General Reaction Procedures. All reactions were done at ice-bath temperatures at 0.04 mol fraction in olefin. Powdered NBS was added in one amount as a solid to a stirred solution of the olefin in methanol. Two or three drops of concentrated sulfuric acid were added immediately after the NBS. The reactions were done on a 0.5- and 2-g scale for analytical analysis and isolation, respectively. Iodometric titration was used to determine completeness of the reaction. The reaction mixtures were added to water containing sodium bisulfite (to destroy residual NBS) and extracted with dichloromethane. The dichloromethane was washed with aqueous sodium bicarbonate to neutralize acid. The solvent was removed at reduced pressure, and the products, with the exception of **10**, **13**, and **14**, were isolated by vacuum distillation.

3-Bromo-4-methoxybutan-2-one (2). This compound has not been reported previously. The structure was established by NMR, IR, and elemental analysis. We found **2** to be unstable: it rapidly turned dark on standing. The NMR of the isolated material always showed impurity peaks at 1.30 and 3.18 ppm. Impurities resulting from decomposition probably accounted for the inaccuracy in the elemental analysis. **2**: NMR δ 2.31 (s, 3 H), 3.37 (s, 3 H), 3.52–4.43 (m, 3 H), IR (CCl_4) 1720 (C=O), 2805 (CH_3O) cm^{-1}; bp 58–61 °C (2.5 mm). Anal. Calcd for $C_5H_9BrO_2$: C, 33.17; H, 5.01; Br, 44.14. Found: C, 33.70; H, 5.22; Br, 43.14.

2-Bromo-3-methoxy-1-phenylpropan-1-one (4). The boiling point (106–110 °C (0.7 mm) [lit.[8] 121–123 °C (1.0 mm)] of **4** was reported previously. **4**: NMR δ 3.39 (s, 3 H), 3.60–4.20 (m, 2 H), 5.00 and 5.10 (dd, 1 H), 7.16–7.62 (m, 3 H), 7.88–8.10 (m, 2 H); IR (CCl_4) 1700 (C=O), 2840 (CH_3O) cm^{-1}.

3-Bromo-4-methoxypentan-2-one (6). This compound has not been reported previously. The structure was established by NMR, IR, and elemental analysis: NMR δ 1.30 (d, 3 H), 2.28 (s, 3 H), 3.32 (s, 3 H), 3.54–4.23 (m, 2 H); IR (CCl_4) 1725 (C=O), 2825 (CH_3O) cm^{-1}; bp 37–38 °C (0.25 mm). Anal. Calcd for $C_6H_{11}BrO_2$: C, 36.95; H, 5.58; Br, 40.97. Found: C, 37.12; H, 5.57; Br, 40.57. VPC analysis of this product showed two peaks (75:25), which were assumed to be diastereomers for the following reasons: the elemental analysis was correct for compounds with the indicated formula; they are not regioisomers since triethylamine has no effect; the NMR spectra for the two compounds must be similar since no major unexplained peaks were present in the actual spectrum.

3-Bromo-4-methoxy-4-methylpentan-2-one (8). The boiling point [66–68°C (3.0 mm) (lit.[3] 59–60 °C (3.0 mm))] and NMR and IR spectra have been reported previously. The spectral data are in agreement.

2-Bromo-3-methoxypropanal (10). This compound has not been reported previously. The structure was established by NMR, IR, and elemental analysis: NMR δ 3.40 (s, 3 H), 3.75 and 3.85 (2 d, 2 H), 4.00–4.39 (m, 1 H), 9.50 (d, 1 H); IR (CCl_4) 1725 (C=O), 2835 (CH_3O) cm^{-1}. Anal. Calcd for $C_4H_7BrO_2$: C, 28.76; H, 4.22; Br, 47.85. Found: C, 28.26; H, 4.42; Br, 47.06. Aldehyde **10** was isolated as follows: Solid sodium bicarbonate was added directly to the methanol solution to remove acid. Solids and methanol were removed by filtration and distillation, respectively, and **10** was obtained by preparative VPC, using a 2.4 m × 9 mm glass column packed with 5% DC-550. We failed to isolate **10** by extraction from aqueous solution followed by fractional distillation. Removal of methanol, followed by direct vacuum distillation of the acidic mixture of **10** and the acetal (**11**), led to a mixture of **11** and α-bromoacrolein (**15**) in a ratio of ca. 30:70, respectively. α-Bromoacrolein was identified by comparison of its NMR spectrum to that of the authentic compound prepared by elimination of 2,3-dibromopropanal with triethylamine: NMR δ 6.80 (d, 1 H, J = 1.9 Hz), 6.84 (d, 1 H, J = 1.9 Hz), 9.10 (s, 1 H). Formation of **15** probably occurred by protonation of the methoxy group in **10** followed by elimination of methanol. The concentration of **10** was continually replenished by equilibration from **11**.

2-Bromo-1,1,3-trimethoxypropane (11). This compound has been reported; bp 25 °C (1 mm) [lit.[9] 60 °C (2 mm)]; IR (CCl_4) 1080 and 1125 (acetal) cm^{-1} (lit.[9] 1070–1185 cm^{-1};[10] NMR δ 3.42 (s, 6 H), 3.68 (d, 2 H), 3.81–4.18 (m, 1 H), 4.45 (d, 1 H).

Methyl 2-Bromo-3-methoxypropanoate (13) and Methyl 3-Bromo-2-methoxypropanoate (14). The NMR spectra of these compounds have been reported.[3] We isolated **13** and **14** by preparative VPC and compared our NMR spectra with the reported values. They are in excellent agreement except for the reported multiplet for **14** at 4.30 ppm; we observed it at 3.86–4.05 ppm. Elimination with triethylamine occurred immediately for **14**, confirming that the bromine is on the β-carbon.

Acknowledgment. Support for this work was provided by the donors of Petroleum Research Fund, administered by the American Chemical Society, and Research Associates of Point Loma College.

Registry No. **1**, 78-94-4; **2**, 64151-19-5; **3**, 768-03-6; **4**, 85083-55-2; **5**, 3102-33-8; **6** (isomer 1), 85083-56-3; **6** (isomer 2), 85083-57-4; **7**, 141-79-7; **8**, 60456-54-4; **9**, 107-02-8; **10**, 10490-50-3; **11**, 759-97-7; **12**, 96-33-3; **13**, 27704-96-7; **14**, 60456-17-9; H_2SO_4, 7664-93-9; NBS, 128-08-5; CH_3OH, 67-56-1.

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(10) We assume that there is a mistake in the reported value of 1185 cm^{-1}. Literature values (cm^{-1}) for other acetals are as follows: dichloroacetaldehyde diethyl acetal (1085 and 1120) and bromoacetaldehyde dimethyl acetal (1065 and 1120). See: "The Aldrich Library of Infrared Spectra", 2nd ed., C. J. Pouchert, Ed., Aldrich Chemical Co., 1974, Milwaukee, WI, p 121.

Regiospecific Synthesis of Isoxazoles by Reaction of 1-Azabutadiene Derivatives with Hydroxylamine Hydrochloride

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Azabutadiene derivatives **1** are easily obtained by reaction of Schiff bases with saturated nitriles in the presence of $AlCl_3$[1] and have been found to be excellent starting materials for the synthesis of five- and six-membered heterocycles. The double condensation of **1** with various sulfur halides yields 1,2,6-thiadiazines.[2] On the other hand, the reaction of **1** with heterocumulenes leads to pyrimidones and thiopyrimidones in excellent yield; the formation of these heterocycles can be explained through an addition followed by electrocyclic ring closure.[3]

In a previous paper we have shown new preparative possibilities of systems **1** in the unequivocal synthesis of 1*H*-pyrrole-2-carboxylates. This heterocyclization reaction results from the exchange between the amino group of the glycines and the imino group of the azabutadiene followed by electrocyclic ring closure of the azapentadienyl anion intermediate.[4]

From these results it seems likely that 1-azabutadienes **1** might be suitable precursors for five-membered heterocycles containing the –N–Z (Z = heteroatom) group in the ring. With this in mind we have studied the reaction of

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(3) J. Barluenga, V. Rubio, and V. Gotor, *J. Org. Chem.*, **45**, 2592 (1980).

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Table I. Isoxazoles 4 from 1-Azabutadienes 1 and Hydroxylamine Hydrochloride 2

4	R^2	R^3	R^4	yield,[a] %	mp, °C	lit. mp, °C	^{13}C NMR,[b] δ
a	C_6H_5	CH_3	C_6H_5	88	124-6	127[8a]	163.4 (s), 108.4 (s), 165.4 (s)
b	C_6H_5	H	C_6H_5	75	143.4	141[8b]	163.2 (s), 97.4 (d), 170.3 (s)
c	C_6H_5	H	CH_3	65	63-5	63-6[8c]	159.9 (s), 99.8 (d), 169.1 (s)
d	C_6H_5	H	p-$CH_3C_6H_4$	76	128-9	130-1[8d]	162.7 (s), 97.6 (d), 170.0 (s)
e	p-$CH_3C_6H_4$	H	C_6H_5	63	136-8	138-9[8b]	162.6 (s), 97.4 (d), 169.8 (s)
f	C_6H_5	CH_3	p-$CH_3C_6H_4$	96	95-7		163.1 (s), 107.7 (s), 165.0 (s)
g	C_6H_5	CH_3	c-C_6H_{11}	76	106-8		163.8 (s), 107.8 (s), 167.5 (s)
h	p-ClC_6H_4	CH_3	C_6H_5	79	130-2		163.7 (s), 108.9 (s), 164.4 (s)
i	C_6H_5	CH_3	p-ClC_6H_4	89	119-21		162.4 (s), 108.2 (s), 165.7 (s)
j	p-ClC_6H_4	CH_3	p-$CH_3C_6H_4$	74	118-9		163.5 (s), 108.5 (s), 164.0 (s)
k	C_6H_5	CH_3	n-C_3H_7	69	oil		163.8 (s), 108.9 (s), 163.9 (s)

[a] Yields are given for 1-azabutadienes 1 in which $R^1 = C_6H_5$. [b] ^{13}C NMR data for isoxazole-ring carbon atoms. See paragraph at the end of paper about supplementary material.

1 with substrates that contain the H_2N–Z– group and, hence, are potentialy able to generate five-membered heterocycles containing two heteroatoms through a double-exchange–cyclization process.

In the present paper we report the reaction of 1 with hydroxylamine hydrochloride 2. Intermediate products that result from the exchange of the imine NH in the azabutadiene with the hydroxylamine group are isolated, and a possible mechanism for this new heterocyclization process is discussed.

Results and Discussion

Azabutadiene derivatives 1 react with hydroxylamine hydrochloride at 90 °C in pyridine to give rise to various isoxazoles 4. The heterocycles 4 are obtained in excellent yields in a regiospecific manner; the results are summarized in Table I.

The formation of isoxazoles 4 can be easily explained by an exchange reaction between the imino group of the azabutadiene and the hydroxylamine group to give the compounds 3. The hydroxylazabutadiene derivatives 3 undergo in the reaction media the corresponding heterocyclization to give heterocycles 4 (Scheme I).

Intermediate products 3 are isolated without difficulty when 1 is allowed to react with 2 at room temperature in pyridine solution (Table II). The heating of compounds 3 at 100 °C in pyridine leads to heterocycles 4 in nearly quantitative yield. However, at room temperature regardless of the reaction time, the formation of isoxazoles 4 is not detected.

By contrast with 1-azabutadienes 1, compounds 3 are isolated in the tautomer form of 1,3-diimines 3y. This is suggested by the ^1H and ^{13}C NMR spectral data for these compounds. The ^1H NMR spectrum shows the coupling between the methyl group ($R^3 = CH_3$) with the proton attached to C_a. In the ^{13}C NMR spectrum C_a displays a signal centered at about 50 ppm.

The heterocyclization of 3 to 4 can be catalyzed either by acids (CF_3COOH, H_3O^+) or bases (MeLi). However, the reaction rate depends on the nature of R^1 in the starting azabutadiene. For instance, when trifluoroacetic acid is added to a solution of 3 (R^1 = Ar) in ether or THF, the corresponding isoxazole is formed in nearly quantitative yield in 30 min, but in the case in which R^1 is an aliphatic group (cyclohexyl), the reaction goes to completion in 30 h. When 1 ($R^1 = c$-C_6H_{11}) reacts with 2 at 90 °C in pyridine solution, only compounds 3 are isolated. The formation of 4 from 3 was followed by ^1H NMR, and the existence of an enamine tautomer 3x could never be detected.

The most general method previously described for isoxazoles applies the condensation of a 1,3-diketone and hydroxylamine, but in the case of unsymmetrical diketones it leads to a mixture of regioisomers.[5] An unequivocal synthesis of unsymmetrical 3,5-diarylisoxazoles by reaction of 1,4-dianions from oximes and aromatic esters[6] has also been reported. However the method described herein is a simple, regiospecific and unequivocal general route from both diaryl- and alkylarylisoxazoles.[7] The high yield obtained in all instances and the availability of the starting materials make this synthesis one of the most convenient methods for the preparation of isoxazoles.

Experimental Section

General Procedures. Melting points were taken on samples in open capillary tubes in a Buchi melting point apparatus and are uncorrected. The NMR spectra were obtained on a Varian FT-80 NMR spectrometer using deuterated chloroform or deuterated Me_2SO as solvents, and shifts are reported in parts per million downfield (δ) from an internal tetramethylsilane (Me_4Si) standard. Infrared spectra were recorded in Nujol suspension on a Pye Unicam SP-1000 spectrophotometer. Mycroanalyses were performed on a Perkin-Elmer Model 240.

Isoxazoles 4. General Procedure. 3-Cyclohexyl-4-methyl-5-phenylisoxazole (4g). Hydroxylamine hydrochloride (0.7 g, 10 mmol) was added to a solution of 3-cyclohexyl-3-imino-2-methyl-1,*N*-diphenylprop-1-enamine (3.2 g, 10 mmol) in 60 mL of pyridine at room temperature, and the mixture was allowed to warm to 80–90 °C. After 4 h of stirring, the solution was slowly poured into ice-cooled 4 N H_2SO_4 (150 mL). The mixture was extracted with three 50-mL portions of ether and the combined organic layers were dried over anhydrous sodium sulfate, filtered, and evaporated. The residue was purified by recrystallization from hot hexane to afford 1.83 g (76%) of **4g**: ^1H NMR ($CDCl_3$, internal Me_4Si) δ 1.2–2.1 (m, 10 H), 2.2 (s, CH_3), 2.4–2.8 (m, CH), 7.3–7.8 (m, 5 H, Ar); ^{13}C NMR ($CDCl_3$, internal Me_4Si) δ 7.7 (c), 25.6, 25.9, 30.8 (t), 35.3 (d), 107.8 (s), 126.3 (d), 128.3 (d), 128.6 (s), 128.7 (d), 163.8 (s), 167.5 (s). Anal. Calcd for $C_{16}H_{19}NO$: C, 79.66; H, 7.88; N, 5.81. Found: C. 79.59; H, 7.62; N, 5.75 (see paragraph at the end of paper about supplementary material).

Products 3. General Procedure. 3-Anilino-2-methyl-3-phenyl-1-*p*-tolylprop-2-enal Oxime (3b). Hydroxylamine hydrochloride (0.7 g, 10 mmol) was added to a solution of 3-imino-2-methyl-1,*N*-diphenyl-3-*p*-tolylprop-1-enamine (3.2 g, 10 mmol) in 60 mL of pyridine and stirred at room temperature for 14 h. The solution was then acidified with ice-cooled 4 N H_2SO_4 (150 mL) and extracted with ether. The dry organic layer was

(5) B. J. Wakefield and D. J. Wright, *Adv. Heterocycl. Chem.*, **25**, 147 (1979).

(6) C. F. Beam, M. C. D. Dyer, R. A. Schwarz, and C. R. Hauser, *J. Org. Chem.*, **35**, 1806 (1970).

(7) We have synthesized original samples of isoxazoles **4d** by methods in the literature[6] and found that the melting point of our compounds and those of 1:1 mixtures with those samples were identical. On the other hand, the melting point of a 1:1 mixture of compounds **4h** and **4i** was found to be 20 °C less than that of **4i**. This mixture displayed duplicate signals for the isoxazole-ring carbon atoms in ^{13}C NMR.

(8) (a) C. Goldschmidt, *Chem. Ber.* **28**, 2540 (1895); (b) U. Turek and H. Behringer, *ibid.*, *98*, 3020 (1965); (c) The Aldrich Library of Infrared Spectra, Aldrich Chemical Company Inc., Milwaukee, WI, p 1069; (d) A. Battaglia, A. Dondoni, and E. Taddei, *J. Heterocycl. Chem.*, **7**, 721 (1970).

Scheme I

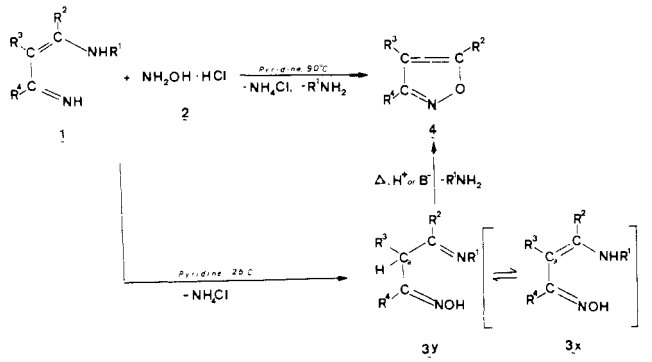

Table II. Compounds 3 from 1-Azabutadienes 1 and Hydroxylamine Hydrochloride 2[a]

3	R^1	R^2	R^3	R^4	yield, %	mp, °C
a	C_6H_5	C_6H_5	CH_3	C_6H_5	75	156-7*
b	C_6H_5	C_6H_5	CH_3	p-$CH_3C_6H_4$	83	170-3*
c	C_6H_5	C_6H_5	H	CH_3	76	131-4*
d	C_6H_5	p-ClC_6H_4	CH_3	C_6H_5	72	163-4*
e	C_6H_5	C_6H_5	H	c-C_6H_{11}	70	126-7*
f	p-$CH_3C_6H_4$	C_6H_5	CH_3	C_6H_5	75	156-7**
g	p-$CH_3C_6H_4$	C_6H_5	H	p-$CH_3C_6H_4$	87	164-5***
h	c-C_6H_{11}	C_6H_5	CH_3	C_6H_5	68	135-7*
i	c-C_6H_{11}	C_6H_5	CH_3	p-$CH_3C_6H_4$	80	120-2*

[a] See paragraph at the end of paper about supplementary material. * Recrystallized from ethanol. ** Recrystallized from hexane. *** Recrystallized from methanol.

evaporated and the residue recrystallized from ethanol to afford 2.5 g (83%) of **3b**: IR (Nujol) ν_{max} 1620, 3340 cm^{-1}; ^1H NMR ($CDCl_3$, internal Me_4Si) δ 0.8 (d, CH_3, J = 6 Hz), 2.4 (s, CH_3), 3.5 (c, 1 H), 4.9 (m, 1 OH), 6.5–7.8 (m, 14 H, Ar); ^{13}C NMR (Me_2SO-d_6, external Me_4Si) δ 15.2 (c), 21.1 (c), 51.9 (d), 100.3 (s), 116.2 (d), 117.5 (d), 126.3, 127.0, 128.0, 128.3, 128.5, 129.6, 138.4 (s), 140.0 (s), 144.8 (s), 162.3 (s). Anal. Calcd for $C_{23}H_{22}N_2O$: C, 80.7; H, 6.43; N, 8.18. Found: C, 80.55; H, 6.22; N, 8.03.

Conversions of oximes 3 into isoxazoles 4 were performed according the following procedures: (a) To a solution of **3** (5 mmol) in dry THF was added an 0.82 N ethereal solution of methyllithium (12 mL). After 1 h of stirring, the mixture was poured into ice-cooled 2 N H_2SO_4 and extracted with ether. The dry organic layer was evaporated and the residue recrystallized from hot hexane. (b) To a solution of **3** (5 mmol) in THF was added CF_3COOH (5 mmol) and the solution stirred for 30 min. Solvents were removed under reduced pressure, and the residue was recrystallized from hexane. (c) A solution of **3** (5 mmol) and 6 N H_2SO_4 (30 mL) in THF was stirred at 60 °C for 3 h, then poured into ice-cooled water, and extracted with ether. The dry organic layer was evaporated and the residue recrystallized from hexane. (d) A solution of **3** in pyridine was heated at 100 °C for 10 h. The resulting solution was poured into ice-cooled 4 N H_2SO_4, extracted with ether, dried over sodium sulfate, concentrated, and the residue recrystallized from hot hexane.

Registry No. **1a**, 71115-28-1; **1b**, 71115-31-6; **1c**, 71443-42-0; **1d**, 78946-76-6; **1e**, 84512-70-9; **1f**, 71115-24-7; **1g**, 72923-07-0; **1h**, 72923-06-9; **1i**, 71115-32-7; **1j**, 72923-09-2; **1k**, 71115-34-9; **2**, 7803-49-8; **3a**, 84850-51-1; **3b**, 84850-52-2; **3c**, 84850-53-3; **3d**, 84850-54-4; **3e**, 84850-55-5; **3f**, 84850-56-6; **3g**, 84850-57-7; **3h**, 84850-58-8; **3i**, 84850-59-9; **4a**, 10557-77-4; **4b**, 2039-49-8; **4c**, 1008-75-9; **4d**, 29329-38-2; **4e**, 16112-19-9; **4f**, 84850-60-2; **4g**, 84850-61-3; **4h**, 84850-62-4; **4i**, 84850-63-5; **4j**, 84850-64-6; **4k**, 84850-65-7.

Supplementary Material Available: Complete ^1H NMR and ^{13}C NMR data for compounds **4** and complete IR, ^1H NMR, and ^{13}C NMR data for compounds **3** (4 pages). Ordering information is given on any current masthead page.

Selective Preparation of Mono- and Diacetals of D-Mannitol

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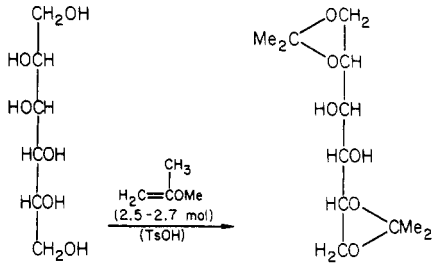
The 1,2:5,6-diisopropylidene acetal (**2**) of D-mannitol (**1**) is an important chiral precursor for natural-product synthesis; on glycol cleavage it is converted into 2 mol of 2,3-*O*-isopropylidene-D-glyceraldehyde.[1] This optically pure D-glyceraldehyde derivative is a convenient and widely used starting point for numerous syntheses[2] of optically active lipids, of various sugars, and of many non-carbohydrate, chiral molecules.

Conventional, acid-catalyzed reaction of D-mannitol (**1**) with acetone under equilibrium conditions gives[1] only modest (~40%) yields of the diacetal **2** in a rather tedious procedure that uses large quantities of solvents. Addition of 2,2-dimethoxypropane to the reagent mixture facilitates the procedure and gives **2** in 31% yield,[3] and use of 2,2-dimethoxypropane in 1,2-dimethoxyethane allows **2** to be obtained in 54–58% yield.[4]

Kinetic acetonation by use of 2-alkoxypropenes, as developed in our laboratories,[5,6] was evaluated for its practical potential in preparation of diacetal **2** from the alditol **1**.



It is shown here that the method provides a simple, large-scale procedure that gives **2** in 92% yield from **1**. Furthermore, the technique may be adapted to furnish a preparative route to the corresponding monoacetal, 1,2-*O*-isopropylidene-D-mannitol (**3**), obtainable from **1** in ~70% yield and itself also a valuable synthetic precursor.

Previous work has shown[5,6] that 2-alkoxypropenes react with sugars in *N*,*N*-dimethylformamide in the presence of a trace of acid under exclusively kinetic control; favored initial attack takes place at primary hydroxyl groups, if present, and ring-closure to stable cyclic acetals is dictated by the availability of suitably disposed hydroxyl groups

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