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Aminocyclitols. X. A Synthesis of Deoxyinosamine and Deoxyinosadiamine and Their Nuclear Magnetic Resonance Spectra

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(±)-1-Deoxy-*muco*-inosamine-4, (±)-1-deoxy-*scyllo*-inosamine-2 and (±)-2-deoxy-*allo*-inosadiamine-3, 5 pentaacetates have been synthesized from 1-deoxy-*myo*-inositol (I). The hydroxyl groups of I are mesylated, and the mesyloxy groups are replaced by azido groups. The reduction of the azido compounds, followed by acetylation, gives the pentaacetyl-deoxyinosamines and deoxyinosadiamine. Their structures have been established by means of a study of the proton magnetic resonance spectra.

Although six deoxyinosamines have been described[1,6] recently, only one deoxyinosadiamine

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[1] M. Nakajima, A. Hasegawa and F. W. Lichtenthaler, *Ann.*, **680**, 21 (1964).

(deoxystreptamine) has been isolated from a biological origin (neomycin,[2] kanamycin,[3] paromomycin[4] and hygromycin B[5]).

In connection with previous papers of this series,[7] new deoxyinosamines and the deoxyinosadiamine have been synthesized, and their structures have been established by means of a study of the proton magnetic resonance spectra. In the present paper, (±)-1-deoxy-*myo*-inositol [(±)-*vibo*-quercitol] (I) is prepared by the method of McCasland et al.,[8] and then used as a starting material.

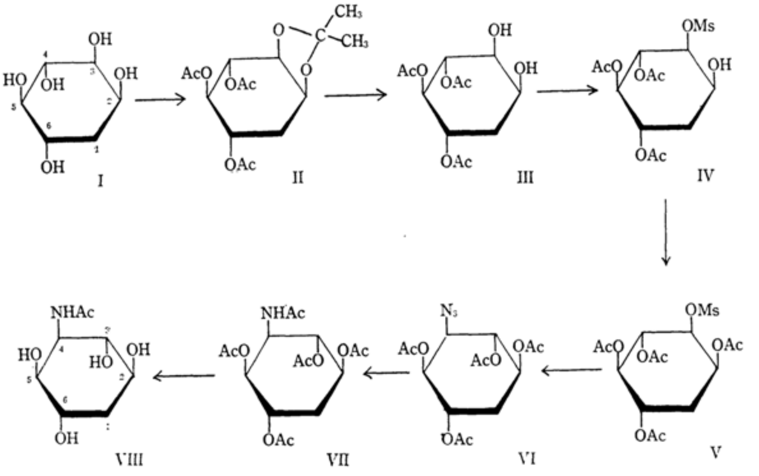
(±)-1-Deoxy-*muco*-inosamine-4.—I is heated in acetone in the presence of anhydrous zinc chloride and glacial acetic acid, and the product is subsequently acetylated to give (±)-4, 5, 6-tri-*O*-acetyl-1-deoxy-2, 3-isopropylidene-*myo*-inositol (II) in a 66% yield. Since the *O*-isopropylidene derivative of a vicinal glycol group on a six-membered ring is formed much more easily when the relationship between the hydroxyl group is cis than when trans,[9] the structure of II is assigned as described above; this assignment is confirmed by the further synthesis of the present study.

The *O*-isopropylidene group is easily removed by heating II in 80% aqueous acetic acid thus giving (±)-4, 5, 6-tri-*O*-acetyl-1-deoxy-*myo*-inositol (III) in a 55% yield. When III is reacted with one mole of methanesulfonyl chloride, (±)-4, 5, 6-tri-*O*-acetyl-1-deoxy-3-*O*-mesyl-*myo*-inositol (IV) is obtained in a 51% yield. IV is acetylated to give the tetra-*O*-acetyl derivative (V).

In a previous paper,[10] it has been described that methanesulfonyl chloride attacks an equatorial hydroxyl group selectively, rather than an axial one, in the *myo*-inositol derivative. Therefore, in the *vibo*-quercitol derivative, methanesulfonyl chloride is expected to behave in the same manner. This can be proved by establishing a structure of V, and the NMR spectrum gives an unequivocal confirmation of the structure. The NMR spectrum of V in deuteriochloroform, as Table I shows, exhibits a sharp signal at 6.99 τ which is ascribed to an equatorial mesyloxy group.[10] At 7.83, 7.93 and 7.97 τ, three sharp signals with a 1 : 1 : 2 intensity ratio appear; these signals are expected from one axial acetoxy group and three equatorial acetoxy ones.[11]

When V is treated with sodium azide in boiling aqueous 2-methoxyethanol for 51 hr., and subsequently acetylated with acetic anhydride in pyridine, (±)-4-azido-1, 4-dideoxy-*muco*-inositol tetraacetate (VI) is obtained in a 28% yield. The reduction of VI with Raney nickel in a hydrogen stream and subsequent acetylation give (±)-pentaacetyl-1-deoxy-*muco*-inosamine-4 (VII) in a 58% yield.

The structure of VII is substantiated by its NMR spectrum. The NMR spectrum in deuteriochloroform, as is given in Table I, exhibits three sharp signals, with a 2 : 1 : 1 intensity ratio, at 7.85, 7.96 and 7.98 τ; these signals correspond to two axial acetoxy groups (7.85 τ) and two equatorial ones (7.96 and 7.98 τ). There is also a sharp signal



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[2] D. A. Kuehl, Jr., M. N. Bishop and K. Folkers, *J. Am. Chem. Soc.*, **73**, 881 (1951).

[3] K. Maeda, M. Ueda, K. Yagishita, S. Kawaji, S. Kondo, M. Murase, T. Takeuchi, Y. Okami and H. Umezawa, *J. Antibiotics*, **A10**, 228 (1957).

[4] T. H. Haskell, J. C. French and Q. R. Bartz, *J. Am. Chem. Soc.*, **81**, 3480 (1959).

[5] P. F. Wiley, M. V. Sigal, Jr., and O. Weaver, *J. Org. Chem.*, **27** 2793 (1962).

[6] M. Nakajima, A. Hasegawa and N. Kurihara, *Tetrahedron Letters*, **17**, 697 (1964).

[7] T. Suami and S. Ogawa, This Bulletin, **37**, 1238 (1964); T. Suami and K. Yabe, ibid., **38**, 855 (1965); T. Suami, F. W. Lichtenthaler and S. Ogawa, ibid., **39**, 170 (1966).

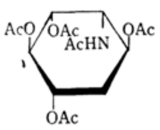
[8] G. E. McCasland and E. Clyde Horswill, *J. Am. Chem. Soc.*, **75**, 4020 (1953).

[9] R. U. Lemieux, "Rearrangements and Isomerizations in Carbohydrate Chemistry," in "Molecular Rearrangements," Vol. II, Interscience Publishers, New York (1964), p. 731.

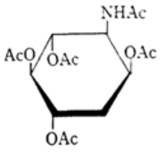
[10] T. Suami, F. W. Lichtenthaler and S. Ogawa, This Bulletin, **38**, 754 (1965).

[11] F. W. Lichtenthaler, *Chem. Ber.*, **96**, 2047 (1963).

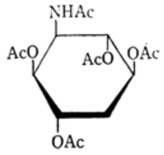
at 8.09 τ which is expected to be from an equatorial acetamido group. Considering the reaction process employed, there are three possible structures for VII: a, b, and c.



a

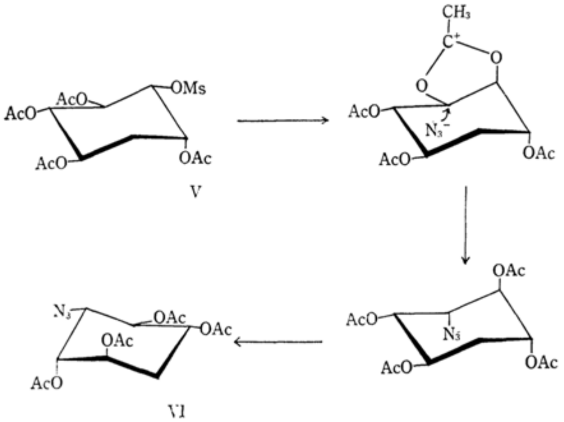


b



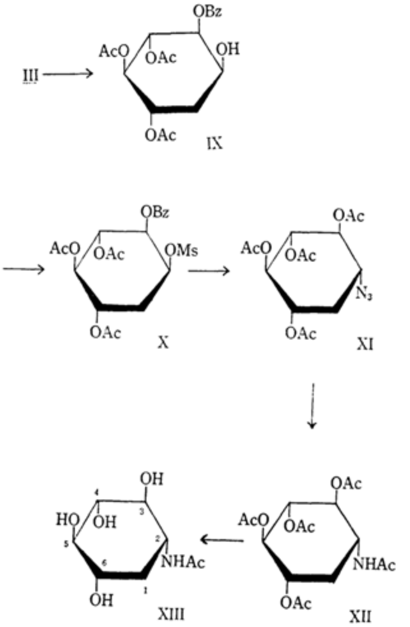
c

If the demesylation took place via a direct S_N2 mechanism, a would be obtained. b and c are anticipated only from an anchimeric mechanism, a neighboring-group participation. Since there is only one axial acetoxy group in both a and b, however a and b can be eliminated, and only c is compatible with the NMR spectrum. The possible mechanism of the reaction from V to VI is shown in the following scheme:



The elimination of the mesyloxy group takes place with a neighboring-group participation to form an intermediate acetoxonium ion, and then it is cleaved by an azide ion in a diaxial opening, as has been already observed in the replacement of the mesyloxy group in *myo*-inositol derivatives.7)

(±)-1-Deoxy-*scyllo*-inosamine-2.—In analogy to the monomesylation of triacetyl deoxyinositol (III→IV), a selective monobenzoylation to the 3-*O*-benzoyl derivative should be possible under conditions analogous to those described in a previous paper.7) When III is treated with one mole of benzoyl chloride in pyridine under ice cooling, the tri-*O*-acetyl-3-*O*-benzoyl derivative (IX) is obtained in a 58% yield. The mesylation of IX by an ordinary method gives (±)-4, 5, 6-tri-*O*-acetyl-3-*O*-benzoyl-1-deoxy-2-*O*-mesyl-*myo*-inositol (X) in a 87% yield. X is quite different in its physical properties from the (±)-4, 5, 6-tri-*O*-acetyl-2-*O*-benzoyl-1-deoxy-3-*O*-mesyl-*myo*-inositol (XIV) prepared by the benzoylation of IV. The NMR spectrum of X (Table I) reveals a sharp signal at 6.97 τ corresponding to an axial mesyloxy group, while that of XIV (Table I) exhibits a sharp signal shifted to a slightly higher field (6.99 τ), as is to be expected from an equatorial mesyloxy group. Therefore, this presents another example of how benzoyl chloride attacks an equatorial hydroxyl group preferentially.7)



Then X is treated with sodium azide as described above to give an azido compound (XI), which is hydrogenated and subsequently acetylated to yield (±)-pentaacetyl-1-deoxy-*scyllo*-inosamine-2 (XII). The NMR spectrum of XII, as is shown in Table I, exhibits two sharp signals, with a 2 : 2 relative intensity, at 8.02 and 8.06 τ; these signals correspond to four equatorial acetoxy groups. There is also a sharp signal at 8.22 τ which is to be ascribed to an equatorial acetamido group.

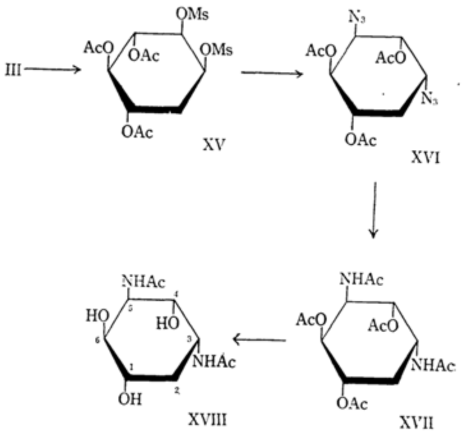
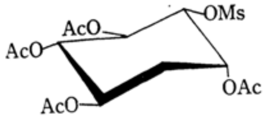
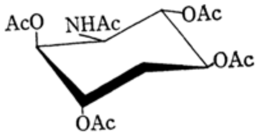
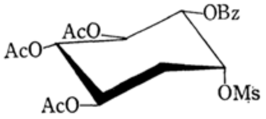
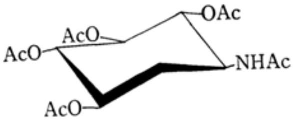
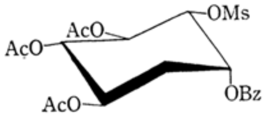
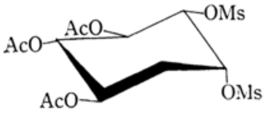
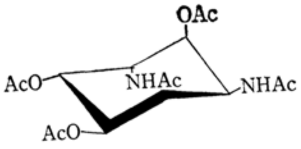
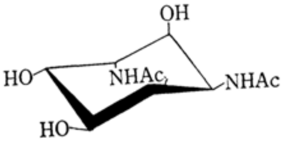


TABLE I. SIGNALS OF PROTON MAGNETIC RESONANCE SPECTRA

Compound	Solvent	Protons of mesyloxy group		Protons of acetoxy group		Protons of acetamido group	
		axial	equat.	axial	equat.	axial	equat.
V. 	$CDCl_3$		6.99(1)	7.83(1)	7.93(1) 7.97(2)		
VII. 	$CDCl_3$			7.85(2)	7.96(1) 7.98(1)		8.09(1)
X. 	$CDCl_3$	6.97(1)			7.96(2) 8.06(1)		
XII. 	$(CD_3)_2SO$				8.02(2) 8.06(2)		8.22(1)
XIV. 	$CDCl_3$		6.99(1)		7.94(1) 7.95(1) 8.00(1)		
XV. 	$CDCl_3$	6.82(1)	6.89(1)		7.92(1) 7.97(2)		
XVII. 	$(CD_3)_2SO$			7.90(1)	8.02(1) 8.13(1)	8.13(1)	8.23(1)
XVIII. 	D_2O					7.92(1)	7.98(1)

Peak positions are given in τ values, and numbers in parentheses are relative intensities of the signals.

(±)-2-Deoxy-*allo*-inosadiamine-3, 5.—While III is treated with an excess amount of methanesulfonyl chloride in pyridine, the di-*O*-mesyl derivative (XV) is obtained in a 85% yield. The NMR spectrum of XV in deuteriochloroform, as is shown in Table I, exhibits two sharp signals, with equal intensities, at 6.82 and 6.89 τ for the protons of an axial and an equatorial mesyloxy group respectively.[10] Two sharp signals with a 1 : 2 relative intensity at 7.92 and 7.97 τ correspond to three equatorial acetoxy groups.[11]

When XV is heated with sodium azide in boiling aqueous 2-methoxyethanol for 41 hr. and subsequently acetylated by an ordinary method, diazido acetate (XVI) is obtained in a 17% yield. XVI is then hydrogenated in the presence of a catalyst in a hydrogen stream, and the reduction product is acetylated to afford pentaacetyl-deoxyinosadiamine (XVII) in a 68% yield. A selective de-*O*-acylation of XVII with ammonia in methanol yields the diacetamido derivative (XVIII). The structure of XVII is substantiated by the NMR spectra of XVII in deuteriodimethyl sulfoxide and of XVIII in deuterium oxide. The NMR spectrum of XVIII, as is shown in Table I, shows two sharp signals, with equal intensities, at 7.92 and 7.98 τ, corresponding to an axial and an equatorial acetamido group respectively. The NMR spectrum of XVII, as may be seen in Table I, exhibits four sharp signals, at 7.90, 8.02, 8.13 and 8.23 τ (1 : 1 : 2 : 1), for the protons in an axial and an equatorial acetoxy group, another equatorial acetoxy group overlapped with an axial acetamido group, and an equatorial acetamido group respectively. Therefore, it might be reasonable to identify the structure of XVII as (±)-2-deoxy-*allo*-inosadiamine-3, 5. Considering the product obtained, the following reaction mechanism might be assumed: a mesyloxy group on C-3 is replaced by the participation of the neighboring acetoxy group in a trans position, thus forming an intermediate acetoxonium ion, and then the dioxolane-ring is attacked by a nucleophilic azide ion. The manner in which the ring is cleaved is considered to be the same diaxial opening described above and also observed in previous papers.[7,12] On the other hand, another mesyloxy group on C-2 might be replaced by an azide ion through a direct S_N2 mechanism. The behavior of the latter mesyloxy group is different from that of the mesyloxy group in an inositol derivative in an analogous situation;[7] this point must be studied further.

Experimental

The melting points were determined on a Mitamura Riken hot stage. The melting points marked with asterisks were measured in a liquid bath and are uncorrected. The NMR spectra of the samples were determined at a frequency of 60 Mc. p. s. with a Japan Electron Optics JNM-C-60 spectrometer in deuterium oxide, deuteriochloroform and deuteriodimethyl sulfoxide, using sodium trimethylsilylpropane-sulfonate or tetramethylsilane as an internal standard.

(±)-1-Deoxy-*myo*-inositol (I).—This substance was prepared from *myo*-inositol in a 30.4% yield by the method developed by McCasland et al.[8]

(±)-4, 5, 6-Tri-*O*-acetyl-1-deoxy-2 : 3 - isopropylidene-*myo*-inositol (II).—A mixture of 10 g. of I, 50 g. of anhydrous zinc chloride, 50 ml. of glacial acetic acid and 400 ml. of acetone was heated to reflux for 36 hr. After the mixture had then cooled, 100 ml. of pyridine was added to the mixture and the precipitate was removed by filtration. The filtrate was evaporated under reduced pressure and the residue was acetylated overnight in a mixture of 60 ml. of acetic anhydride and 40 ml. of pyridine. After the precipitate had been removed, 150 ml. of chloroform was added to the filtrate. Then the solvent layer was washed with cold water, a 5% sodium carbonate solution and cold water successively. The solvent was evaporated, after drying over anhydrous sodium sulfate, to give 22 g. of an oily residue. The resiude was added to 300 ml. of ligroin and kept in a refrigerator overnight, thus yielding 14.7 g. of the crude product. The crude product was recrystallized from petroleum ether to give 12.8 g. (66.4%) of needles. m. p. *80—82°C. An analytical sample was obtained by a further recrystallization from petroleum ether. m. p. *82—83°C.

Found: C, 54.31; H, 6.62. Calcd. for $C_{15}H_{22}O_8$: C, 54.54; H, 6.71%.

(±)-4, 5, 6-Tri-*O*-acetyl - 1 - deoxy - *myo* - inositol (III).—A mixture of 2.3 g. of II and 100 ml. of 80% acetic acid was heated on a boiling water bath for two hours, and then evaporated under reduced pressure. The solid residue was recrystallized from ethanol to yield 1.3 g. of a crude product. The product was recrystallized from 5 ml. of ethanol, thus affording 1.1 g. (54.5%) of an analytically-pure product melting at 170—171°C.

Found: C, 49.70; H, 6.21. Calcd. for $C_{12}H_{18}O_8$: C, 49.65; H, 6.25%.

(±)-4, 5, 6-Tri-*O*-acetyl-1-deoxy- 3 -*O*-mesyl-*myo*-inositol (IV).—To a solution of 5.1 g. of III in 50 ml. of pyridine, 2.0 g. of methanesulfonyl chloride was added drop by drop under agitation at 0—5°C. After the solution had been stirred for two hours, it was left to settle in a refrigerator overnight. The crystals were then collected by filtration, 3.3 g. (50.9%) of the crude product, m. p. 131—131.5°C were thus obtained. An analytical sample was obtained by recrystallization from ethanol. m. p. 131—132°C.

Found: C, 42.54; H, 5.55; S, 8.31. Calcd. for $C_{13}H_{20}O_{10}S$: C, 42.39; H, 5.47; S, 8.70%.

(±)-2, 4, 5, 6-Tetra-*O*-acetyl-1-deoxy - 3 - *O*-mesyl-*myo*-inositol (V).—A 1.6 g. portion of IV was acetylated with acetic anhydride and pyridine. The crude product was recrystallized from ethanol to give 1.7 g. (95.4%) of crystals melting at *171—172°C.

Found: C, 43.97; H, 5.33; S, 7.78. Calcd. for $C_{15}H_{22}O_{11}S$: C, 43.90; H, 5.40; S, 7.81%.

(±)-2, 3, 5, 6-Tetra-*O*-acetyl-4-azido-1, 4-dideoxy - *muco*-inositol (VI).—A mixture of 0.5 g. of V, 0.25 g. of sodium azide and 30 ml. of 80% aqueous 2-methoxyethanol was heated to reflux for 51 hr. After the solvent had then been removed, a residue was acetylated with acetic anhydride and pyridine. The excess of the reagent was removed, and the residue was dissolved in 2 ml. of ethanol to yield 0.21 g. of the crude product. The crude product was recrystallized from ethanol to give 0.12 g. (27.6%) of crystals melting at 143—144°C.

Found: C, 47.28; H, 5.38; N, 11.63. Calcd. for $C_{14}H_{19}N_3O_8$: C, 47.06; H, 5.36; N, 11.76%.

(±)- Pentaacetyl - 1 - deoxy - *muco* - inosamine-4 (VII).—A 0.30 g. portion of VI was hydrogenated in 50 ml. of ethanol with Raney nickel T4[13] under a stream of hydrogen (50 p. s. i. g.). After the catalyst had then been removed, the solution was evaporated under reduced pressure. The residue was acetylated to yield 0.22 g. of the product. The crude product was recrystallized from ethanol to give 0.18 g. (57.5%) of plates melting at *215—216°C.

Found: C, 51.63; H, 6.38; N, 3.80. Calcd. for $C_{16}H_{23}NO_9$: C, 51.47; H, 6.21; N, 3.75%.

The hydrolysis of VII with 6 N hydrochloric acid for 6 hr. at 100°C gave the hydrochloride, which showed

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[12] T. Suami and S. Ogawa, This Bulletin, **38**, 2026 (1965).

[13] S. Nishimura, This Bulletin, **32**, 61 (1959).

a single spot of R_f 0.25 (R_f of D-glucosamine hydrochloride: 0.27) in ascending paper chromatography in an acetic acid-ethyl acetate-pyridine-water (1 : 5 : 5 : 3) system[14] at 18—20°C. R_f/R_f glucosamine: 0.94.

(±)-*N*-Acetyl-1-deoxy-*muco*-inosamine-4 (VIII).—The selective de-*O*-acetylation of 0.63 g. of VII in methanol saturated with ammonia gave 0.17 g. of a crude product. The product was recrystallized from ethanol to yield 0.11 g. (32.6%) of crystals melting at 192—193°C.

Found: C, 46.61; H, 7.54; N, 6.69. Calcd. for $C_8H_{15}NO_5$: C, 46.82; H, 7.37; N, 6.83%.

(±)-4, 5, 6-Tri-*O*-acetyl-3-*O*-benzoyl-1-deoxy-*myo*-inositol (IX).—To a solution of 6.3 g. of III in 40 ml. of pyridine, 3.3 g. of benzoyl chloride was added drop by drop under ice cooling with agitation. After the mixture had settled at room temperature overnight, it was poured into 300 ml. of ice-cold water to give a precipitate which was then collected by filtration. The crude product was recrystallized from 100 ml. of ethanol to give 5.0 g. (58.2%) of crystals melting at *185—187°C.

Found: C, 57.85; H, 5.54. Calcd. for $C_{19}H_{22}O_9$: C, 57.86; H, 5.62%.

(±)-4, 5, 6-Tri-*O*-acetyl-3-*O*-benzoyl-1-deoxy-2-*O*-mesyl-*myo*-inositol (X).—A 3.1 g. portion of methanesulfonyl chloride was added to a solution of 7.1 g. of IX in 50 ml. of pyridine under ice cooling with agitation. Then the mixture was poured into ice-cold water to give 7.8 g. of a precipitate. The crude product was recrystallized from ethanol to give 7.4 g. (87.0%) of plates melting at 175—176°C.

Found: C, 50.65; H, 5.19; S, 6.83. Calcd. for $C_{20}H_{24}O_{11}S$: C, 50.84; H, 5.12; S, 6.79%.

(±)-3, 4, 5, 6-Tetra-*O*-acetyl-2-azido-1, 2-dideoxy-*scyllo*-inositol (IX).—A mixture of 2.2 g. of X, 0.6 g. of sodium azide and 60 ml. of 80% aqueous 2-methoxyethanol was heated to reflux for 64 hr. Then the mixture was treated as has been described in the preparation of VI to give 1.0 g. of the crude product, which was then recrystallized from ethanol, giving 0.9 g. (55.1%) of needles melting at 137—138°C.

Found: C, 47.28; H, 5.35; N, 11.89. Calcd. for $C_{14}H_{19}N_3O_8$: C, 47.06; H, 5.36; N, 11.76%.

(±)-Pentaacetyl-1-deoxy-*scyllo*-inosamine-2 (XII).—A 0.95 g. portion of XI was hydrogenated in 30 ml. of ethanol with Raney nickel T4,[13] and then the reduction product was treated as has been described in the preparation of VII to give 0.51 g. of the crude product melting at 226—227°C. The crude product was recrystallized from ethanol to give 0.32 g. (32.7%) of crystals melting at 226—227°C.

Found: C, 51.57; H, 6.03; N, 3.61. Calcd. for $C_{16}H_{23}NO_9$: C, 51.47; H, 6.21; N, 3.71%.

Paper Chromatography.—The hydrochloride of XII was prepared and developed in paper chromatography using the procedure described above. It showed a single spot of R_f 0.14. R_f/R_f glucosamine: 0.52.

(±)-*N*-Acetyl-1-deoxy-*scyllo*-inosamine-2 (XIII).—The selective de-*O*-acetylation of 0.22 g. of XII by ammonia in methanol yielded 0.10 g. of the crude product, which was then recrystallized from a mixture of benzene and methanol to give 0.08 g. (72.7%) of crystals melting at 205—206°C.

Found: C, 47.31; H, 7.43; N, 6.63. Calcd. for $C_8H_{15}NO_5$: C, 46.82; H, 7.37; N, 6.83%.

(±)-4, 5, 6-Tri-*O*-acetyl-2-*O*-benzoyl-1-deoxy-3-*O*-mesyl-*myo*-inositol (XIV).—A 0.6 g. portion of benzoyl chloride was added to a solution of 0.78 g. of IV in 20 ml. of pyridine; the mixture was then warmed for 30 min. and left to stand overnight at room temperature. Then the mixture was poured into ice-cold water to give 0.29 g. of needles melting at 151—152°C. For analysis the product was recrystallized twice from ethanol; yield, 0.17 g. (17.0%); m. p. 159—160°C.

Found: C, 50.97; H, 4.95; S, 6.39. Calcd. for $C_{20}H_{24}O_{11}S$: C, 50.84; H, 5.12; S, 6.79%.

(±)-4, 5, 6-Tri-*O*-acetyl-1-deoxy-2, 3-di-*O*-mesyl-*myo*-inositol (XV).—To a solution of 5.7 g. of III in 50 ml. of pyridine, 5.7 g. of methanesulfonyl chloride was added drop by drop with agitation at 0—5°C. After settling overnight in a refrigerator, the solution was poured into one liter of ice-cold water to obtain crystals, which were then collected by filtration. Yield, 7.4 g. (84.6%); m. p. 179—182°C. An analytical sample was obtained by recrystallizations from ethyl acetate. m. p. 181—182°C.

Found: C, 37.86; H, 4.80; S, 14.53. Calcd. for $C_{14}H_{22}O_{12}S_2$: C, 37.66; H, 4.97; S, 14.36%.

(±)-1, 4, 6-Tri-*O*-acetyl-3, 5-diazido-2, 3, 5-trideoxy-*allo*-inositol (XVI).—A mixture of 1.35 g. of XV, 1.2 g. of sodium azide and 40 ml. of 80% aqueous 2-methoxyethanol was heated to reflux for 41 hr. Then the mixture was treated as has been described in the preparation of VI to give 0.17 g. (16.5%) of crystals melting at 133—133.5°C.

Found: C, 42.61; H, 4.80; N, 24.45. Calcd. for $C_{12}H_{16}N_6O_6$: C, 42.35; H, 4.74; N, 24.70%.

(±)-Pentaacetyl-2-deoxy-*allo*-inosadiamine-3, 5 (XVII).—A 1.03 g. portion of XVI was hydrogenated much as has been described in the preparation of VII to give 0.70 g. (67.6%) of crystals melting at *290—291°C.

Found: C, 51.40; H, 6.49; N, 7.43. Calcd. for $C_{16}H_{24}N_2O_8$: C, 51.60; H, 6.50; N, 7.52%.
XVII was hydrolyzed with 6 N hydrochloric acid, and the hydrochloride was developed by paper chromatography. The hydrochloride showed a single spot of R_f 0.14 at 18—20°C. R_f/R_f glucosamine: 0.52.

(±)-*N*, *N'*-Diacetyl-2-deoxy-*allo*-inosadiamine-3, 5 (XVIII).—The selective deacetylation of 0.53 g. of XVII as has been described in the preparation of VIII gave 0.16 g. (45.3%)of needles melting at 251—252°C.

Found: C, 49.00; H, 7.12; N, 11.25. Calcd. for $C_{10}H_{18}N_2O_5$: C, 48.77; H, 7.37; N, 11.38%.

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