## 78. *The Anhydrides of Polyhydric Alcohols. Part V.* 2 : 5-*Diamino* 1 : 4-3 : 6-*Dianhydro Mannitol and Sorbitol and their Sulphanilamide Derivatives.*

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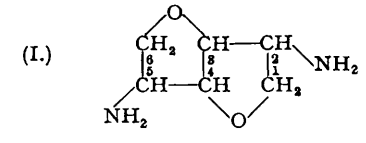
By treatment of the 2 : 5-ditosyl derivatives of 1 : 4-3 : 6-dianhydro mannitol and sorbitol with methyl alcoholic ammonia the corresponding diamines have been obtained. These have been characterised by the formation of a series of well-defined salts and other derivatives. The sulphanilamide derivatives were found to be less active than sulphathiazole in routine bacteriostatic tests.

SUCROSE can be hydrogenated with or without previous hydrolysis to mannitol and sorbitol and, on the other hand, invert sugar can be reduced electrochemically to give the same products. In Parts I and IV of this series the dianhydrides produced by the action of mineral acids on these two polyhydric alcohols are described and shown to possess the hydrofuranol type of ring. This paper describes the preparation of the diamines, and their corresponding sulphanilamides, derived from dianhydro mannitol and sorbitol according to the following scheme.

Dianhydro hexitol ⟶ 2 : 5-Ditosyl dianhydro hexitol ⟶ 2 : 5-Diamino dianhydro hexitol ⟶ Disulphanilamido dianhydro hexitol.

2 : 5-*Ditosyl dianhydro mannitol* and 2 : 5-*ditosyl dianhydro sorbitol* are well-defined crystalline substances which by treatment with methyl alcoholic ammonia under pressure are converted into the corresponding diamines. 2 : 5-*Diamino* 1 : 4-3 : 6-*dianhydro mannitol* (I) is crystalline but 2 : 5-*diamino* 1 : 4-3 : 6-*dianhydro sorbitol* (II) is a liquid. The former was obtained in 80% and the latter in 60% yield. Each diamine was characterised by well-defined salts. 2 : 5-Diamino 1 : 4-3 : 6-dianhydro mannitol gave a crystalline *oxalate, adipate, picrate, hydrochloride, sulphate,* and *dimethylene mucate* (*galactosaccharate*) (with dimethylene mucic acid, [(Mrs.) H. Gregory, Stacey, and Wiggins, unpublished work]). It also formed, with salicylaldehyde, crystalline 2 : 5-*disalicylideneamino* 1 : 4-3 : 6-*dianhydro mannitol.* 2 : 5-Diamino 1 : 4-3 : 6-dianhydro sorbitol gave a crystalline *oxalate, picrate, hydrochloride, sulphate, dimethylene saccharate* (*glucosaccharate*), and *dimethylene mucate.* It also formed, with salicylaldehyde, a beautifully crystalline azomethine, namely 2 : 5-*disalicylideneamino* 1 : 4-3 : 6-*dianhydro sorbitol.* The diamines themselves either as sulphates or as dimethylene mucates showed no bacteriostatic effect *in vitro* against *Staphylococcus aureus.*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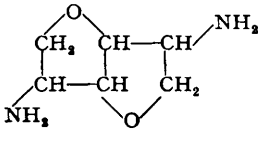
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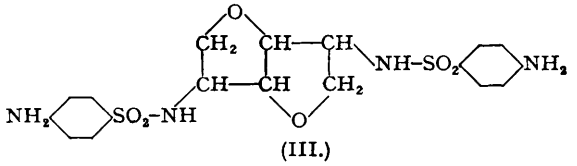
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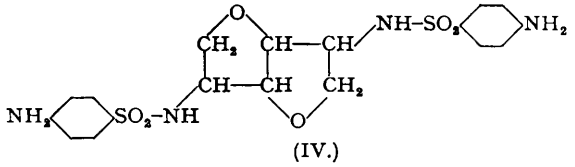
(IV.)

By the treatment of the diamines with *p*-acetamidobenzenesulphonyl chloride, 2 : 5-*di*-(p-*acetamidobenzenesulphonamido*) 1 : 4-3 : 6-*dianhydro mannitol* and 2 : 5-*di*-(p-*acetamidobenzenesulphonamido*) 1 : 4-3 : 6-*dianhydrosorbitol* were obtained. Hydrolysis by hydrochloric acid or by sodium hydroxide converted these into the corresponding *sulphanilamides* (III and IV). The sulphanilamides were also prepared by treatment of the diamines with *p*-nitrobenzenesulphonyl chloride and catalytic reduction of the resulting 2 : 5-*di*-(p-*nitrobenzenesulphonamido*) 1 : 4-3 : 6-*dianhydro mannitol* and 2 : 5-*di*-(p-*nitrobenzenesulphonamido*) 1 : 4-3 : 6-*dianhydrosorbitol.*

The sulphanilamide derivatives of dianhydro mannitol and dianhydro sorbitol were extremely weak bases. No salts could be prepared in hydroxylic solvents. The hydrochlorides, which could be precipitated from

acetone–benzene, showed no definite melting points and slowly decomposed on heating. They were hydrolysed by water, giving the free base and two equivalents of hydrochloric acid. The sulphanilamides were only slightly soluble in water (0·02 g./100 c.c.) but were fairly soluble in dilute hydrochloric acid. They proved to be inferior in bacteriostatic activity to sulphathiazole.

## Experimental.

2 : 5-*Ditosyl* 1 : 4-3 : 6-*Dianhydro Mannitol.*—Dianhydro mannitol (59 g.) was dissolved in dry pyridine (300 c.c.) and tosyl chloride (155 g.) added in portions at 0°. The solution was allowed to warm to room temperature and kept thereat for 24 hours. Thereafter it was poured into ice-water; the syrup so precipitated crystallised on keeping. The solid *product* was ground with water, filtered, washed several times with water, and recrystallised from ethyl alcohol–acetone (yield, 162 g.). It formed prisms, m. p. 93—94°, $[\alpha]_D^{20°}$ +92·2° in chloroform (*c*, 2·582) (Found: C, 53·0; H, 4·8. $C_{20}H_{22}O_8S_2$ requires C, 52·8; H, 4·9%).

2 : 5-*Diamino* 1 : 4-3 : 6-*Dianhydro Mannitol.*—2 : 5-Ditosyl 1 : 4-3 : 6-dianhydro mannitol (50 g.) was heated in an autoclave with methyl alcoholic ammonia (saturated at 0°) (1500 c.c.) at 170—180° for 30 hours. The brown solution was evaporated to dryness under reduced pressure so that all the ammonia was removed. Barium hydroxide (42 g.) dissolved in hot water (400 c.c.) was added and the mixture heated at 100° for 1 hour. The solution was then evaporated to dryness and the residue dried very carefully by distilling several lots of benzene over it so that every trace of moisture was removed. Thereafter the residue was extracted under reflux six times with chloroform (250 c.c. portions); the chloroform extracts were combined and evaporated to a syrup (12·8 g.) which distilled at 150° (bath temperature)/0·01 mm. as a yellow oil which rapidly crystallised. All operations subsequent to and including the treatment with barium hydroxide were carried out in a carbon dioxide free atmosphere. The distillate was recrystallised with some difficulty from dioxan–ether. The 2 : 5-*diamino* 1 : 4-3 : 6-*dianhydro mannitol* was very hygroscopic and could only be kept as a crystalline solid in sealed tubes under nitrogen. It had m. p. 59—62°, $[\alpha]_D^{20°}$ +33·6° in chloroform (*c*, 2·322) (Found: N, 18·8. $C_6H_{12}O_2N_2$ requires N, 19·4%).

*Salts of* 2 : 5-*Diamino* 1 : 4-3 : 6-*Dianhydro Mannitol.*—The diamine (0·1 g.) was dissolved in hot alcohol, and oxalic acid (0·09 g.), also dissolved in alcohol, added. A fine white precipitate separated. An equal volume of water was added and the mixture boiled until a clear solution was obtained. Crystals of the *oxalate* separated on cooling. Yield, 0·07 g., m. p. 246—247° (decomp.) (Found: N, 11·9. $C_8H_{14}O_6N_2$ requires N, 12·0%).

The diamine (0·11 g.) was mixed with adipic acid in alcoholic solution, water added, and the mixture boiled until clear. The *adipate* crystallised on cooling. Yield, 0·1 g., m. p. 189° (Found: N, 10·0. $C_{12}H_{22}O_6N_2$ requires N, 9·7%).

The diamine (0·06 g.) was dissolved in hot water and a hot aqueous solution containing picric acid (0·38 g.) added. A red syrup was precipitated which soon crystallised. The *picrate* was recrystallised from hot water forming prismatic crystals, m. p. 227—228° (decomp.) (Found: N, 19·3. $C_{18}H_{18}O_{16}N_8$ requires N, 18·6%).

To the diamine (0·1 g.) concentrated hydrochloric acid was added until the mixture was acid to Congo red. The solution was then mixed with alcohol, boiled, and allowed to cool when crystals of the *dihydrochloride* separated. This did not have a definite m. p. but decomposed between 280° and 300° (Found: C, 33·4; H, 6·1; N, 12·7; Cl, 32·7. $C_6H_{14}O_2N_2Cl_2$ requires C, 33·1; H, 6·5; N, 12·9; Cl, 32·7%).

The diamine (0·1 g.) was dissolved in the minimum quantity of water and concentrated sulphuric acid (0·07 g.) in a little water was added. The *sulphate* separated as a granular precipitate which was filtered off and washed with alcohol. It recrystallised from aqueous alcohol in prisms, which decomposed above 310° (Found: N, 11·9. $C_6H_{14}O_6N_2S$ requires N, 11·6%).

A solution of the diamine (0·15 g.) in alcohol was added to a hot aqueous solution of dimethylene mucic acid (0·22 g.) [(Mrs.) H. Gregory, Stacey, and Wiggins, unpublished work]. Crystals of the *dimethylene mucate* (0·25 g.) separated on cooling, m. p. 246—247° (with decomp.) (Found: C, 41·2; H, 6·2; N, 6·7. $C_{14}H_{22}O_{10}N_2,1\frac{1}{2}H_2O$ requires C, 41·6; H, 6·2; N, 6·9).

*Bacteriostatic test.* 2 : 5-Diamino dianhydro mannitol sulphate did not inhibit the growth of *Staphylococcus aureus in vitro.*

2 : 5-*Disalicylideneamino* 1 : 4-3 : 6-*Dianhydro Mannitol.*—2 : 5-Diamino 1 : 4-3 : 6-dianhydro mannitol (0·3 g.) was dissolved in a few c.c. of water containing three drops of 5 N-sodium hydroxide. Salicylaldehyde was added and the mixture vigorously shaken for 1 hour. The yellow solid which separated was collected, washed with water, and recrystallised from alcohol–acetone forming long feathery needles (0·35 g.), m. p. 188—189° (Found: C, 68·6; H, 5·6; N, 8·1. $C_{20}H_{20}O_4N_2$ requires C, 68·2; H, 5·7; N, 8·0%).

2 : 4-*Di*-(p-*acetamidobenzenesulphonamido*) 1 : 4-3 : 6-*Dianhydro Mannitol.*—(*a*) To a solution of diamino dianhydro mannitol (4 g.) in water (40 c.c.) was added *p*-acetamidobenzenesulphonyl chloride (14·72 g.). Enough acetone was added to effect complete solution and then sodium hydroxide (2·44 g.) was added. After being stirred for 45 minutes the mixture was kept overnight at room temperature and the crystals which had separated were filtered off and recrystallised from acetone–water, yielding feathery needles (14·7 g.), m. p. 278—279°.

(*b*) To a solution of the diamine (0·72 g.) in water (7 c.c.) were added acetone (13 c.c.) and *p*-acetamidobenzenesulphonyl chloride (2·56 g.). When complete solution was effected sodium bicarbonate (1·4 g.) was added and the mixture stirred for 45 minutes. Thereafter the product was worked up as in (*a*). Yield, 2 g., m. p. 278—279°.

(*c*) Diamino dianhydro mannitol (1·08 g.) was dissolved in dry pyridine (10 c.c.) and a solution of *p*-acetamidobenzenesulphonyl chloride (3·83 g.) in dry pyridine (10 c.c.) added at 0°. The solution was allowed to warm to room temperature and kept thereat for 2 days. It was then poured into ice-water and the solid obtained was collected, washed with water, and recrystallised several times from acetone–water. Yield, 2 g., m. p. 278—279°. The *products* from the three methods of preparation were identical. The material did not show any rotatory power with the sodium yellow or mercury green lines (Found: C, 49·0; H, 5·1; N, 10·0. $C_{22}H_{26}O_8N_4S_2$ requires C, 49·0; H, 4·8; N, 10·4%).

2 : 5-*Disulphanilamido* 1 : 4-3 : 6-*Dianhydro Mannitol.*—(*a*) To the di-*N*-acetyl derivative (10 g.) dissolved in acetone (100 c.c.) 2N-hydrochloric acid (200 c.c.) was added and the mixture refluxed for 6 hours. The solution was then filtered with charcoal and evaporated to dryness under reduced pressure. The residue was recrystallised from water (3·24 g.), m. p. 227—228°.

(*b*) A solution of the di-*N*-acetyl derivative (1 g.) in 10% sodium hydroxide (10 c.c.) was heated at 100° for 2 hours. Thereafter the solution was cooled and neutralised with dilute hydrochloric acid. The solid precipitated was collected and recrystallised from water (0·6 g.). The *compound* had m. p. 227—228°, and was identical with the material obtained by method (*a*) above (Found: C, 47·5; H, 4·8; $NH_2$ (by nitrite titration), 6·9. $C_{18}H_{18}O_6N_4S_2$ requires C, 47·6; H, 4·8; $NH_2$, 7·1%).

The substance showed no rotatory power with sodium light. It did not form salts in aqueous solution and was not alkaline to litmus.

2 : 5-*Disulphanilamido* 1 : 4-3 : 6-*Dianhydro Mannitol Dihydrochloride.*—Anhydrous hydrogen chloride was passed

into a solution of disulphanilamido dianhydro mannitol in anhydrous acetone–benzene (1 : 1). The salt, which was precipitated, was washed twice with acetone–benzene (1 : 1) and dried in a vacuum. It could not be recrystallised from anhydrous solvents. It effervesced between 180° and 215°, darkening at the latter temperature. On adding it to cold water, the free base (m. p. 227°) and 2 equivalents of hydrochloric acid (alkali titration) were obtained.

*Reacetylation of Disulphanilamido Dianhydro Mannitol.*—The material (0·1 g.) was shaken with a mixture (1 : 1) of acetic acid and acetic anhydride (10 c.c.) until completely dissolved; on pouring the solution into water, a solid separated which after being recrystallised from acetone–water had m. p. 278° alone or in admixture with a specimen of di(acetamidobenzenesulphonamido) dianhydro mannitol.

2 : 5-*Di*-(p-*nitrobenzenesulphonamido*) 1 : 4-3 : 6-*Dianhydro Mannitol.*—2 : 5-Diamino dianhydro mannitol (3 g.) was dissolved in dry pyridine and a solution of *p*-nitrobenzenesulphonyl chloride (9·31 g.) also in dry pyridine was carefully added at 0°. After keeping for 24 hours at room temperature the solution was poured into ice-water. A solid separated; this was filtered off, washed with water, and recrystallised from acetone–water. The *compound* formed small prisms (9·3 g.), m. p. 213—214°, $[\alpha]_D^{20°}$ +7·5° in acetone (*c*, 2·65) (Found: N, 11·0. $C_{18}H_{18}O_{10}N_4S_2$ requires N, 10·9%).

*Reduction of Di-*(p-*nitrobenzenesulphonamido*) *Dianhydro Mannitol.*—(*a*) *Tin and hydrochloric acid.* A suspension of di-(*p*-nitrobenzenesulphonamido) dianhydro mannitol (0·5 g.) in a mixture of alcohol (250 c.c.) and hydrochloric acid (*d*, 1·16; 24 c.c.) was heated under reflux with tin (0·96 g.) for 1 hour. After keeping overnight the solution was neutralised with 10% sodium carbonate solution and evaporated to dryness. The residue was extracted with absolute alcohol, the extracts were combined and evaporated to dryness, and this residue was again extracted with alcohol. This process was repeated until a residue reasonably free from inorganic salts was obtained. This, recrystallised from alcohol, gave a small yield of disulphanilamido dianhydro mannitol (0·1 g.), m. p. 227—228°, identical with the compound from the hydrolysis of the corresponding di-*N*-acetyl derivative.

(*b*) *Catalytic hydrogenation.* Di-(*p*-nitrobenzenesulphonamido) dianhydro mannitol (1 g.) in dry methyl alcohol (100 c.c.) was hydrogenated at room temperature under 1 atmosphere with Raney nickel as catalyst. The theoretical amount of hydrogen was absorbed, the reaction being complete in 30 minutes. The catalyst was filtered off and extracted 6 times with boiling methyl alcohol (100 c.c. portions). The combined methyl alcoholic solutions on evaporation to 20 c.c. gave a crystalline disulphanilamido 2 : 5-diamino dianhydro mannitol, m. p. 227—228°, alone or in admixture with the material obtained by the hydrolysis of the corresponding di-*N*-acetyl derivative.

2 : 5-*Ditosyl* 1 : 4-3 : 6-*Dianhydro Sorbitol.*—Dianhydro sorbitol (58 g.) dissolved in dry pyridine (300 c.c.) was treated with tosyl chloride (150 g.) at 0°. After keeping for 24 hours at room temperature and then pouring into ice-water the 2 : 5-ditosyl derivative was obtained; recrystallised from ethyl alcohol it formed prisms (190 g.), m. p. 101—102°, $[\alpha]_D^{23°}$ +57·8° in chloroform (*c*, 4·945) (Found: C, 53·3; H, 4·7; S, 13·8. $C_{20}H_{22}O_8S_2$ requires C, 52·8; H, 4·9; S, 14·1%).

2 : 5-*Diamino* 1 : 4-3 : 6-*Dianhydro Sorbitol.*—The ditosyl derivative (50 g.) was heated at 150—160° with methyl alcoholic ammonia (1500 c.c.) (saturated at 0°) in an autoclave for 30 hours. After being evaporated to dryness the product was heated with barium hydroxide (42 g.) dissolved in hot water (400 c.c.) for 1 hour in an atmosphere of nitrogen. The product was then isolated as in the case of the mannitol stereoisomer. 2 : 5-Diamino 1 : 4-3 : 6-dianhydro sorbitol distilled at 105—110° (bath temp.)/0·01 mm. as a yellow oil which would not crystallise, and had $n_D^{18°}$ 1·5165, $[\alpha]_D$ +43·6° in water (*c*, 1·538). Yield, 8·5 g. (54%). Some loss occurred on distillation (Found: C, 49·9; H, 7·9. $C_6H_{12}O_2N_2$ requires C, 50·0; H, 8·3%).

*Salts of* 2 : 5-*Diamino* 1 : 4-3 : 6-*Dianhydro Sorbitol.*—The diamine (0·1 g.) was treated in alcoholic solution with oxalic acid (0·09 g.) and the *oxalate* isolated in the usual way; it had m. p. 253—254° (decomp.) (Found: N, 11·6. $C_8H_{14}O_6N_2$ requires N, 12·0%).

The *picrate* was precipitated from aqueous solution and recrystallised from hot water. It formed small needles, m. p. 200° (decomp.) (Found: N, 19·0. $C_{18}H_{18}O_{16}N_8$ requires N, 18·6%).

The diamine was dissolved in anhydrous dioxan and hydrogen chloride bubbled through the solution. An equal volume of alcohol was added and the solution boiled until clear. The *hydrochloride* separated on cooling. It did not melt below 320° (Found: C, 31·1; H, 6·6; N, 12·7. $C_6H_{14}O_2N_2Cl_2$ requires C, 31·1; H, 6·5; N, 12·9%).

The diamine (0·1 g.) was dissolved in the minimum quantity of water, and concentrated sulphuric acid (0·07 g.) in a small amount of water was added. A granular precipitate of the *sulphate* separated which was washed with alcohol and recrystallised from aqueous alcohol. The crystals did not melt below 330° (Found: N, 11·9. $C_6H_{14}O_6N_2S$ requires N, 11·6%).

An alcoholic solution of the diamine (0·08 g.) was added to a hot aqueous solution of dimethylene saccharic acid (0·12 g.). An equal volume of alcohol was added and the precipitated *dimethylene saccharate* recrystallised from hot alcohol; it had m. p. 220—221° (decomp.) (Found: N, 7·8. $C_{14}H_{22}O_{19}N_2$ requires N, 7·4%).

Prepared as above, the *dimethylene mucate* had m. p. 235—236° (decomp.) (Found: C, 44·5; H, 5·6. $C_{14}H_{22}O_{10}N_2$ equires C, 44·4; H, 5·8%).

*Bacteriostatic test.* Neither the sulphate nor the dimethylene mucate of diamino dianhydro sorbitol inhibited the growth of *Staphylococcus aureus in vitro.*

2 : 5-*Disalicylideneamino* 1 : 4-3 : 6-*Dianhydro Sorbitol.*—The diamine (0·3 g.) was dissolved in a little water containing 3 drops of dilute sodium hydroxide solution. Salicylaldehyde (0·51 g.) was added and the mixture shaken for 1 hour. The base which had separated was collected and recrystallised from alcohol–acetone; it formed prisms (0·4 g.), m. p. 166—167° (Found: C, 68·3; H, 5·6. $C_{20}H_{20}O_4N_2$ requires C, 68·2; H, 5·6%).

2 : 5-*Di-*(p-*acetamidobenzenesulphonamido*) 1 : 4-3 : 6-*Dianhydro Sorbitol.*—(*a*) To a solution of the diamine (2·3 g.) in water (20 c.c.) *p*-acetamidobenzenesulphonyl chloride (8 g.) was added together with enough acetone to effect solution. Sodium hydroxide (1·34 g.) was added and dissolved by shaking. The solution was kept overnight, the acetone removed, and the syrupy precipitate triturated with alcohol until solid. It separated from acetone–water in a poorly crystalline condition, m. p. 256—258° (decomp.).

(*b*) The diamine (3·16 g.) was dissolved in dry pyridine and *p*-acetamidobenzenesulphonyl chloride (11·4 g.), also in pyridine, added carefully at 0°. After being allowed to stand for 2 days at room temperature the solution was poured into ice-water and the solid product washed with water and recrystallised from acetone–water. Yield, 6·1 g. Again the material was poorly crystalline; it had m. p. 263—264°, $[\alpha]_D$ +51·4° in acetone–water (1 : 1) (*c*, 0·6) (Found: C, 49·4; H, 4·7; N, 10·9. $C_{22}H_{26}O_8N_4S_2$ requires C, 49·0; H, 4·8; N, 10·4%).

2 : 5-*Disulphanilamido* 1 : 4-3 : 6-*Dianhydro Sorbitol.*—(*a*) The di-*N*-acetyl derivative (0·9 g.) was refluxed for 6 hours with 2N-hydrochloric acid (20 c.c.) and acetone (5 c.c.). The solution was filtered with charcoal and neutralised with dilute sodium hydroxide solution; 2 : 5-disulphanilamido 1 : 4-3 : 6 dianhydro sorbitol was then precipitated. After being recrystallised from alcohol–water the compound (0·3 g.) had m. p. 239—240°.

(*b*) The di-*N*-acetyl compound (1·1 g.) was added to 10 c.c. of sodium hydroxide solution (10%) and the mixture heated at 100° for 2 hours. The solution was then neutralised with dilute hydrochloric acid, and the precipitate so produced was filtered off and recrystallised from alcohol–water. Yield, 0·7 g. It had m. p. 239—240°, $[\alpha]_D$ + 49·2° in acetone (*c*, 0·406). The *compound* was identical with that obtained in (*a*) (Found: C, 47·4; H, 4·9; $NH_2$ (nitrite titration), 7·3. $C_{18}H_{22}O_6N_4S_2$ requires C, 47·6; H, 4·8; $NH_2$, 7·1%).

2 : 5-*Disulphanilamido* 1 : 4-3 : 6-*Dianhydro Sorbitol Dihydrochloride.*—Anhydrous hydrogen chloride was passed into a solution of disulphanilamido dianhydro sorbitol in anhydrous acetone–benzene (1 : 1). The salt, which was precipitated, was washed twice with acetone–benzene and dried in a vacuum. It could not be recrystallised from anhydrous solvents. It effervesced above 130°. On adding the salt to cold water it completely hydrolysed to give the free base (m. p. 239—240°) liberating 2 equivalents of hydrochloric acid (alkali titration).

2 : 5-*Di*-(p-*nitrobenzenesulphonamido*) 1 : 4-3 : 6-*Dianhydro Sorbitol.*—2 : 5-Diamino dianhydro sorbitol (4·05 g.) was dissolved in dry pyridine and a solution of *p*-nitrobenzenesulphonyl chloride (14·08 g.) also in pyridine was carefully added at 0°. After keeping at room temperature for 24 hours the mixture was poured into ice-water and the precipitated solid washed with water and dissolved in acetone–water, filtered with charcoal, and allowed to crystallise. The p-*nitrobenzenesulphonyl* derivative (10·6 g.) separated in small needles having m. p. 216—217°, $[\alpha]_D^{20°}$ + 56·0° in acetone (*c*, 2·138) (Found : C, 42·3; H, 3·4; N, 10·5. $C_{18}H_{18}O_{10}N_4S_2$ requires C, 42·0; H, 3·5; N, 10·9%).

*Catalytic Hydrogenation of Di*(p-*nitrobenzenesulphonamido*) *Dianhydro Sorbitol.*—The material (1 g.) in dry methyl alcohol (100 c.c.) was hydrogenated in the presence of Raney nickel at room temperature and 1 atmosphere. The absorption was complete in 15 minutes. Thereafter the catalyst was filtered off and extracted 6 times with boiling methyl alcohol, and the combined methyl alcoholic extracts were evaporated to dryness. The residue, recrystallised from alcohol–water, had m. p. 236—237°, not depressed in admixture with the product from the hydrolysis of di-(*p*-acetamidobenzenesulphonamido) dianhydro sorbitol. It was, therefore, disulphanilamido 1 : 4-3 : 6-dianhydro sorbitol.

*Reacetylation of Disulphanilamido Dianhydro Sorbitol.*—To a suspension of the material (0·1 g.) in dilute acetic acid acetic anhydride (5 c.c.) was added and the mixture shaken until complete solution was effected. The solution was then poured into ice-water (200 c.c.) and the crystalline precipitate collected, washed with water, and recrystallised from acetone–water; it formed small prisms (0·1 g.), m. p. 263—264°, $[\alpha]_D^{21°}$ +49·6° in acetone–water (1 : 1) (*c*, 0·383).

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