zothiophene-5-dioxide sample was prepared by H. A. Pacevitz.[8]

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(8) H. Gilman, A. L. Jacoby and H. A. Pacevitz, *J. Org. Chem.*, **3**, 120 (1938).

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Pentamethyl and Triacetyl Derivatives of *myo*-Inositol[1]

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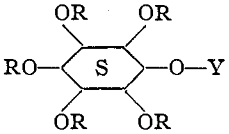
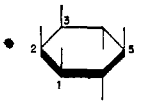
To facilitate another investigation which is still in progress we have prepared the partially alkylated or acylated inositols described below.

In 1947 McGowan[2] reported the preparation of the first pentamethyl ether of *myo*-inositol (I, R = CH_3, Y = H). His product was a sirup but did



I



II

yield a crystalline monoacetate of m.p. 101°. By saponification of this monoacetate we have now obtained the pentamethyl ether itself in crystalline form, m.p. 51°. We have further characterized the ether by converting it to its monobenzoate, m.p. 133°.

Such a pentaalkyl ether (or its monoester) can have only a single structure I, but twenty diastereomers are possible. However, the configuration II of the starting material here used limits the possible diastereomers to four, since no inversions of configuration would be expected.

In 1915 Griffin and Nelson[3] reported a procedure for conversion of *myo*-inositol to acetylated mono- and dimethyl ethers. While repeating this procedure we isolated a previously unreported by-product of m.p. 71°. This compound is apparently the first crystalline triacetate[4] of *myo*-inositol, as indicated by its analysis, and its transformation into the known hexaacetate on further acetylation.

Three structures are possible for an inositol triacetate, and ten diastereometric configurations are probable when the starting material II is used.

Each product here reported appears to consist of a single pure isomer, but the configurations (and structure for the triacetate) remain undetermined.

Experimental

M.p.'s (corrected) were taken on Köfler micro-block; microanalyses by Mr. R. S. Pyke.

***myo*-Inositol Pentamethyl Ether Monoacetate of M.P. 101°.**—Fifty grams of anhydrous *myo*-inositol when methylated by the procedure of West and Holden,[5] as modified by McGowan,[2] gave 10 g. of crude pentamethyl ether (colorless sirup, b.p. 123–145° (150 μ)), which on acetylation as described[2] gave 7.0 g. (22%) of the pentamethyl monoacetate, m.p. 100–101° (reported[2] 101°).

***myo*-Inositol Pentamethyl Ether of M.P. 51°.**—A solution of the pentamethyl ether monoacetate (7.0 g.) in 140 ml. of 1 *N* sodium hydroxide was kept at 25° for one hour, then neutralized with 12 *M* hydrochloric acid. The solution was saturated with potassium carbonate and extracted repeatedly with chloroform. The dried extract on evaporation left 6.0 g. of brown sirupy residue, which on vacuum-distillation gave 3.0 g. of a colorless viscous sirup, b.p. 127° (150 μ). The sirup solidified on standing overnight at 5°. The solid product was recrystallized twice from ligroin (b.p. 80–100°) giving 2.5 g. (42%) of ***myo*-inositol pentamethyl ether**, colorless crystals, m.p. 50–51°. The crystals are soluble in water, alcohol, or benzene.

Anal. Calcd. for $C_{11}H_{22}O_6$: C, 52.78; H, 8.86. Found: C, 52.38; H, 8.49.

On reacetylation of the crystalline pentamethyl ether, the monoacetate of m.p. 101° was again obtained.

***myo*-Inositol Pentamethyl Ether Monobenzoate of M.P. 133°.**—To the pentamethyl ether (0.60 g.) in 3.0 ml. of dry pyridine was added with stirring 0.50 ml. of benzoyl chloride. After five minutes the crystals which had separated were collected, washed with water, with 0.5 *M* sodium carbonate, and again with water, and dried. By recrystallization from ligroin, 0.65 g. (76%) of ***myo*-inositol pentamethyl ether monobenzoate**, colorless crystals, m.p. 132–133°, were obtained. The crystals are soluble in alcohol or benzene.

Anal. Calcd. for $C_{18}H_{26}O_7$: C, 61.00; H, 7.40. Found: C, 60.99; H, 7.31.

***myo*-Inositol Triacetate of M.P. 71°.**—*myo*-Inositol (20 g.) was treated by the methylation and acetylation procedure[3] of Griffin and Nelson. The filtered ethereal mother liquors, from which the acetylated mono- and dimethyl ethers had already crystallized, on long standing formed a third crop of crystals. This third crop was recrystallized from ethanol, giving 0.30 g. (0.8%) of colorless crystals, m.p. 70–71°. The crystals are soluble in chloroform but not in water. Analysis indicates that the new compound is a triacetate of (unmethylated) *myo*-inositol.

Anal. Calcd. for $C_{12}H_{18}O_9$: C, 47.06; H, 5.92. Found: C, 47.49; H, 5.89.

The triacetate (0.27 g.) on further acetylation with 5 ml. of hot acetic anhydride containing 0.1 g. of zinc chloride gave the expected *myo*-inositol hexaacetate (0.30 g., 79%) of m.p. 212–213° (reported[6] 211–212°).

Although the use of methylating agents in the procedure may seem superfluous, no more direct method for preparing this triacetate has yet been found.

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(1) Aided by a grant from the Research Council of Ontario.

(2) J. C. McGowan, *J. Soc. Chem. Ind.*, **66**, 446 (1947).

(3) E. Griffin and G. Nelson, THIS JOURNAL, **37**, 1566 (1915).

(4) The only previously reported triacetate of *myo*-inositol was an amorphous, gummy, hygroscopic substance of unstated m.p. and uncertain purity. See H. Müller, *J. Chem. Soc.*, **101**, 1781 (1912).

(5) E. S. West and R. F. Holden, *Org. Syntheses*, **20**, 97 (1940).

(6) L. Maquenne, *Ann. Chim.*, [6] **12**, 100 (1887).

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Antimony(III) Fluoride–Dioxane Addition Compound[1]

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Antimony(III) fluoride, which is reported to occur as a molecular crystal,[2] does not show the tendency to form addition compounds with many organic compounds that is so characteristic of the other halides of antimony(III). Solubility measurements in various organic solvents,[3] such as benzene or chlorobenzene, gave no indication of reaction. There was, however, some evidence of reac-

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(1) This research was supported by the Research Corporation and is taken in part from the M.S. thesis of R. H. Glazier.

(2) A. Byström and A. Westgren, *Arkiv. Kemi, Mineral Geol.*, **17B**, No. 2, 1 (1943); R. W. G. Wyckoff, "Crystal Structures," Interscience Publishers, Inc., New York, N. Y., 1951, Chap. V, table p. 17b.

(3) D. W. Breck, J. L. Harvey and H. M. Haendler, *J. Phys. Colloid Chem.*, **53**, 906 (1949).