303. *Partial Periodate Oxidation of* D-*Glucitol and its Borate Complex.*

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The products of oxidation of D-glucitol with a limited quantity of periodate have been characterised and determined. The results show that the order of susceptibility of the C–C bonds in D-glucitol to cleavage by periodate is: 3,4 (αT-glycol) > 2,3 (αT-glycol) > 4,5 (αC-glycol) > 5,6 (α-glycol) > 1,2 (α-glycol). In borate buffer the favoured complex between D-glucitol and borate involves the participation of the 2- and the 4-hydroxyl group. Under suitable conditions the glucitol–borate complex produces *ca.* 30% of L-xylose.

It has been shown [1] that the oxidation of D-mannitol, galactitol, and D-glucitol with a limited quantity of sodium periodate involves preferential attack on αT-glycol groups (Barker and Bourne's nomenclature [2]). Similar oxidation of erythritol showed that a $CH(OH)\cdot CH_2\cdot OH$ group was more readily cleaved than a $CH(OH)\cdot CH(OH)$ group, but the reverse was found for hexitols.[3] Of acyclic compounds the *threo*- (αT) was oxidised more rapidly than the *erythro*-isomer (αC) and for *threo*-compounds the rate of oxidation decreases with increasing length of the substituents on the glycol group.[4] Thus it became interesting to study quantitatively the relative ease of cleavage of the various C–C bonds of D-glucitol with periodate, particularly as D-glucitol contains two αT- and α-glycol groups, and one αC-glycol group. It was further thought that limited oxidation of D-glucitol in water and in phosphate (pH 10) and borate buffer (pH 10) would clarify the structure of the D-glucitol–borate complex. Buffer of pH 10 was chosen as the concentration of the complexes between polyhydroxy-compounds and borate is greatest in alkali.[5] Characterisation and determination of the products of oxidation were aided by the use of ^{14}C-tracer techniques.

D-Glucitol was separately oxidised in water and in 0·5M-phosphate (pH 10) and 0·5M-borate buffer (pH 10) with 0·25 mol. of sodium periodate. The molar ratio of borate or phosphate to D-glucitol was 3. The products expected from the cleavage of the various C–C bonds were disclosed by chromatographic, ionophoretic, and colorimetric methods. The results shown in Table 1 indicate that 97—100% of the carbon was accounted for.

When oxidised in water, 75% of the D-glucitol remained unchanged and less than 0·1% of the carbon appeared as formic acid (Table 1). As the amount of periodate taken was only 5% of that needed for the complete oxidation of D-glucitol it is likely that the other products arose almost exclusively by direct oxidation of D-glucitol rather than by further oxidation of primary products. It is thus possible to assess the susceptibilities of the various glycol groups of D-glucitol to periodate by calculating the relative amounts of periodate used for the production of the corresponding products.

Table 2 shows that the susceptibilities of the various glycol groups of D-glucitol to periodate fall in the following order: αT > αC > α. Of the two αT- and two α-glycol groups, the 3,4- and the 5,6-group, respectively, are cleaved more readily.

Various mechanisms for the oxidation of glycols by periodate and structures of the intermediate complex, compound or ion, have been suggested. The generally accepted theory [6] is that a cyclic ester intermediate, which may be neutral or mono- or di-negatively

[1] Schwarz, *J.*, 1957, 276.
[2] Barker and Bourne, *J.*, 1952, 905.
[3] Courtois and Guernet, *Bull. Soc. chim. France*, 1957, 1388.
[4] Zuman, Sicher, Kuprička, and Svoboda, *Coll. Czech. Chem. Comm.*, 1958, **23**, 1237.
[5] Foster, *Adv. Carbohydrate Chem.*, 1957, **12**, 81.
[6] Bobbit, *Adv. Carbohydrate Chem.*, 1956, **11**, 1, and references therein.

charged, is formed from the glycol and H_5IO_6 or its dissociation products.* Buist, Bunton, and Miles [7] suggested the formation of an intermediate with a puckered five-atom ring in which the iodine atom is octahedral. They discussed the effect of the stereochemistry and electronic factors of this cyclic intermediate on the equilibrium constant for the formation of the intermediate and the rate constant for the decomposition to products. None of the proposed structures has however been proved. Nor do our results assist towards this or the reaction mechanism; but they are best understood by assuming a planar zig-zag conformation with large substituents in staggered positions,[8] the glycol group being attacked by H_5IO_6 or its dissociation products to form an intermediate with a puckered five-atom ring in which the iodine atom is octahedral.[7]

The O–O distance [9] in crystalline ammonium trihydrogen paraperiodate, $(NH_4)_2H_3IO_6$, in which the iodine is octahedral, is 2·73 Å. This is close to the calculated distance (2·83 Å) for α- and αT-glycol groups in which the large substituents on adjacent carbon atoms are fully staggered.[10] Thus, α- and αT-glycol groups can form with H_5IO_6 cyclic ester intermediates (I and II, respectively) with almost strainless five-atom rings and

TABLE 1. *Oxidation of* D-*glucitol with* 0·25 *mol. of sodium periodate in water and in borate and phosphate buffers* (pH 10).

	In water		In borate		In phosphate	
	Yield		Yield		Yield	
Product	(mol.)	C (%)	(mol.)	C (%)	(mol.)	C (%)
(D-Glucitol)	0·750	75·00	0·912	91·20	0·898	89·80
D-Arabinose	0·005	0·42	0·008	0·66	0·005	0·42
L-Xylose	0·009	0·75	0·021	1·75	0·011	0·92
D-Erythrose	0·047	3·13	0·003	2·00	0·008	0·53
L-Threose	0·015	1·00	0·001	0·07		
DL-Glyceraldehyde	0·269	13·45	0·015	0·75	0·037	1·86
Glycolaldehyde	0·085	2·83	0·072	2·40	0·082	2·73
Formaldehyde	0·015	0·25	0·016	0·27	0·012	0·20
Formic acid	0·005	0·08	0·042	0·70	0·078	1·30
Total		96·91		99·80		97·76

TABLE 2. *Percentage of periodate* (0·25 *mol.*) *consumed by the various* C–C *bonds of* D-*glucitol in water.*

C–C Bond	Type of glycol	Based on yield of:	IO_4^- used (%)	C–C Bond	Type of glycol	Based on yield of:	IO_4^- used (%)
1,2	α	D-Arabinose	2·0	4,5	αC	L-Threose	6·0
5,6	α	L-Xylose	3·6	2,3; 4,5	αT, αC	Glycollaldehyde	34·0
1,2; 5,6	α, α	Formaldehyde	6·0	3,4	αT	Glyceraldehyde	53·8
2,3	αT	D-Erythrose	18·8	Oxidation of products		Formic acid	2·0

without significant distortion of the planar zig-zag conformation of the carbon chain. αC-Glycol groups, the calculated O–O distance [10] of which is 3·68 Å, can form a cyclic intermediate (III) only after considerable distortion of the carbon chain from the planar zig-zag conformation. As the non-bonded interaction in the three cyclic intermediates is (I) < (II) < (III) the order of susceptibility to cleavage by periodate should be α- > αT- > αC-glycol groups.[11] However, α-glycol groups of D-glucitol are least readily attacked by periodate. Whereas the oxygen atoms of αT-glycol groups seem to be already in a position required for the formation of the cyclic ester intermediate, owing to the almost unhindered rotation of the hydroxymethyl groups about the C–C bonds, only a fraction, *f*, of the molecules will have the oxygen atoms of α-glycol groups in this

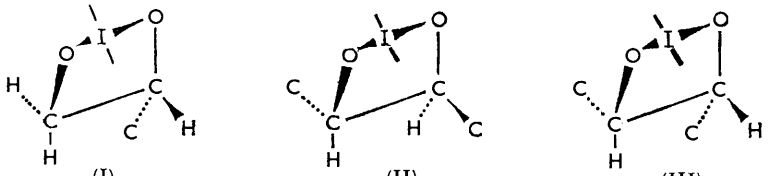
* [Added 30.11.60.] Since this paper was submitted, Keen and Symons (*Proc. Chem. Soc.*, 1960, 383) have found that ions such as $H_4IO_6^-$ or, possibly, $H_2IO_5^-$ are the major component of saturated aqueous solutions of sodium periodate.

[7] Buist, Bunton, and Miles, *J.*, 1957, 4567; 1959, 743.
[8] McCoubrey and Ubbelohde, *Quart. Rev.*, 1951, **5**, 364.
[9] Helmholtz, *J. Amer. Chem. Soc.*, 1937, **59**, 2036.
[10] Barker, Bourne, and Whiffen, *J.*, 1952, 3865.
[11] Barton and Cookson, *Quart. Rev.*, 1956, **10**, 44.

position and an entropy term, $RT \ln 1/f$, has to be added to the free-energy change involved in the reaction. This could make α-glycol groups less susceptible to oxidation by periodate than αT-glycol groups of D-glucitol. For αC-glycol groups, a compression energy term has to be added to the free-energy change of the reaction. It is possible that the difference between these two terms makes the α-glycol groups of D-glucitol least readily attacked by periodate.

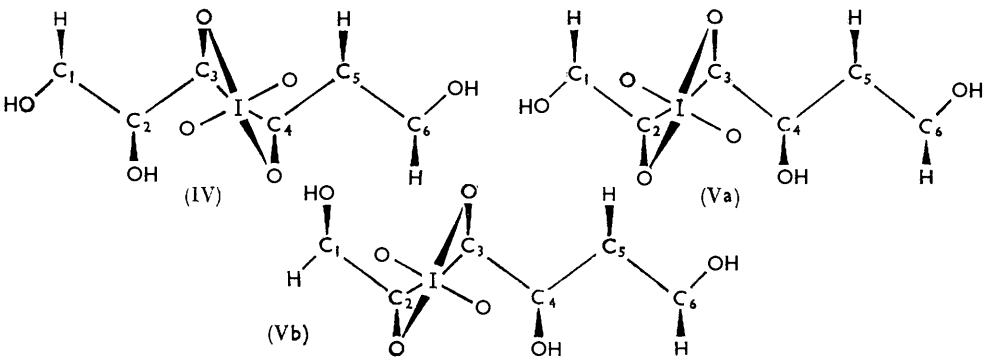
The difference in ease of cleavage of the two αT-glycol groups can be due to the stereochemistry of the ester intermediates. In (IV) and (V) the hypothetical intermediates involving $C_{(3)}$–$C_{(4)}$ and $C_{(2)}$–$C_{(3)}$, respectively, are depicted with a puckered five-atom ring in which the iodine atom is octahedral. In (IV) and (Va) the 1- and 6-hydroxyl groups extend the planar zig-zag conformation of the carbon chain: in both cases a hydroxyl group and a hydrogen atom, $HO_{(2)}$, and $H_{(5)}$, $HO_{(4)}$ and $H_{(1)}$, respectively, lie close to two of the oxygen atoms attached to the iodine atom. The situation remains the same when the hydroxymethyl groups of (IV) rotate freely. However, a fraction of the molecules of (V) will have the 1-hydroxyl group in a position as shown in (Vb), in which two hydroxyl groups, $HO_{(1)}$ and $HO_{(4)}$, lie close to two of the oxygen atoms attached to the iodine atom. The resulting greater steric compression could make the αT-glycol group involving $C_{(2)}$ and $C_{(3)}$ less susceptible to oxidation by periodate than that involving $C_{(3)}$ and $C_{(4)}$.

Since it is established that αT-glycol groups are more readily oxidised by periodate than αC-glycol groups it is reasonable to assume that, in a statistical sense, the 5-hydroxyl group is more readily available for the formation of an intermediate involving $C_{(5)}$ and



(I) (II) (III)

Four of the oxygen atoms attached to the iodine atom are omitted. Two of these lie towards the observer in positions equivalent to the oxygen atoms attached also to the carbon atoms.



(IV) (Va) (Vb)

Atoms lying below the plane of the carbon atoms and two of the oxygen atoms attached to the iodine atom are omitted. The latter lie towards the observer in positions equivalent to O_3, O_4 and O_2, O_3, respectively.

$C_{(6)}$ than the 2-hydroxyl group for the formation of an intermediate involving $C_{(1)}$ and $C_{(2)}$. Thus, of the two α-glycol groups, the 5,6-bond is more readily cleaved than the 1,2-bond.

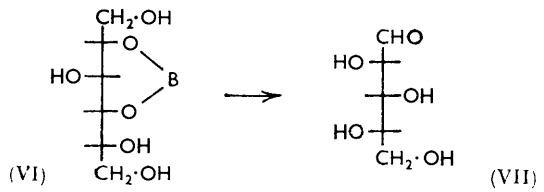
It was now possible to examine the effect of borate ions on the reaction. From the sequence of M_G values of substituted aldoses in borate solution at pH 10, Foster [5] concluded that the *aldehydo*-form of D-glucose is the principal one involved in complex formation

and the pair of the 2- and the 4-hydroxyl group are sterically most favourable for complex formation. This, and the relative stabilities of cyclic acetals of D-glucitol,[10] suggested that a similar complex is favoured for D-glucitol. Partial periodate oxidation of such a complex (VI) should yield a larger quantity of L-xylose (VII) than a reaction in the absence of borate.

There was a possibility of anomalous reactions at pH 10.[7] Certain discrepancies have been found in the periodate oxidation in phosphate buffer at pH 7·5.[12] It was thought that similar discrepancies might arise with a borate buffer and that comparison between the oxidation of D-glucitol in 0·5M-borate (pH 10) and 0·5M-phosphate buffer (pH 10) would be valid.

Oxidation of D-glucitol with 0·25 mol. of sodium periodate in the presence of borate (pH 10) or phosphate (pH 10) yielded very different results from those obtained with the unbuffered solution (Table 1). Only *ca.* 10% of the D-glucitol was oxidised in the alkaline solutions whereas the theoretical amount of 25% was oxidised in the unbuffered solution. Extensive secondary reactions (cf. formic acid production) under the alkaline conditions made detailed comparison with the unbuffered solution impossible.

The ratios of the yields of L-xylose to D-arabinose (primary reactions) in the unbuffered, borate-, and phosphate-buffered solutions are 1·8, 2·6, and 2·2, respectively. The ratios of the yield of L-xylose in borate- and phosphate-buffered solutions relative to that in the unbuffered solution are 2·3 and 1·2, respectively. This indicated that the yield of L-xylose could be significantly increased by the presence of borate ions. Accordingly, D-glucitol dissolved in 4M-borate buffer (pH 10·7) (borate to D-glucitol ratio 5 : 1) was oxidised with 2·5 mol. of sodium periodate. The component which had been characterised by carrier



(VI) (VII)

dilution analysis as L-xylose was the major product. This was separated by paper chromatography and determined quantitatively. It was found that the yield of L-xylose could be raised in this way to *ca.* 30%.

The results indicate that D-glucitol forms a complex with borate ions similar to that of D-glucose,[5] *i.e.*, participation of $HO_{(2)}$ and $HO_{(4)}$. However, a complex involving $HO_{(1)}$ and $HO_{(4)}$ is not excluded as the resulting 7-atom ring could have the 2- and the 3-hydroxyl group in *trans*-relation, when they would react with periodate more slowly than a $CH(OH)\cdot CH_2\cdot OH$ group.[13] On the other hand, it has been shown [14] that borate ions have no tendency to form complexes involving 7-atom or larger rings. The results clearly establish that the principal site of attack of periodate on the D-glucitol–borate complex is the 5,6-bond. The pair of hydroxyl groups involved in the formation of the complex are probably those on $C_{(2)}$ and $C_{(4)}$.

It is of interest that complex-formation with boric acid has been used similarly to effect selective substitution in D-glucose,[15,16] D-mannitol,[15,17] D-glucose diethyl mercaptal,[15] and methyl α- and β-glucopyranoside,[18] and to block the oxidation of carbohydrates in tissues by lead tetra-acetate.[19]

[12] Bell, Palmer, and Johns, *J.*, 1949, 1536; Bell and Greville, *J.*, 1950, 1902.
[13] Bourne, Hartigan, and Weigel, *J.*, 1961, 1088.
[14] Frahn and Mills, *Chem. and Ind.*, 1956, 578.
[15] Brigl and Grün, *Annalen*, 1932, **495**, 60.
[16] von Vargha, *Ber.*, 1933, **66**, 704.
[17] von Vargha, *Ber.*, 1933, **66**, 1394.
[18] Bell, *J.*, 1935, 175; Sugihari and Peterson, *J. Amer. Chem. Soc.*, 1956, **78**, 1760.
[19] Staple, *Nature*, 1955, **176**, 1125; *J. Histochem. Cytochem.*, 1957, **5**, 472.

Experimental

Materials.—D-[^{14}C]Glucitol, uniformly labelled, was obtained from the Radiochemical Centre, Amersham. Glycollaldehyde was prepared as described by Powers *et al.*[20]

Chromatography.—The solvents used in chromatography were (*a*) butan-1-ol–benzene–pyridine–water (5 : 1 : 3 : 2); (*b*) ethyl acetate–acetic acid–water (9 : 2 : 2); (*c*) methyl ethyl ketone saturated with water; [1] (*d*) butan-1-ol–ethanol–water (4 : 1 : 5) (organic phase).

Determination of Radioactivity.—Radioactivities were determined after conversion of the compound into carbon dioxide, and thence into barium carbonate.[21] The amount used was sufficient to give a thickness of greater than 20 mg. per cm.^{2}. The β-emission of a radioactive specimen was measured by means of a Geiger–Müller end-window counter and for times sufficient to give a standard counting error of less than ±2%.

Periodate Oxidations.—To mixtures of 0·166M-aqueous D-glucitol (1 mol.) with 0·5M-borate buffer (pH 10·6; 3 mol.) or 0·5M-phosphate buffer (pH 10·1; 3 mol.) (final pH 10·0) was added standard sodium periodate solution (0·25 mol.). After 10 min., Amberlite IR-120(H$^+$) was stirred into the buffered solutions to adjust the pH to 5.

Identification of Products.—D-Glucitol, D-arabinose, L-xylose, and DL-glyceraldehyde were identified by paper chromatography in solvents (*a*) and (*b*) and by treatment of the chromatograms with acetone–silver nitrate–alcoholic sodium hydroxide [22] or *p*-anisidine hydrochloride in butanol.[23] D-Erythrose and L-threose were separated from other products by paper chromatography in solvent (*c*),[1] resolved, and identified by paper ionophoresis in molybdate solution.[24] Glycollaldehyde was shown to be present by treatment with diphenylamine in acetic acid and measurement of absorption at 680 mμ.[25] Formaldehyde was shown to be present by the chromotropic acid method.[26] Formic acid was assumed to constitute the total titratable acid present.

Characterisation and Determination of Products.—(i) *D-Glucitol, D-arabinose, and L-xylose.* D-[^{14}C]Glucitol was separately oxidised with sodium periodate (0·25 mol.) in water, 0·5M-borate buffer (pH 10), and 0·5M-phosphate buffer (pH 10) as described above. A carrier compound (D-glucitol, D-arabinose, or L-xylose) was dissolved in each solution and allowed to equilibrate overnight. Boric acid was removed by repeated distillation with methanol, and phosphoric acid by precipitation with aqueous barium hydroxide. The products were separated by chromatography on Whatman paper no. 3 in solvent (*a*). D-[^{14}C]Glucitol was converted into the hexa-acetate which was recrystallised from ethanol until consecutive samples possessed constant m. p. and specific radioactivity. D-Arabinose and L-xylose were converted into their phenylosazones which were recrystallised twice from water and twice from benzene, after which consecutive samples possessed constant m. p. and specific radioactivity. The details of the analysis are shown in Table 3.

(ii) *D-Erythrose and DL-glyceraldehyde.* D-[^{14}C]Glucitol samples were oxidised with sodium periodate (0·25 mol.) as described above. The solutions were treated with potassium borohydride at room temperature for 12 hr. and then with Amberlite IR-120(H$^+$). Erythritol and glycerol were added as carrier compounds. After equilibration for 1 hr. boric acid was removed by repeated distillation with methanol. [^{14}C]Erythritol and [^{14}C]glycerol were separated by chromatography on Whatman paper no. 3 in solvent (*d*) and converted into the tetra- and tri-benzoate, respectively, which were recrystallised from aqueous pyridine and aqueous ethanol, respectively, until consecutive samples possessed constant m. p. and specific radioactivity. The details of the analysis are shown in Table 3.

(iii) *L-Threose.* D-[^{14}C]Glucitol (20 mg.) was separately oxidised with sodium periodate (0·25 mol.) in water and 0·5M-borate buffer (pH 10) as described above. The borate-buffered solution was treated with Amberlite IR-120(H$^+$), and the boric acid removed as above. L-[^{14}C]Threose and D-[^{14}C]erythrose were separated by paper chromatography in solvent (*c*).

[20] Powers, Tabakoglu, and Sable, *Biochem. Prep.*, 1955, **4**, 56.
[21] Skipper, Bryan, White, and Hutchison, *J. Biol. Chem.*, 1948, **73**, 371; Calvin, Heidelberger, Reid, Tolbert, and Yankwich, "Isotopic Carbon," Wiley, New York, 1949; Henriques, Kistiakowsky, Margnetti, and Schneider, *Ind. Eng. Chem. Analyt.*, 1946, **18**, 349.
[22] Trevelyan, Procter, and Harrison, *Nature*, 1950, **166**, 444.
[23] Hough, Jones, and Wadman, *J.*, 1950, 1702.
[24] Bourne, Hutson, and Weigel, *Chem. and Ind.*, 1959, 1047.
[25] Dische and Borenfreund, *J. Biol. Chem.*, 1949, **180**, 1297.
[26] Adcock, *Analyst*, 1957, **82**, 427.

The eluted tetroses were subjected to ionophoresis in molybdate solution (pH 5).[24] *p*-Anisidine hydrochloride in butanol and ultraviolet light [23] revealed the positions of the tetroses on the ionophoretograms. From a comparison of the β-emission of the two spots the ratio of the yields of L-threose and D-erythrose was found. The yield of L-threose was calculated from the results of the determination of D-erythrose. The results are shown in Table 4.

TABLE 3. *Carrier dilution analysis of products from oxidation of* D-[^{14}C]*glucitol with 0·25 mol. of sodium periodate.*

W_G (mg.)	S_o (μc per g.-atom of carbon)	Product	Medium	W_c (mg.)	M. p. of deriv.	S_i (μc per g.-atom of carbon)	Yield (mol.)
72	3164	(D-Glucitol)	Water	250	99°	561·90	0·750
72	3164	D-Arabinose	Water	254	163	3·43	0·005
72	3164	L-Xylose	Water	341·4	164	5·03	0·009
72	3164	D-Erythrose *	Water	242·3	185	29·40	0·047
72	3164	DL-Glyceraldehyde †	Water	254	72	117·52	0·269
72	3164	(D-Glucitol)	Borate	247	99	664·40	0·912
72	3164	D-Arabinose	Borate	233	163	6·14	0·008
72	3164	L-Xylose	Borate	226	164	17·54	0·021
72	3164	D-Erythrose *	Borate	256	185	1·77	0·003
72	3164	DL-Glyceraldehyde †	Borate	242·3	72	7·06	0·015
400	821	(D-Glucitol)	Phosphate	724·7	98	272·00	0·898
72	3164	D-Arabinose	Phosphate	275·1	163	3·24	0·005
72	3164	L-Xylose	Phosphate	195·5	164	10·25	0·011
72	3164	D-Erythrose *	Phosphate	255·3	185	4·71	0·008
72	3164	DL-Glyceraldehyde †	Phosphate	242·3	72	17·66	0·037

W_G = Weight of D-[^{14}C]glucitol oxidised. S_o = spec. radioactivity of D-[^{14}C]glucitol. W_c = weight of carrier added. S_i = spec. radioactivity of parent compound of isolated derivative.
* Carrier compound was erythritol. † Carrier compound was glycerol.

TABLE 4. *Determination of* L-*threose from oxidation of* D-[^{14}C]*glucitol with 0·25 mol. of periodate.*

Product	Medium	Radioactivity (counts per min.)	Ratio, L-threose : D-erythrose	Yield of L-threose * (mol.)
L-Threose	Water	216	0·31	0·015
D-Erythrose	Water	693		
L-Threose	Borate (pH 10)	50	0·23	0·001
D-Erythrose	Borate (pH 10)	222		

* Based on yield of D-erythrose.

(iv) *Glycollaldehyde.* The results of Dische and Borenfreund [25] show that glycollaldehyde, after treatment with diphenylamine in acetic acid, can be determined by measurement of the optical density at 680 mμ and that the presence of glyceraldehyde will cause an error not greater than *ca.* 3%. Measurement of the optical density of standard solutions of glycollaldehyde (0·1%) containing also D-erythrose, formate, phosphate, borate, and iodate showed that only the latter interfered seriously with the determination of glycollaldehyde. Iodate was thus removed from the unbuffered and phosphate-buffered reaction mixtures by treatment with Amberlite IRA-400(OAc). To avoid possible loss of glycollaldehyde by adsorption on the resin as the glycollaldehyde–borate complex, the borate-buffered reaction mixture was treated with much glucitol, with which borate reacts preferentially, before treatment with the resin.

D-Glucitol (0·91 g.) was oxidised as described above. After removal of the iodate, glycollaldehyde was determined colorimetrically.[25] The measurement of the optical densities at 680 mμ (Ilford filter no. 608) of the solutions obtained from the unbuffered and borate- and phosphate-buffered mixtures corresponded to yields of 0·085, 0·072, and 0·082 mole of glycollaldehyde per mole of D-glucitol, respectively.

(v) *Formaldehyde.* D-Glucitol (0·91 g.) was oxidised as described above. After removal by steam-distillation from the reaction mixtures, formaldehyde was determined by the chromotropic acid method.[26] Measurement of the optical densities (Ilford filter no. 606) of the solutions obtained from the unbuffered and borate- and phosphate-buffered mixtures corresponded to yields of 0·015, 0·016, and 0·012 mole respectively of formaldehyde per mole of D-glucitol.

(vi) *Formic acid.* D-Glucitol (0·91 g.) was separately oxidised as described above. Formic acid was determined by titration with 0·01N-sodium hydroxide, a pH-meter being used for end-point detection. A blank titre was found after oxidation of ethylene glycol with 0·125 mol. of sodium periodate. From the buffered solutions the formic acid was first separated by acidification and distillation of the reaction mixture. True titres: 2·5 ml. (water), 21·0 ml. (borate buffer), 39·0 ml. (phosphate buffer). These correspond to yields of 0·005, 0·042, and 0·078 mole, respectively, of formic acid per mole of D-glucitol.

Determination of L-*Xylose from Oxidation of* D-*Glucitol in* 4M-*Borate Buffer* (5 *Mol.*) *with* 2·5 *Mol. of Sodium Periodate.*—To a solution of D-glucitol (1·82 g.) in 4M-borate buffer (pH 10·7) (12·5 ml.) was added a solution of sodium periodate (5·45 g.) in water (25 ml.). After 10 min. sodium ions and boric acid were removed by treatment with Amberlite IR-120(H^+) and repeated distillation with methanol, respectively. The residue was extracted with methanol. Paper chromatography in solvent (*a*) showed that the major component had an R_F value identical with that which had been characterised by carrier dilution analysis as L-xylose. Elution and quantitative determination of this component by the anthrone method [27] showed a 30·6% yield.

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[27] Bailey, *Biochem. J.*, 1958, **68**, 669.