17.6° (*c* 1.40, $CHCl_3$) (lit.[11] $[\alpha]^{22}_D$ +23° no concentration or solvent given); mp 108–9 °C (lit.[11] mp 110 °C); IR ($CHCl_3$) 3650, 3400, 3320, 3070, 3040, 3010, 2995, 2890, 2850, 1735, 1600, 1490, 1450, 1370, 1240, 1090, 1050 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 7.34–7.10 (m, 15 H, ArH), 4.84–4.46 (m, 7 H, $ArCH_2$ and H-2), 4.05 (d, 1 H, *J* = 8.95 Hz, H-1), 3.72–3.57 (m, 4 H, H-3, H-4, H-6, and H-6′), 3.51–3.44 (m, 1 H, H-5), 1.98 (s, 3 H, CH_3), 1.89 (bs, 2 H, NH_2). Anal. Calcd for $C_{29}H_{33}NO_6$: C, 70.86; H, 6.77; N, 2.85. Found: C, 70.99; H, 7.00; N, 2.64.

***N*-[1-Benzyl *N*-(*tert*-butyloxycarbonyl)-4-L-aspartyl]-2-*O*-acetyl-3,4,6-tri-*O*-benzyl-β-D-glucopyranosylamine (13).** To a solution of 11 (35.8 mg, 73 μmol), in dimethylformamide (DMF, 1 mL), were added 4-(dimethylamino)pyridine (DMAP, 8.9 mg, 73 μmol), 1-benzyl *N*-(*tert*-butyloxycarbonyl)-L-aspartoate 12 (48.5 mg, 150 μmol) and 1-[3-(dimethylamino)propyl]-3-ethylcarbodiimide hydrochloride (EDCI, 28.8 mg, 150 μmol). The mixture was stirred for 18 h at ambient temperature under N_2. The solution was then diluted with H_2O (15 mL) and washed with ethyl acetate (3 × 15 mL), and the combined organics were washed with brine (15 mL), dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by SiO_2 chromatography (33% ethyl acetate in hexane) to give 13 (46.3 mg, 58.1 μmol, 80%): $[\alpha]^{22}_D$ +35.9° (*c* 2.26, $CHCl_3$); mp 153–4 °C; IR ($CHCl_3$) 3400, 3020, 3000, 2920, 2860, 1735, 1700, 1600, 1490, 1450, 1365, 1240, 1160, 1060 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 7.35–7.22 (m, 18 H, ArH), 7.15–7.09 (m, 2 H, ArH), 6.32 (d, 1 H, *J* = 9.10 Hz, NH), 5.72 (d, 1 H, *J* = 9.09 Hz, NH), 5.14 (s, 2 H, $ArCH_2O_2C$), 5.01 (t, 1 H, *J* = 9.28 Hz, H-1), 4.86–4.43 (m, 8 H, $ArCH_2$, H-2 and $NCHCO_2$),

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(15) Wieland, F.; Heitzer, R.; Schaeffer, W. *Proc. Natl. Acad. Sci. U.S.A.* **1983**, *80*, 5470.

3.77-3.66 (m, 4 H, H-3, H-4, H-6, and H-6'), 3.52-3.48 (m, 1 H, H-5), 2.83 (dd, 1 H, J = 16.35 and 4.10 Hz, CH_2CON), 2.65 (dd, 1 H, J = 16.35 and 4.10 Hz, CH_2CON), 1.88 (s, 3 H, CH_3), 1.39 (s, 9 H, $(CH_3)_3C$). Anal. Calcd for $C_{45}H_{52}N_2O_{11}$: C, 67.82; H, 6.58; N, 3.52. Found: C, 67.79; H, 6.35; N, 3.25.

Acknowledgment. We thank the NIH (Grant HL 25848) for financial support in this work. An NSF Predoctoral Fellowship to D.M.G. is gratefully acknowledged. NMR spectra were obtained through the auspices of the Northeast Regional NSF/NMR facility at Yale University, which was supported by NSF Chemistry Division Grant CHE 7916210. We thank Dr. Ken Chow for early survey work.

Registry No. 3, 74372-90-0; 4, 112289-38-0; 5, 132884-63-0; 6, 132884-64-1; 7, 133004-71-4; 8, 132884-65-2; 9, 132884-66-3; 10, 132884-67-4; 11, 77611-19-9; 12, 30925-18-9; 13, 132884-68-5; CH_3CN, 75-05-8.

Addition of Glycinate Enolate Equivalents to 1,4-Benzodiazepine Imino Phosphates. Preparation of Synthetically Useful 2-(Ethyl glycinat-α-ylidene)-1,4-benzodiazepines and Related Derivatives[1]

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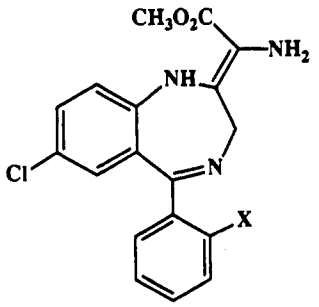
Received October 19, 1990

As part of our continuing interest in the synthesis of novel tricyclic 1,4-benzodiazepines, we required 2-dehydroglycinate-1,4-benzodiazepines of type 1 or related derivatives as precursors to compounds with a heterocyclic ring fused to the *a* face of the benzodiazepine ring. The usual approach to synthesize compounds of this type involves a linear route[2] involving a multistep construction of the dehydroglycinate portion at C-2 via a malonylidene intermediate.



1: X= H
2: X= Cl

Imino phosphates of 1,4-benzodiazepines are useful imidoyl derivatives known to be activated for nucleophilic attack.[3] We have previously reported one example of direct nucleophilic introduction of dehydroglycinate functionality[4] via reaction of a nitrone-activated glycinate

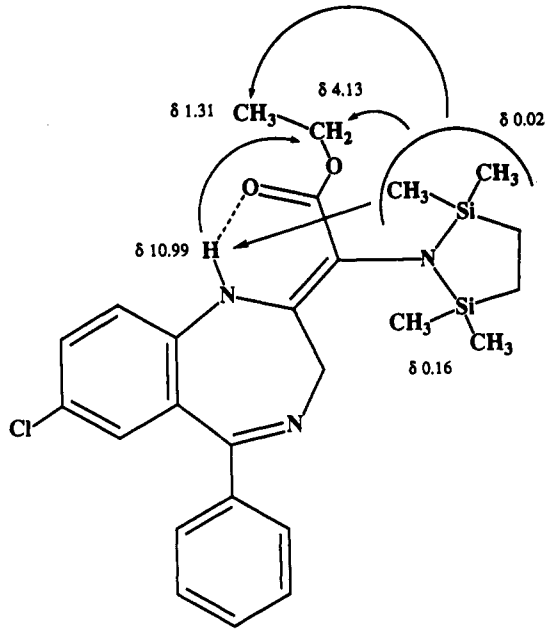
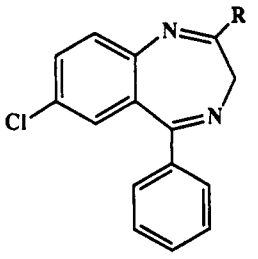
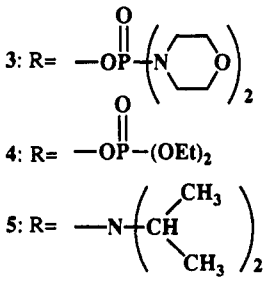


Figure 1. Observed NOE's in 2D-NOESY spectrum of 12 in $CDCl_3$.

derived carbanion and an imino phosphate. The growing number of reports of glycinate enolate synthons[5] presented us with an opportunity to explore direct nucleophilic introduction of the desired dehydroglycinate functionality via reaction of various glycinate enolates with appropriate imino phosphates. Such a method for elaborating imino phosphates derived from secondary cyclic amides should find general utility. For the present study, we chose imino dimorpholinophosphate 3[6] and imino diethylphosphate 4[2a] due to their excellent reactivity with various nucleophiles.[7]





Imino phosphate 3 was treated with the ester enolate of 2,2,5,5-tetramethyl-1-aza-2,5-disilacyclopentane-1-acetate 6[5c] (LDA, -78 °C, THF) to give the desired adduct

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‡ Present address: Gilead Sciences, Foster City, CA 94404.

(1) A preliminary account of this work has been presented at the 199th National Meeting of the American Chemical Society, ORGN 469, Boston, MA, April 22-27, 1990.

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