267. *Cyclitols. Part IV.* Methyl Ethers of* myo*Inositol.*†

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Dambonitol is shown to be 1 : 3-di-*O*-methyl*myo*inositol and bornesitol to be one of the enantiomers of the 1-methyl ether. Methylation of *myo*-inositol has given the (±)-1- and the 2-methyl derivative. Dambonitol acetate has been synthesised by methylation of 1 : 4 : 5 : 6-tetra-*O*-acetyl-*myo*inositol which is accompanied by acetyl migration.

*myo*Inositol (I) can give rise to four structurally different monomethyl ethers: the symmetrical 2- and 5-compound and the resolvable 1- and 4-derivative. Three of these ethers have so far been found in Nature: the inactive sequoyitol, the dextrorotatory ononitol, and bornesitol which occurs in both enantiomorphous forms. Several synthetic monomethyl ethers have also been reported. Two dimethyl ethers, the inactive dambonitol and the active liriodendritol, have been isolated from natural sources. The present paper discusses the structure of these compounds except that of the very recently described liriodendritol.[2]

Sequoyitol occurs in the heartwood of *Sequoia sempervirens*,[3] in the sugar pine [4] (*Pinus lambertiana*), and in *Macrozamia riedlei*.[5] Anderson *et al.*[6] have shown that sequoyitol is 5-*O*-methyl*myo*inositol and we have confirmed this conclusion by a synthesis which will be reported in a subsequent publication. Foster,[7] using a sample of sequoyitol isolated from *Macrozamia riedlei* by Riggs, found that it gave two spots on paper ionophoresis in borate buffer and suggested that both the 2- and the 5-methyl ether were present. Our sample, also a generous gift from Dr. Riggs, showed the same behaviour

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† The prefix *myo* is used [1] instead of the older, non-specific *meso*, to describe the 1 : 2 : 3 : 5/4 : 6-inositol.

[1] Fletcher, Anderson, and Lardy, *J. Org. Chem.*, 1951, **16**, 1238.
[2] Plouvier, *Compt. rend.*, 1955, **241**, 765.
[3] Sherrard and Kurth, *J. Amer. Chem. Soc.*, 1929, **51**, 3139.
[4] Anderson and Ballou, *ibid.*, 1953, **75**, 648.
[5] Riggs, *J.*, 1949, 3199; *Austral. J. Chem.*, 1954, **7**, 123.
[6] Anderson, Deluca, Bieder, and Post, *J. Amer. Chem. Soc.*, in the press.
[7] Foster, *Chem. and Ind.*, 1953, 592.

but we found that the second spot was due to an impurity, macrozamin; after purification it was ionophoretically homogeneous and identical with a sample of Sherrard and Kurth's original sequoyitol [3] (obtained by the courtesy of Dr. A. J. Stamm).

(+)-Bornesitol was isolated from " Borneo rubber " by Girard,[8] from commercial rubber of unidentified origin by Flint and Tollens,[9] and, more recently, from opepe wood; [10] the (−)-isomer has also been found [11] in Nature. An attempt to find the " Borneo rubber " led us to *Dyera lowii*, the only latex-producing tree which is nowadays tapped in Borneo; however, it was found to contain dambonitol (see below). Foster and Stacey [12] have claimed that bornesitol is one of the enantiomers of 1-*O*-methyl*myo*inositol because its ionophoretic mobility in borate buffer is much less than that of *myo*inositol; this was taken as evidence that one of the pairs of *cis*-hydroxyl groups is blocked by the methyl group. It was later found,[7] however, that sequoyitol—with all its *cis*-hydroxyls free—also moves much more slowly than *myo*inositol; and that some cyclitols devoid of *cis*-1 : 2-diol groups show ionophoretic mobility.[13] Foster and Stacey's argument is therefore not conclusive; nevertheless, as will be shown in this paper, the 1-*O*-methyl structure postulated by them is correct.

Ononitol has recently been isolated by Plouvier; [11] since it is optically active it must be one of the enantiomers of 4-*O*-methyl*myo*inositol. This conclusion has been confirmed by the synthesis of its racemate (to be reported later).

An obvious way of preparing a structurally well-defined methyl ether would be by the methylation of 1 : 3 : 4 : 5 : 6-penta-*O*-acetyl*myo*inositol.[14] Our experiments, using methyl iodide and silver carbonate, failed to introduce a methyl group into this compound. Employing silver oxide and a higher temperature, Anderson and Landel [15] obtained a methyl ether which, however, proved to be structurally identical with bornesitol. Apparently, under the effect of the alkaline silver oxide, acetyl migration occurred setting up an equilibrium between two penta-acetates, and the equatorial 1-hydroxyl group of the one was methylated in preference to the axial 2-hydroxyl group of the other.

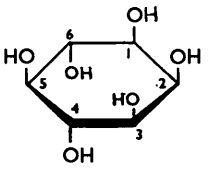
Preparation of a methyl ether by direct methylation of *myo*inositol was described by Griffin and Nelson.[16] Repetition of their experiments gave a complex mixture from which two monomethyl ethers were isolated in poor yield. One isomer, m. p. 200°, presumably identical with Griffin and Nelson's compound, m. p. 204°, has been shown to be structurally identical with bornesitol by comparison of their R_F values in four solvent systems, of their ionophoretic mobility in borate buffer, and of the infrared spectra of their penta-acetates. The infrared spectrum of each penta-*O*-acetyl-*O*-methyl*myo*inositol is different. This isomer is therefore the racemate of bornesitol, (±)-1-*O*-methyl*myo*inositol; it was identical with Anderson and Landel's methyl ether.[15] The other isomer, m. p. 237°, had an infrared spectrum and ionophoretic mobility different from those of sequoyitol, bornesitol, and ononitol; it must therefore be the 2-methyl ether. All the structurally possible monomethyl ethers of *myo*inositol are therefore now known.

At the time these experiments were performed (1951) the separations were carried out by fractional crystallisations and we made no use yet of chromatography. Later, sequoyitol was isolated from one of the fractions by chromatography on cellulose powder.

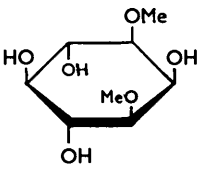
Dambonitol, which occurs in a variety of latex-producing trees, has recently been isolated from *Dyera costulata* and *D. lowii* by Comollo and Kiang [17] and, independently, by us. Comollo and Kiang proposed the 2 : 5-dimethyl structure, for they found that

[8] Girard, *Compt. rend.*, 1871, **73**, 426.
[9] Flint and Tollens, *Annalen*, 1892, **272**, 288.
[10] King and Jurd, *J.*, 1953, 1192.
[11] Plouvier, *Compt. rend.*, 1955, **241**, 983.
[12] Foster and Stacey, *Chem. and Ind.*, 1953, 279.
[13] Angyal and McHugh, following paper.
[14] Iselin, *J. Amer. Chem. Soc.*, 1949, **71**, 3822; May, *J. Org. Chem.*, 1952, **17**, 286.
[15] Anderson and Landel, *J. Amer. Chem. Soc.*, 1954, **76**, 6130.
[16] Griffin and Nelson, *ibid.*, 1915, **37**, 1552.
[17] Comollo and Kiang, *J.*, 1953, 3319.

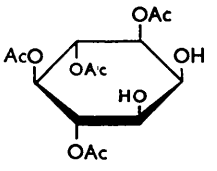
dambonitol would not react with acetone and that it consumed two mols. of periodate without the production of formic acid. After communication of our conflicting results to them, Kiang and Loke [18] re-investigated the periodate oxidation and found that one mol. of formic acid was produced, albeit slowly. Similar results were obtained by Anderson and Drummond.[19] The periodate oxidation indicates a *m*-dimethoxy-structure, and Kiang and Loke concluded that dambonitol is the 1 : 3-dimethyl ether. Their proof, however, depends on negative evidence, *i.e.*, that an *iso*propylidene compound was not obtained; in view of the slowness of the reaction of *myo*inositol with acetone (see below), this cannot be regarded as definite proof for the absence of a *cis*-1 : 2-glycol system.



(I)



(II)



(III)

Dambonitol shows no ionophoretic mobility in 0·15M-borate buffer. This is evidence for the absence of *cis*-1 : 2-diol groups since all cyclitols possessing such a group move under these conditions.[13] Dambonitol must therefore have one methoxyl group on $C_{(2)}$ or the two methoxyl groups on $C_{(1)}$ and $C_{(3)}$. Partial demethylation gave *only one* monomethyl ether, (±)-bornesitol. These facts, taken in conjunction with the *meso*-nature of the compound and the evidence from periodic acid oxidations, prove that dambonitol is 1 : 3-di-*O*-methyl*myo*inositol (II); bornesitol is therefore one of the enantiomers of 1-*O*-methyl*myo*inositol.

Subsequently, dambonitol has been synthesised by the methylation of 1 : 4 : 5 : 6-tetra-*O*-acetyl*myo*inositol [20] (III). It was assumed that, as in the methylation of the penta-acetyl compound, acetyl migration will occur and the equatorial hydroxyl groups will be methylated in preference to the axial one. Dambonitol acetate was indeed found to be the main product of the reaction; besides this, after deacetylation, 1- and 2-*O*-methyl*myo*inositol were isolated and a small amount of another dimethyl ether, presumably the 1 : 2-isomer. Absence of the other monomethyl ethers in the mixture indicates that acetyl migration occurred only between *cis*-related hydroxyl groups.

1 : 4 : 5 : 6-Tetra-*O*-acetyl*myo*inositol (III) was prepared by mild hydrolysis of 1 : 4 : 5 : 6-tetra-*O*-acetyl-2 : 3-*O*-*iso*propylidene*myo*inositol. The preparation of the latter described previously [20,21] was improved by the use of very large amounts (up to 40%) of zinc chloride dissolved in acetone; but even under these vigorous conditions the reaction of *myo*inositol with acetone is much slower than that of other cyclitols. This effect is undoubtedly due to the unfavourable conformation of three contiguous *cis*-hydroxyl groups.[13]

In the paper-chromatographic characterisation of *O*-methylinositols it was noticed that all the naturally occurring ethers were contaminated by the parent inositol, which was not usually removed by recrystallisation but very effectively by chromatography on cellulose powder.[4] Thus King's bornesitol, Riggs's sequoyitol, and Plouvier's ononitol contained some *myo*inositol. Quebrachitol contained both *myo*- and (−)-inositol.[22] In the sugar pine, pinitol and sequoyitol are accompanied by (+)- and *myo*inositol.[4] It appears that the inositols are intermediates in the biosynthesis of their methyl ethers. Significantly, in both *Dyera* species dambonitol is accompanied by a monomethyl ether and by traces of *myo*inositol. The former was isolated by chromatography on cellulose powder and was identified as (+)-bornesitol, the intermediate in a step-wise methylation.

[18] Kiang and Loke, *J.*, 1956, 480.
[19] Dr. L. Anderson, personal communication, Aug. 1954.
[20] Dangschat, *Naturwiss.*, 1942, **30**, 146.
[21] Angyal and Macdonald, *J.*, 1952, 686.
[22] Smith, *Biochem. J.*, 1954, **57**, 140.

It is of interest that all the monomethyl ethers of *myo*- and the optically active inositols in which the methoxyl group is equatorial have been encountered in Nature, but none of those with an axial methoxyl group; the enzymes responsible for methylation apparently avoid the steric strain caused by bulky axial groups. The two methoxyl groups in dambonitol are also equatorial.

Experimental

M. p.s are corrected.

Cellulose-powder Chromatography.—Whatman cellulose powder (standard grade) was mixed to a thin paste with acetone–water (4 : 1 v/v) and stirred for 5 min. in a Waring blendor. The mixture was then poured, in small portions, into a chromatographic column already containing some 1 : 4-aqueous acetone, and allowed to settle without stirring, tapping, or compression. Each addition was made before the cellulose previously added had fully settled; the tap at the bottom of the column was fully opened but the column never allowed to run dry. Columns thus prepared could be used many times as long as they were kept covered with solvent. The surface of the packing was protected by a filter-paper disc. The mixture to be chromatographed was introduced in the minimum amount of water, diluted with 4 vols. of acetone (or with as much as could be added without causing precipitation). A drop of methyl-orange (R_F 1·0) was added to mark the solvent front: no fractions were collected until the dye appeared in the effluent. Lissamine-red 6BS (R_F 0·35) is a convenient aid in showing the progress of the elution; the dyes also show at a glance whether the packing of the column is faulty. Acetone–water (4 : 1 v/v) was used as the moving phase in all experiments.

Methylation of myo*Inositol.*—A solution of *myo*inositol (50 g.) and barium hydroxide octahydrate (320 g.) in water (750 ml.) was stirred under reflux while dimethyl sulphate (125 g.) was added during 1½ hr. After a further 6 hours' boiling, the mixture was acidified with 10N-hydrochloric acid and filtered with the aid of kieselguhr. The solid was extracted with boiling water (600 ml.), and the extract combined with the filtrate and evaporated to dryness. The residue was extracted with boiling ethanol (2 × 250 ml.).

The undissolved material (115 g.) was remethylated and the process repeated twice more, with decreasing amounts of barium hydroxide and dimethyl sulphate. The undissolved material (22 g.) then contained less than 2 g. of acetylatable substance.

The combined ethanolic extracts deposited a solid (7·5 g.) containing considerable amounts of *myo*inositol. The mother-liquors were evaporated and acetylated with acetic anhydride and sulphuric acid; the acetyl derivatives were stirred to a paste with ethanol and filtered, giving a solid (30 g.) and, on evaporation of the ethanol, a syrup (13 g.).

The solid was fractionally crystallised from water. The least soluble material (1·5 g.; m. p. 220—225°) was *penta*-O-*acetyl*-2-O-*methyl*myo*inositol.* Several recrystallisations from ethanol, and then from ethyl acetate, raised the m. p. to 235—236° (Found: C, 50·5; H, 5·95. $C_{17}H_{24}O_{11}$ requires C, 50·5; H, 6·0%).

The next fraction (7 g.; m. p. 140—145°) gave (±)-penta-*O*-acetyl-1-*O*-methyl*myo*inositol (5·8 g.; m. p. 147—148°) on crystallisation from water (500 ml.). Repeated crystallisation from ethanol raised the m. p. to 152—153° (Griffin and Nelson [16] give m. p. 141°; Anderson and Landel,[15] m. p. 154—154·5°), mixed m. p. with Anderson's sample, 152—153°.

A later fraction, m. p. 125—140°, was deacetylated and chromatographed on cellulose powder, giving sequoyitol (m. p. 239°), 1-*O*-methyl*myo*inositol (m. p. 200°), and *myo*inositol. The dimethyl ether described by Griffin and Nelson [16] was not encountered in our work.

2-O-*Methyl*myo*inositol.*—Deacetylation of the acetate with dry methanol containing a trace of sodium methoxide and crystallisation from ethanol gave the *methyl ether*, m. p. 212° (Found: C, 43·4; H, 7·1. $C_7H_{14}O_6$ requires C, 43·3; H, 7·25%).

(±)-1-O-*Methyl*myo*inositol.*—Deacetylation of the acetate and crystallisation from aqueous ethanol gave the methyl ether, m. p. 199—200° (lit.,[15,16] m. p. 200—201°, 204°), mixed m. p. with Anderson's sample, 200—201°; with bornesitol, 191—194° (Found: C, 43·1; H, 7·2%).

The ionophoretic behaviour of these compounds is reported elsewhere.[13]

Purification of Sequoyitol.—Riggs's crude sequoyitol was crystallised several times from aqueous ethanol until the m. p. rose to 236—237°. Admixture of Sherrard and Kurth's "sequoyite" [3] caused no lowering of the m. p. (This material still contains some inositol: chromatographically pure sequoyitol melts at 239°.) The combined mother-liquors were evaporated to dryness and extracted three times with ethanol; from water–ethanol (2 : 5) the

insoluble residue gave large crystals which melted, alone and on admixture with macrozamin, at 202—203°. The R_F value (0·44 in 80% acetone) was the same as that of macrozamin.

Infrared Spectra of the Methylinositol Acetates.—To avoid any differences caused by crystal structure, the spectra were taken on 6% solutions in $CHCl_3$ with a Perkin-Elmer Model 12C instrument and a rock-salt prism.

Absorption bands were found at the following frequencies:

Penta-*O*-acetylbornesitol and -(±)-1-*O*-methyl*myo*inositol: 904(m), 921(w), 950(s), 968(w), 983(w), 1037(s), 1068(s), 1087(w), 1120(w), 1128(m), and 1163(w).

Penta-*O*-acetyl-2-*O*-methyl*myo*inositol: 911(w), 930(m), 946(w), 979(m), 1040(s), 1122(m), and 1172(w).

Penta-*O*-acetylononitol: 902(w), 921(w), 947(m), 978(m), 1035(s), 1055(s), 1084(w), 1100(w), 1113(w), and 1148(w).

Penta-*O*-acetylsequoyitol: 915(w), 951(s), 1066(m), 1093(w), 1139(w), and 1167(m).

Dambonitol from Dyera costulata.—Jelutong (*D. costulata*) latex (1·5 gallons) was coagulated by addition of phosphoric acid in the Timber Research Laboratory, Kuala Lumpur; the serum (1 l.) was shipped to Sydney. On arrival, it was neutralised with concentrated ammonia solution (25 ml.), filtered from some gelatinous precipitate, refiltered with charcoal, and evaporated to dryness *in vacuo*. The residue, crystallised from ethanol, gave two crops (45 and 12 g.) of dambonitol, m. p. 204—206°. Recrystallisation from ethanol (650 ml.) gave large crystals (50 g.), m. p. 206—207°, of dambonitol (lit.,[17] m. p. 210°); paper chromatography in 80% acetone showed the presence of bornesitol and *myo*inositol. Contrary to the statement of Comollo and Kiang [17] dambonitol is not hygroscopic. It was recovered unchanged after attempts to condense it with acetone [21] or to oxidise it by *Acetobacter suboxydans*.

To isolate the bornesitol, crude dambonitol (3 g.) was recrystallised from ethanol (30 ml.), and the solid residue (0·6 g.) left by evaporating the mother-liquor was chromatographed on a 1½ in. × 9 in. cellulose-powder column; 10 ml. fractions were collected. Fractions 23—34, which paper chromatography showed to contain predominantly methylinositol, gave 40 mg. of solid which, on slow crystallisation from aqueous ethanol, yielded dextrorotatory crystals (20 mg.), m. p. 202—203°; mixed m. p. with bornesitol, 203°; with (±)-1-*O*-methyl*myo*inositol, 191—193°.

Dambonitol from D. lowii.—The latex was treated with phosphoric acid by natives in the Borneo jungle and the resulting coagulum pressed as dry as possible at the Chicle Development Co., Bintulu. The serum (250 ml.), which smelt strongly of butyric acid when received in Sydney, was concentrated to 25 ml., an equal volume of ethanol added and the mixture filtered with charcoal. Evaporation of the filtrate left a gum (2·3 g.) which was dissolved in water (8 ml.). Addition of ethanol (24 ml.) gave a gummy precipitate which was separated by filtration. The filtrate was evaporated and the residue chromatographed through cellulose powder: 10 ml. fraction were collected. Fractions 7—15 (1·3 g. of solid) gave dambonitol (0·8 g.), m. p. 203—204°, on crystallisation from ethanol. Repeated crystallisation raised the m. p. to 208°; paper chromatography showed no bornesitol or inositol. The tetra-acetate, prepared by treatment with acetic anhydride and pyridine for 1 hr. at 100° and crystallisation from aqueous ethanol, melted at 202° (lit.,[17] 195°). From fractions 16—23 (66 mg. of solid) a few crystals of bornesitol were isolated; in later fractions the presence of *myo*inositol was shown by paper chromatography.

Partial Demethylation of Dambonitol.—Dambonitol (500 mg.) and concentrated hydriodic acid (1 ml.) were heated on a steam-bath for 15 min. The mixture was evaporated to dryness in a desiccator over sodium hydroxide, and the residue dissolved in water and extracted with chloroform to remove iodine. The concentrated aqueous layer was then placed on a cellulose-powder column. Fractions 5—9 (120 mg.) contained mainly dambonitol, and fractions 23—30 (70 mg.) mainly *myo*inositol. Fractions 10—18 all showed a spot at R_F 0·32 and were evaporated together (220 mg.): addition of ethanol and filtration gave crystals, m. p. 196—200°, mixed m. p. with (±)-1-*O*-methyl*myo*inositol, 198—200°. Acetylation with acetic anhydride and sulphuric acid, followed by crystallisation from ethanol, gave (±)-penta-*O*-acetyl-1-*O*-methyl-*myo*inositol, m. p. 150—152°, mixed m. p. 151—152°.

(±)-3 : 4 : 5 : 6-*Tetra*-O-*acetyl*-1 : 2-O-iso*propylidene*myo*inositol*.—The previously described preparation [20, 21]—reaction of *myo*inositol with acetone containing zinc chloride and acetic acid—proved unreliable and a better method has been developed. The main difficulty was thought to be the low solubility of inositol in the reaction mixture; it was subsequently found

that inositol could be dissolved in acetone containing very large amounts of zinc chloride, presumably by complex formation. Acetic acid had to be omitted, however, otherwise extensive acetylation occurred. Under these conditions, the reaction of *myo*inositol with acetone was reproducible but still very slow : half of the inositol was unchanged after 50 hours' refluxing.

Finely powdered anhydrous *myo*inositol (10 g.), anhydrous zinc chloride (80 g.), and dry acetone (200 ml.) were heated under reflux for 40 hr. Dry acetone (200 ml.) was added and the heating continued for another 10 hr. Dry pyridine (200 ml.) was added to the cooled mixture and, after a few hours at 0°, the precipitated pyridine–zinc chloride complex was filtered off and washed with a little acetone. The combined filtrates were mixed with acetic anhydride (100 ml.) and heated on a steam-bath until most of the acetone had evaporated. After a further hour's heating, the mixture was cooled, mixed with chloroform (100 ml.), and diluted with water. The chloroform layer was washed several times with sodium carbonate solution, dilute hydrochloric acid, and water. After drying (Na_2CO_3), the chloroform was evaporated and the residual oil was triturated with light petroleum (100 ml.). The crystals (7·0 g.) which formed were filtered off and recrystallised from ethanol, to give colourless prisms (6·4 g.), m. p. 122—123° (lit.,[20] 123—124°).

Unchanged inositol was recovered as its hexa-acetate (11 g.) by acetylating the pyridine–zinc chloride complex with acetic anhydride and pyridine. Thus the yield of the *iso*propylidene derivative was 60% based on unrecovered inositol.

(±)-1 : 4 : 5 : 6-*Tetra*-O-*acetyl*myo*inositol* (III).—The above tetra-acetate (4·0 g.) was heated with 80% acetic acid (20 ml.) on the steam-bath for 1 hr. The solution was evaporated *in vacuo*, redissolved in water, and again evaporated. The residual resin crystallised from water, yielding colourless prisms of the hydrated tetra-acetate which, after drying for 3 hr. at 80° and then at 100° *in vacuo*, weighed 2·77 g. (77%) and melted at 139°. Further crystallisation raised the m. p. to 142—143° (Found : C, 48·4; H, 5·9. Calc. for $C_{14}H_{20}O_{10}$: C, 48·3; H, 5·8%). Dangschat [20] gives the m. p. as 132—133° and states that 3 : 4 : 5 : 6-tetra-*O*-acetyl-1 : 2-*O*-*iso*propylidene*myo*inositol is *not* hydrolysed by acetic acid.

Methylation of 1 : 4 : 5 : 6-*Tetra*-O-*acetyl*myo*inositol* (III).—The above acetate (0·5 g.), methyl iodide (20 ml.), dioxan (30 ml.), and freshly prepared silver oxide [23] (5 g.) were refluxed for 60 hr. Paper chromatography of a hydrolysed sample showed much dimethyl ether besides small amounts of monomethyl ether and inositol. The mixture was filtered and evaporated; the residue was dissolved in hot aqueous ethanol and gave crystals (0·29 g.) on cooling. Several crystallisations from water yielded tetra-*O*-acetyldambonitol (70 mg.), m. p. and mixed m. p. 202° (Found : C, 51·25; H, 6·3. Calc. for $C_{16}H_{24}O_{10}$: C, 51·05; H, 6·45%).

The combined mother-liquors were evaporated, hydrolysed with hydrochloric acid, and evaporated, and the residue (0·17 g.) was chromatographed over cellulose powder; 10 ml. fractions were collected. Fractions 20—30 gave an oil (35 mg.) from which, by three crystallisations from ethyl acetate, plates of (presumably) 1 : 2-*di*-O-*methyl*myo*inositol*, m. p. 162—163°, were obtained (Found : C, 46·5; H, 7·7. $C_8H_{16}O_6$ requires C, 46·15; H, 7·75%).

Fractions 31—40 contained 18 mg. which, after two crystallisations from ethanol, gave 2-*O*-methyl*myo*inositol, m. p. and mixed m. p. 210—211°. Fractions 41—60 contained 25 mg. which gave, by crystallisation from ethanol, 1-*O*-methyl*myo*inositol, m. p. and mixed m. p. 198—200°. Fractions 80—100 contained *myo*inositol.

Methylation with silver carbonate instead of silver oxide gave similar results.

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[23] Bates *et al.*, " Polarimetry, Saccharimetry and the Sugars," Nat. Bur. Stand., Circular C440, Washington, 1942, p. 507.