# 448. *Anhydrides of Polyhydric Alcohols. Part XII. The Reaction between Mannitol and Hydrochloric Acid.*

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The reaction between mannitol and hydrochloric acid has been studied in detail. Either 1 : 6-dichloro mannitol or 1 : 4-3 : 6-dianhydromannitol can be obtained as the main product according to the experimental conditions. In addition, numerous other anhydrides of mannitol or their derivatives have been isolated. The presence of 1 : 5-anhydromannitol (styracitol), 1 : 4-anhydromannitol (mannitan), and 1 : 5-3 : 6-dianhydromannitol (neomannide) has been detected. A new hexitol dianhydride of unknown constitution has been isolated as such and as its bismethanesulphonate, and yet another dianhydride as its bismethanesulphonate only. A derivative of monochloro monoanhydromannitol, probably 6-chloro 1 : 4-anhydromannitol, and a dichloro-derivative of monoanhydromannitol, probably either 2 : 6- or 1 : 2-dichloro 1 : 4-anhydromannitol, were encountered.

When mannitol is treated with boiling concentrated hydrochloric acid for a long period, 1 : 4-3 : 6-dianhydromannitol is formed in yields about 35% (Wiggins, *J.*, 1945, 4; Montgomery and Wiggins, *J.*, 1947, 433). On the other hand, mannitol heated with fuming hydrochloric acid under pressure affords 1 : 6-dichloro mannitol in yields of about 40% (Haworth, Heath, and Wiggins, *J.*, 1944, 155). It is of interest to discover what other products are formed in each of these reactions and to discover the relationship between them. Some effort toward this end has now been made, and although the position has been found to be very complicated, and a complete picture of the reactions going on has not yet been acquired, the results so far obtained are of sufficient interest to warrant publication.

A solution of mannitol in concentrated hydrochloric acid was boiled for 72 hours, and the syrup so obtained separated by distillation into four fractions, the first two of which crystallised and after recrystallisation yielded 1 : 4-3 : 6-dianhydromannitol in 27% yield. The last two fractions were combined with the residues from the crystallisation and fractionally distilled into seven parts. The methanesulphonyl derivatives of each of these fractions were prepared. The first three fractions gave 2 : 5-*bismethanesulphonyl* 1 : 4-3 : 6-*dianhydromannitol*, m. p. 104°, identical with authentic material. These fractions also contained 2-*chloro* 5-*methanesulphonyl* 1 : 4-3 : 6-*dianhydromannitol* (5-chloro 2-methanesulphonyl 1 : 4-3 : 6-dianhydromannitol) (see below), thus showing the presence of 2(5)-chloro 1 : 4-3 : 6-dianhydromannitol (I). In the higher-boiling fractions, three new *dianhydrohexitols* were isolated. One (A) was isolated as such (m. p. 118—119°, $[\alpha]_D$ — 33·6°) and as its *bismethanesulphonate*, m. p. 139—140°. The other two, (B) and C), were isolated as their bismethanesulphonates only (m. p. 190—191° and 113—114° respectively). A convenient way of isolating dianhydrohexitol (A) was by benzylidenation of the accompanying materials in the fraction in which it occurred, and it was then possible to isolate it by distillation since it formed no benzylidene derivative. Treatment of the highest-boiling fraction with benzaldehyde gave a crystalline *chloro benzylidene anhydrohexitol*, m. p. 172°. Mannitan (1 : 4-anhydromannitol), although not isolated here, was produced by brief treatment of mannitol with hydrochloric acid (Wiggins, *loc. cit.*).

Thus it is seen that this reaction produces a monoanhydromannitol, a derivative of a monoanhydrohexitol which may also be a mannitol derivative, and no fewer than four different dianhydrides. Since the conditions under which these substances have been formed preclude the existence of the less stable three- or four-membered rings (no examples of the latter have ever been described in the hexitol series), only hydrofuranol or hydropyranol ring systems need be considered. This being the case, there are only three such dianhydrides of mannitol which are sterically possible, namely 1 : 4-3 : 6-, 1 : 5-3 : 6-, and 1 : 5-2 : 6-. Since we have obtained four dianhydrides, Walden inversion must have occurred in the formation of one or more of them, so that they cannot all be of the mannitol configuration. Of the three possible dianhydrides of mannitol, two have been described previously, namely " *iso*mannide " (1 : 4-3 : 6-dianhydromannitol) and " neomannide " (1 : 5-3 : 6-dianhydromannitol) (II*a*, II*b*), the latter having been synthesized and its structure proved by Hockett and Sheffield (*J. Amer. Chem. Soc.*, 1946, **68**, 937). A third dianhydromannitol called " β-mannide " was obtained by Siwoloboff (*Annalen*, 1886, **233**, 372) and had m. p. 119°, $[\alpha]_D$ + 94°, but the constitution of this has not yet been determined; by a process of elimination, however, it should be 1 : 5-2 : 6-dianhydromannitol, though no other workers have yet obtained this substance. The properties of these anhydrides of mannitol are summarised in the Table.

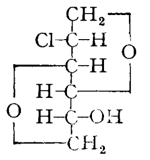
The dianhydrohexitol (A) showed m. p. 118°. This is very close to that described by Siwoloboff for β-mannide, though the specific rotation was $[\alpha]_D$ — 33·6°, a value quite different

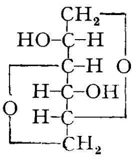
*Properties of mannitol anhydrides.*

| Anhydride. | M. p. | $[\alpha]_D$. | Reference. |
|---|---|---|---|
| " *iso*Mannide " (1 : 4-3 : 6-dianhydromannitol) | 86—87° | +91·0° | Wiggins (*J.*, 1945, 4). |
| " Neomannide " (1 : 5-3 : 6-dianhydromannitol) | 112·8—113·3 | + 6·4 | Hockett and Sheffield (*loc. cit.*). |
| " *β*-Mannide " | 119 | +94·05 | Siwoloboff (*loc. cit.*). |
| " Mannitan " (1 : 4-anhydromannitol) | 145—147 | −23·75 | Valentin (*Coll. Czech. Chem. Comm.*, 1936, **8**, 35). |
| " Styracitol " (1 : 5-anhydromannitol) | 145—155 | −50·9 | Hockett and Conley (*J. Amer. Chem. Soc.*, 1944, **66**, 464). |

from that of β-mannide, so that the two cannot be identical. Dianhydrohexitols (B) and (C) were isolated here only as the bismethanesulphonates. The bismethanesulphonate of 1 : 5-3 : 6-dianhydromannitol (neomannide) has now been prepared and found to be identical in respect to m. p., specific rotation, and mixed m. p. with the bismethansulphonate of dianhydride (B). No clue as to the identity of the dianhydride (C) has been obtained.

Of the monoanhydro-derivatives isolated from the reaction in question, mannitan is readily detected and recognised when the time of heating is approximately 12 hours. The other monoanhydride isolated was a monochloro monobenzylidene derivative. This compound is probably 6-chloro 3 : 5-benzylidene 1 : 4-anhydromannitol (III) which could very well be produced by the partial ring-opening of the main product of the reaction, namely 1 : 4-3 : 6-dianhydromannitol, followed by its benzylidenation. This, however, is a tentative suggestion.


(I)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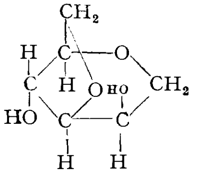
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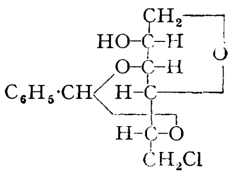
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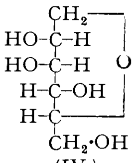
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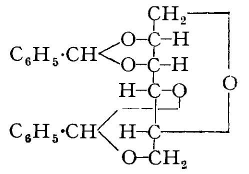
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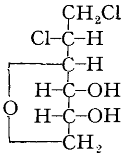
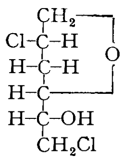

(VI.)


(VII.)

When mannitol is treated with fuming hydrochloric acid under pressure, the main product is 1 : 6-dichloro mannitol, isolated in about 40% yield (Haworth, Heath, and Wiggins, *J.*, 1944, 155). Now it has been found that 1 : 4-3 : 6-dianhydromannitol is formed as well in this reaction, because it has been isolated as such from the by-products as its bismethanesulphonate and as 2-chloro 1 : 4-3 : 6-dianhydromannitol. The latter was identified as its 5-methanesulphonate and by the fact that it was transformed by phosphorus pentachloride into 2 : 5-dichloro 1 : 4-3 : 6-dianhydromannitol identical with an authentic specimen. These products were isolated by fractional distillation of the residue from the crystallisation of the 1 : 6-dichloro mannitol, and were encountered in the lower-boiling fraction. From a higher-boiling fraction after benzylidenation, a chloro benzylidene anhydrohexitol, m. p. 172—173°, was isolated. This was identical with the compound encountered among the products of the action of boiling hydrochloric acid on mannitol. In addition, a new dibenzylidene hexitol, m. p. 192°, was obtained, identical with the dibenzylidene derivative of styracitol, here described for the first time. The styracitol (IV) was prepared from tetra-acetyl oxyglucal by the method of Hockett and Conley (*loc. cit.*). This on benzylidenation gave *dibenzylidene styracitol* identical with the dibenzylidene hexitol, m. p. 192°. Its structure will most probably be represented by (V) because dibenzylidene mannopyranose has been shown to have this orientation of its benzylidene residues. Another monoanhydrohexitol derivative was isolated from the high-boiling fraction, namely a *dichloro anhydrohexitol.* Since it has been definitely shown that 2-chloro 1 : 4-3 : 6-dianhydromannitol was present, the dichloro anhydrohexitol may well have arisen through the partial ring scission of this derivative of the dianhydride. If the

1 : 4-ring opened, then 1 : 2-dichloro 3 : 6-anhydromannitol (VI) would result, whereas if the 3 : 6-ring suffered scission, then 2 : 6-dichloro 1 : 4-anhydromannitol (VII) would be formed, and future work must decide this issue. Owing to the symmetry of the mannitol molecule, (VI) and (VII) are identical with 5 : 6-dichloro 1 : 4-anhydromannitol and 1 : 5-dichloro 3 : 6-anhydromannitol respectively.

From these results the way in which these products occur may be suggested. Boiling hydrochloric acid effects the dehydration of mannitol as do other acid catalysts. This may involve, however, the preliminary attachment of the anion, in this case a chloro-group, to the primary carbon atoms. The main dehydration product, nevertheless, is the dihydrofuranol ring system—1 : 4-3 : 6-dianhydromannitol—which is formed in stages : first the 1 : 4-ring, then the dianhydride. This is so because 1 : 4-anhydromannitol (mannitan) is isolated when the reaction is allowed to proceed for a short time only. This dehydration is accompanied by the formation of smaller amounts of other types of ring products, namely the 1 : 5-ring in styracitol and the 1 : 5-3 : 6-rings in neomannide. The treatment with hydrochloric acid also effects partial replacement of the secondary hydroxyl groups shown by the isolation of 2-chloro 1 : 4-3 : 6-dianhydromannitol under both sets of conditions, although much more is found amongst the products of the more drastic reaction. Since much more 1 : 6-dichloro mannitol than 1 : 4-3 : 6-dianhydromannitol is isolated under these more drastic conditions, we may envisage the main reaction as being an equilibrium between the following reactants :

$$\text{1 : 4-3 : 6-Dianhydromannitol} + \text{HCl} \rightleftharpoons \text{1 : 6-dichloro mannitol} + H_2O.$$

Thus the reaction of mannitol with excess of hydrochloric acid under pressure will favour the production of 1 : 6-dichloro mannitol, whilst the reaction under reflux, in which much less hydrochloric acid is present, favours the formation of the 1 : 4-3 : 6-dianhydride. The total yield of 1 : 6-dichloro mannitol obtained after numerous re-treatments of the residues from the reaction of mannitol with fuming hydrochloric acid under pressure is limited to about 40% (Haworth, Heath, and Wiggins, *loc. cit.*). This is doubtless due to substitution of the secondary hydroxyl groups in 1 : 4-3 : 6-dianhydromannitol or in 1 : 6-dichloro mannitol, and also to the formation of ring systems which are still more difficult to open than the hydrofuranol type.

## EXPERIMENTAL.

*Action of Concentrated Hydrochloric Acid on Mannitol.*—(*a*) Mannitol (900 g.) was boiled under reflux with concentrated hydrochloric acid (5 l.) for 3½ days, and the resulting dark liquid evaporated to a syrup. This was dissolved in water, and the solvent again evaporated to remove most of the hydrochloric acid. The residual liquid was distilled into the following fractions : I, 219·8 g., b. p. 135—145°/11 mm.; II, 27·0 g., b. p. 150—157°/11 mm.; III, 95·0 g., b. p. 160—190°/10 mm.; IV, 62·0 g., b. p. 160—190°/0·01 mm.

Fraction I crystallised, and on recrystallisation from ethyl acetate yielded 1 : 4-3 : 6-dianhydromannitol (155·8 g.), m. p. 85—87°. Fraction II similarly gave 8 g. of dianhydromannitol. The mother liquors were evaporated and the residue refractionated, and a further 28·3 g. of dianhydromannitol separated. The mother liquors were again evaporated and the residues combined with fractions III and IV, and the whole carefully refractionated together. The final fractions collected were : (1), 7·3 g., b. p. 135—145°/13 mm., $n_D^{18^\circ}$ 1·5011; (2), 21·9 g., b. p. 145—150°/12 mm., $n_D^{18^\circ}$ 1·5041; (3), 15·0 g., b. p. 150—168°/11 mm., $n_D^{18^\circ}$ 1·5042; (4), 20·3 g., b. p. 165—173°/12 mm., $n_D^{18^\circ}$ 1·5071; (5), 22·8 g., b. p. 177—185°/10 mm., $n_D^{18^\circ}$ 1·5092; (6), 43·7 g., b. p. 190—196/10 mm., $n_D^{18^\circ}$ 1·5130; (7), 13·3 g., b. p. 196—210°/10 mm., residue, 40·0 g.

Fractions (1—5) did not crystallise and were each dissolved (1 g.) in dry pyridine (15 c.c.) and treated with methanesulphonyl chloride (2 c.c.) at 0°. After being kept for 3 days at room temperature the mixture was poured into ice–water and the product extracted with chloroform. The combined extracts were washed successively with 5N-sulphuric acid, sodium hydrogen carbonate solution, and water, and dried ($MgSO_4$). The solvent was evaporated, and the residue fractionally recrystallised from alcohol–acetone. The following compounds were obtained from each fraction.

*Fraction* 1. (*a*) 2 : 5-Bismethanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 101—102°; (*b*) 2-chloro 5-methanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 114—115°, $[\alpha]_D^{23^\circ}$ + 64·4° in chloroform (*c*, 1·645).

*Fraction* 2. (*a*) 2 : 5-Bismethanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 101—102°; (*b*) 2-chloro 5-methanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 114—115°.

*Fraction* 3. 2 : 5-Bismethanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 101—102°.

*Fraction* 4. (*a*) 2 : 5-Bismethanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 101—102; (*b*) *bismethanesulphonyl dianhydrohexitol* (*A*), m. p. 140—141°, $[\alpha]_D^{21^\circ}$ −28·9° in chloroform (*c*, 1·38) (Found : C, 32·2; H, 4·3. $C_8H_{14}O_8S_2$ requires C, 31·8; H, 4·6%).

*Fraction* 5. (*a*) 2 : 5-Bismethanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 101°; (*b*) bismethanesulphonyl dianhydrohexitol (A), m. p. 139—140°; (*c*) *bismethanesulphonyl dianhydrohexitol* (B), m. p. 190—191°, $[\alpha]_D$ + 14° in acetone (*c*, 0·85) (Found : C, 32·0; H, 4·9; S, 21·9. $C_8H_{14}O_8S_2$ requires C, 31·8; H, 4·6; S, 21·2%). An attempt to prepare a benzylidene derivative from this fraction resulted in recovery of the starting material in good yield.

*Fraction* 6. This partly crystallised. The crystals were drained from syrup. The *dianhydro-*

*hexitol* (A), after recrystallisation from ethyl acetate, had m. p. 118—119°, $[\alpha]_D$ − 33·6° in water (*c*, 1·25) (Found : C, 49·7; H, 7·2. $C_6H_{14}O_4$ requires C, 49·5; H, 6·9%). Treatment of fraction 6 with methanesulphonyl chloride gave (*a*) bismethanesulphonyl dianhydrohexitol (A), m. p. 139—140°, (*b*) bismethanesulphonyl dianhydrohexitol (B), m. p. 190—191°, (*c*) a *bismethanesulphonate* of the dianhydrohexitol (C), m. p. 113—114°, $[\alpha]_D^{19°}$ − 22·6° in chloroform (*c*, 1·855) (Found : C, 31·9; H, 4·1; S, 20·8%).

*Benzylidenation of fraction* 6. The syrup (3 g.) was shaken with benzaldehyde (50 c.c.) and zinc chloride (4 g.) for 24 hours. The solution was then treated with excess of aqueous sodium carbonate and the excess of benzaldehyde removed by steam distillation. The residue, obtained by evaporation of the solution to dryness, was extracted 4 times with 100-c.c. portions of chloroform. The combined extracts were dried ($MgSO_4$) and evaporated to dryness. The residue (2·1 g.) distilled at 220—230° (bath temp.)/12 mm. as a colourless oil (0·5 g.) which crystallised completely, and on recrystallisation from ethyl acetate had m. p. 118—119° and was identical with the dianhydrohexitol (A). Benzylidenation has therefore purified this from other material which has undergone condensation with benzaldehyde.

*Fraction* 7. This was partly crystalline. The crystals were the dianhydrohexitol (A), m. p. 118—119°. Treatment of this fraction 7 with methanesulphonyl chloride gave (*a*) bismethanesulphonyl dianhydrohexitol (A), m. p. 139—140°, (*b*) bismethanesulphonyl dianhydrohexitol (B), m. p. 191—192°.

*Benzylidenation of fraction* 7. The syrup (3·35 g.) was shaken with benzaldehyde (50 c.c.) and zinc chloride (4 g.) and the product isolated as described previously. It partly crystallised, and after recrystallisation from alcohol a compound with the composition of a chloro benzylidene anhydrohexitol was isolated, m. p. 172—173°.

*Action of Fuming Hydrochloric Acid on Mannitol.*—Mannitol (75 g.) was heated in sealed tubes at 100° for 36 hours with fuming hydrochloric acid (600 c.c.) as described by Haworth, Heath, and Wiggins (*loc. cit.*). The resulting brown liquid was evaporated under reduced pressure to a semi-solid mass which was dissolved in water, filtered with charcoal, and again evaporated. The product was taken up in a little water and allowed to cool; crystals of 1 : 6-dichloro mannitol, m. p. 174°, then separated (18 g.). The mother liquors were evaporated and the residue distilled under reduced pressure. The following fractions were collected : A, 20 g., b. p. 150—180° (bath temp.)/25 mm.; B, 9·7 g., b. p. 200—220° (bath temp.)/0·1 mm. A small fraction between A and B was not examined further.

*Fraction A.* This partly crystallised, and the crystals were separated on a tile. They were 1 : 4-3 : 6-dianhydromannitol (1 g.), m. p. 86—87° alone or in admixture with an authentic specimen. The tile was extracted with boiling water and the extract evaporated to a syrup which was distilled and collected in two portions : (*a*) b. p. 115—120° (bath temp.)/0·04 mm., $n_D^{18°}$ 1·5030 (Found : C, 44·8; H, 5·8; Cl, 16·8%); (*b*) b. p. 120—125° (bath temp.)/0·02 mm., $n_D^{18°}$ 1·5035 (Found : C, 44·7; H, 5·8; Cl, 16·2. Calc. for $C_6H_9O_3Cl$: C, 43·8; H, 5·4; Cl, 21·5%).

Both (*a*) and (*b*) were essentially monochloro dianhydromannitol. Fraction (*a*) was heated with phosphorus pentachloride at 130° for 1 hour, and the resulting mixture diluted with chloroform and washed successively with dilute sodium hydrogen carbonate solution and water. The extract, after being dried ($MgSO_4$), was evaporated and the product distilled at 90° (bath temp.)/0·07 mm. The distillate crystallised and was recrystallised from ether–petrol; m. p. 62—64° alone or in admixture with 2 : 5-dichloro 1 : 4-3 : 6-dianhydromannitol.

Fraction (*a*) was also treated with methanesulphonyl chloride, and the product found to contain 2 : 5-bismethanesulphonyl 1 : 4-3 : 6-dianhydromannitol, m. p. 102—103°, and 2-*chloro* 5-*methanesulphonyl* 1 : 4-3 : 6-*dianhydromannitol*, m. p. 115—117°, $[\alpha]_D^{23°}$ + 66·4° in chloroform (*c*, 1·645) (Found : Cl, 15·2. $C_7H_{11}O_5ClS$ requires Cl, 14·8%).

*Fraction B.* This partly crystallised. The crystals of a *dichloro anhydrohexitol* were separated and recrystallised from ethyl acetate, and had m. p. 216—217°, $[\alpha]_D^{22°}$ + 27·8° in alcohol (*c*, 1·295) (Found : C, 35·8; H, 5·1; Cl, 35·4. $C_6H_{10}O_3Cl_2$ requires C, 35·8; H, 5·0; Cl, 35·5%). Benzylidenation of fraction B (9 g.) with benzaldehyde (50 c.c.) and anhydrous zinc chloride (10 g.), the mixture being poured, after 24 hours' shaking, into petrol–water, gave a syrup which partly crystallised and on fractional recrystallisation yielded (1) *dibenzylidene styracitol* (see below), m. p. 192° undepressed by admixture with an authentic specimen (Found : C, 70·8; H, 6·0. $C_{20}H_{20}O_5$ requires C, 70·6; H, 6·0%). (2) A *chloro benzylidene anhydrohexitol*, m. p. 171—172° (Found : C, 57·1; H, 5·3. $C_{13}H_{15}O_4Cl$ requires C, 57·7; H, 5·5%).

*Neomannide* (1 : 5-3 : 6-*Dianhydromannitol*).—This was synthesised according to the procedure outlined by Hockett and Sheffield (*loc. cit.*).

*Bismethanesulphonyl Neomannide.*—Neomannide (50 mg.) was dissolved in dry pyridine (3 c.c.), and methanesulphonyl chloride (0·2 c.c.) added at 0°. The mixture was kept overnight, then poured into ice-water. The crystalline precipitate was collected, washed with water, and recrystallised from alcohol–acetone, forming small needles, m. p. 188—189° alone or in admixture with the bismethanesulphonyl dianhydrohexitol (B) obtained above; $[\alpha]_D$ + 11·1 in acetone (*c*, 0·90). The *bismethanesulphonyl dianhydrohexitol*, m. p. 190°, had $[\alpha]_D$ + 11·5 in acetone (*c*, 0·965) (Found : C, 32·3; H, 4·9. $C_8H_{14}O_8S_2$ requires C, 31·8; H, 4·6%).

*Styracitol* (1 : 5-*anhydromannitol*).—This was obtained by the hydrogenation of tetra-acetyl oxyglucal according to the procedure of Hockett and Conley (*loc. cit.*).

*Dibenzylidene styracitol.* Styracitol (0·3 g.) and zinc chloride (0·2 g.) were shaken with benzaldehyde (5 c.c.) for 24 hours. Sodium carbonate (0·5 g.) in water was added and the excess of benzaldehyde removed by steam distillation under reduced pressure. The solution was evaporated to dryness and the residue extracted with chloroform. The chloroform extract was evaporated, and a crystalline residue obtained. This, recrystallised from alcohol, formed feathery needles, m. p. 192°.

2 : 3 : 4 : 6-*Tetrakismethanesulphonyl styracitol* Styracitol (0·1 g.) was dissolved in dry pyridine and methanesulphonyl chloride (0·5 g.) at 0°. The mixture was kept for 24 hours and then poured into ice-water. The precipitated *tetrakismethanesulphonate* was collected, washed with water, dried, and recrystallised from acetone containing a little ligroin. It formed small needles, m. p. 171—172° Found : C, 25·5; H, 4·3. $C_{10}H_{20}O_{13}S_4$ requires C, 26·0; H, 4·2%).

2 : 5-*Bismethanesulphonyl* 1 : 4-3 : 6-*dianhydromannitol.*—Dianhydromannitol (20 g.) was dissolved in dry pyridine (100 c.c.), and methanesulphonyl chloride (30 c.c., 2·1 mols.) carefully added at 0°. The mixture was kept for several days at room temperature and then poured into ice–water. The precipitated 2 : 5-*bismethanesulphonate* was collected and washed with water; recrystallised from alcohol–acetone, it formed colourless plates (28 g.), m. p. 104°, $[\alpha]_D^{18°}$ + 138·7° in chloroform (*c*, 1·775) (Found : C, 32·2; H, 4·6. $C_8H_{14}O_8S_2$ requires C, 31·8; H, 4·6%).

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