

227. *The Constitution of Xylan from the Green Seaweed Caulerpa filiformis.*

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After alcoholic and aqueous extraction of *Caulerpa filiformis*, dilute alkaline extraction of the residual weed affords a xylan. Methylation and other studies provide evidence for a structure comprising chains of β -D-xylopyranose residues linked between C₍₁₎ and C₍₈₎.

COMPARATIVELY little is known about the polysaccharides of the green seaweeds. Water-soluble materials from *Cladophora rupestris*¹ and from *Ulva lactuca*² consist of complex polysaccharide material containing a wide variety of sugar residues. In spite of repeated fractionation with various reagents, no separation of these materials was achieved. Chloroform extraction of acetylated cladophoran did, however, give a small yield of a glucose-rich fraction. Preston and his colleagues,³ examining the cell-wall material of a number of green seaweeds by X-ray and hydrolytic methods, have also revealed the complexity of their polysaccharide constituents.

After removal of water-soluble sulphated polysaccharide material (which contains glucose, galactose, mannose, xylose, rhamnose, and uronic acid residues) from the green seaweed, *Caulerpa filiformis*, mild treatment of the insoluble residue with chlorite⁴ was followed by extraction with dilute sodium hydroxide solution at room temperature. Acidification of the alkaline extract afforded a crude xylan (ca. 5.0% of the dried weight of weed) (Ash, 2.0; SO₄²⁻, 3.6%) comprising xylose (90%) and glucose (10%). Precipitation with various copper salts^{1,5,6} or with Cetavlon⁷ failed to reduce the glucose content, but extraction with water and dialysis afforded xylan (B) (4.5%) containing ca. 4% of glucose (ash, 0.5%; SO₄²⁻, nil). A pure xylan (C) (3.3% dried weight of weed) devoid of glucose and sulphate residues was isolated after exhaustive extraction of material (B) with water. In view of the limited quantity of weed available, and the loss of xylan during complete purification, large-scale investigations were carried out on polysaccharide (B). The extraction of the weed and its treatment with chlorite were carried out under mild conditions in order to minimise the possible degradation of the xylan. During the purification no obvious signs of degradation were apparent; in particular the specific viscosities and periodate consumption of xylan (B) and xylan (C) were similar.

Xylan (B) afforded 92% of crystalline D-xylose on hydrolysis. By two series of five methylations, each with methyl sulphate and sodium hydroxide in an atmosphere of nitrogen,⁵ a methylated xylan was prepared containing 31.5% of methoxyl. Four treatments with Purdie's reagents gave a product containing 37.2% of methoxyl. Fractionation using chloroform-light petroleum separated a negligible quantity of lower methylated material (OMe, 22%). The major fractions (OMe, 37–37.4%) were recombined and methylated with sodium and methyl iodide in liquid ammonia.⁸ The isolated material had $[\alpha]_D^{18} -60^\circ$ and contained 37.8% of methoxyl (Calc. for C₇H₁₄O₄: OMe, 38.7%). Repeated methylation and dialysis had not apparently degraded the xylan seriously, since a xylan acetate prepared by mild acetylation had a specific viscosity of the same order as had the methylated xylan measured under identical conditions.

The methylated xylan was hydrolysed with methanolic hydrogen chloride giving the glycosides, and from them the methylated sugars were obtained by hydrolysis with dilute

¹ Fisher and Percival, *J.*, 1957, 2666.

² Brading, Georg-Plant, and Hardy, *J.*, 1954, 319.

³ Cronshaw, Myers, and Preston, *Biochim. Biophys. Acta*, 1958, **27**, 89.

⁴ Wise, *Ind. Eng. Chem. Anal.*, 1945, **17**, 63.

⁵ Chanda, Hirst, Jones, and Percival, *J.*, 1950, 1289.

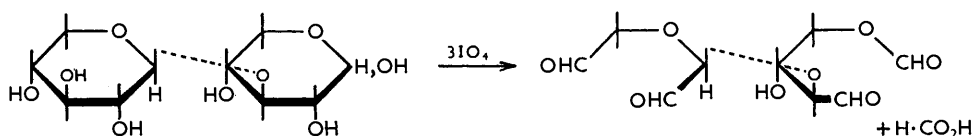
⁶ Erskine and Jones, *Canad. J. Chem.*, 1956, 821.

⁷ Bera, Foster, and Stacey, *J.*, 1955, 3788.

⁸ Hodge, Karjala, and Hilbert, *J. Amer. Chem. Soc.* 1951, **73**, 3312.

hydrochloric acid. Separation on a cellulose column gave the following molar percentage separation of methylated xyloses: 2 : 3 : 4-tri-*O*-methylxylose (2.9), 2 : 4-di-*O*-methylxylose (95.2) and a mixture of 2- and 4-monomethylxylose (1.9). Both the tri- and the di-*O*-methyl sugars were crystalline. The amount of trimethylxylose corresponds to one non-reducing end group for every 34 xylose residues, but this figure can only be regarded as very approximate as the weight of each fraction was recorded after extensive purification. The value of one non-reducing end group for every 47 anhydro-xylose units (average of two experiments), determined on the methylated hydrolysate by hypiodite oxidation after separation on a paper chromatogram,⁵ is probably a more accurate figure. The presence of monomethylxylose (*ca.* 1.9%) is easily accounted for on the grounds of under-methylation, and of demethylation during hydrolysis. Taken as a whole the methylation results show that the xylan is made up of linear chains of 1 : 3-linked β -D-xylopyranose residues, and that the majority, at least, of the chains are unbranched; the β -configuration being inferred from the negative rotation.

Periodate-oxidation studies were undertaken to obtain further evidence on the constitution of this polymer. A linear 1 : 3-linked xylan, with a reducing group at one end of the molecule, would be attacked by periodate only at the ends of the chains with the reduction of 3 mols. of periodate and the formation of 1 mol. of formic acid and a stable formyl ester, but no formaldehyde.⁹ On this assumption the consumption of one mol. of



periodate for about 14 xylose units gives an average chain length of 42, and the yield of formic acid gives a similar figure, since about 1 mol. of formic acid is liberated from 43 xylose units. The small yield of formaldehyde may be due to a small amount of hydrolysis of the formyl ester during the oxidation. On the other hand it is very probable that during the extraction of the xylan with alkali some modification of the reducing group takes place. In the absence of definite knowledge regarding the nature of this group the interpretation of the periodate oxidation results can only be regarded as tentative. The liberation of free xylose (90%) on acid hydrolysis of the oxopolysaccharide does, however, provide further evidence for a 1 : 3-xylosidic linkage.¹⁰

The contaminating glucose in xylan (B) was separated from the methylated hydrolysate as 2 : 3 : 4 : 6-tetra-*O*-methylglucose (trace, paper chromatography), crystalline 2 : 4 : 6-tri-*O*-methylglucose, and a small quantity of 4 : 6-di-*O*-methylglucose. Taken in conjunction with the small change in rotation of the xylan, on removal of this glucan, all the evidence points to this polymer's being of the laminarin type with 1 : 3- β -linked gluco-pyranose units. The periodate consumption of such a molecule would be of the same order as that of the xylan, since again only the end residues are liable to attack by this reagent. Evidence for this is the very similar uptake of periodate by xylan (B) (containing 4% of glucose) and by xylan (C) which is glucose-free.

Bearing in mind all the results it seems justifiable to assign to the xylan from *Caulerpa filiformis* a structure containing linear chains of D-xylopyranose units joined by 1 : 3- β -linkages.

Although xylose is a constituent of the polysaccharides of many green seaweeds³ this is the first time a pure xylan has been isolated from this source. 1 : 4- β -Linked xylans constitute the major polysaccharide of the hemicelluloses of land plants and a xylan containing 80% of 1 : 4-linked and 20% of 1 : 3-linked β -D-xylopyranose units has been

⁹ Hough, Taylor, Thomas, and Woods, *J.*, 1958, 1212.

¹⁰ Hough and Perry, *Chem. and Ind.*, 1956, 768.

separated from the red seaweed, *Rhodymenia palmata*.¹¹ The present xylan is unique in consisting solely of 1 : 3-linked residues.

EXPERIMENTAL

Paper partition chromatography was carried out on Whatman No. 1 filter paper. Paper ionophoresis was in borate buffer (pH 10) at 750 v for 5 hr. Evaporations were carried out at 40°/15 mm.

The green seaweed, *Caulerpa filiformis*, collected from rock pools near Cape Town in February, 1957, was kindly made available to us by Dr. A. M. Stephen and Mr. R. H. Simons. The weed was dried at 60° and ground to a pale green powder. Extraction of this dried powder (15 g.) with 80% aqueous alcohol under reflux removed most of the colouring matter. Chromatographic examination of the alcoholic extract, after deproteinisation¹² and deionisation,¹³ indicated the presence of sucrose and glucose. The residual weed was extracted with dilute hydrochloric acid (pH 3—4) at 70° for 6 hr. (twice) and the combined extracts, after dialysis and freeze-drying, gave an off-white powder (Found: N, 2.5%). Subsequent extracts were therefore treated with trichloroacetic acid before dialysis. After several days the precipitated protein was removed on the centrifuge and the supernatant liquid dialysed against running water for several days. Concentration of the dialysed solution followed by freeze-drying gave an off-white powder (1.5 g.), $[\alpha]_D + 120^\circ$ (*c* 3.0 in water) [Ash (direct), 13.5; (as sulphate), 15.4; SO₄²⁻ in ash, 3.8% of dry weight of polysaccharide; total SO₄²⁻, 7.5; uronic anhydride, *ca.* 8; N, 1.5%].

Chromatography of the hydrolysate (*N*-sulphuric acid at 100° for 4 hr.) by the method of Flood, Hirst, and Jones¹⁴ (*a*) and of Pridham¹⁵ (*b*) showed that the polysaccharide material contained the following approximate molar proportions of the sugars: galactose (*a*) 4, (*b*) 4; glucose (*a*) 14, (*b*) 15; mannose (*a*) 2, (*b*) 2; xylose (*a*) 2, (*b*) 1; and rhamnose (*a*) 0.5, (*b*) 0.2. The presence of anhydro- and/or ketose sugar residues in the hydrolysate and in the polysaccharide was indicated by the red colour given by a Seliwanoff test,¹⁶ and the yellow-brown spots produced on filter paper with the anthrone reagent.¹⁷ Extraction with cold and hot water and with 0.5% sodium carbonate solution of the alcohol-extracted weed gave similar polysaccharide material (visual chromatographic examination of the hydrolysate).

Extraction of a Xylan.—The residual weed (from 50 g. of original dried weed), after extraction with alcohol and dilute acid, was heated to 70° in water (250 c.c.) containing glacial acetic acid (1 c.c.) and sodium chlorite⁴ (4 × 3 g.) was added at hourly intervals during 4 hr. The weed was then separated at the centrifuge, washed with water and extracted with *N*-sodium hydroxide (125 c.c.) at room temperature with stirring during 3 days. The alkaline solution, freed from weed, was acidified with glacial acetic acid to pH 5. A gel-like precipitate was deposited which was washed with water to remove acid, followed by alcohol of increasing concentration and finally with ether. The white, powdery solid (2.5 g.), after drying over phosphoric oxide in a vacuum-desiccator, had $[\alpha]_D - 28^\circ$ (*c* 0.8 in 0.1*N*-NaOH) (Ash, 2.0; SO₄²⁻, 3.6%). Hydrolysis and quantitative chromatography¹⁴ showed that the polysaccharide contained xylose (90%) and glucose (10%).

Purification of the Xylan.—The xylan was precipitated from alkaline solution as the copper complex with Fehling's solution,⁵ with copper acetate,⁶ and with Benedict's solution.¹ Precipitation was also brought about with 10% aqueous cetyltrimethylammonium bromide.⁷ Hydrolysates from each of the regenerated xylans contained *ca.* 10% of glucose. Suspension of the crude polysaccharide (5 g.) in water (500 c.c.) with agitation for 24 hr. gave on filtration and drying a white powder (A) (4.75 g.), $[\alpha]_D - 31^\circ$ (*c* 0.5 in 0.5*N*-NaOH) (Found: SO₄²⁻, 2.4%). After dialysis against running water for 4 days the polysaccharide (4.5 g.) was isolated by freeze-drying of the aqueous suspension. This material (B) was devoid of sulphate and contained xylose 95%,¹⁵ 97%,¹⁴ and glucose 5%,¹⁵ 3%¹⁴ (ash, 0.5%). Crystalline xylose, *m. p.* and mixed *m. p.* 143—144°, $[\alpha]_D + 41.9^\circ$ (*c* 0.9), was separated from the hydrolysate in 92% yield. This

¹¹ Chanda and Percival, *Nature*, 1950, **166**, 787.

¹² Laidlaw and Reid, *J. Sci. Food Agric.*, 1952, **3**, 19.

¹³ Anderson and Wylam, *Chem. and Ind.*, 1956, 191.

¹⁴ Flood, Hirst, and Jones, *J.*, 1949, 1659.

¹⁵ Pridham, *Analyt. Chem.*, 1956, 1967.

¹⁶ Seliwanoff, *Ber.*, 1887, **20**, 181.

¹⁷ Johanson, *Nature*, 1953, **172**, 956.

was further characterised as the dibenzylidene dimethyl acetal derivative, m. p. mixed m. p. 210°. Repeated extraction of polysaccharide (B) (1.5 g.) with water finally led to the isolation of xylan (C) (1.1 g.), $[\alpha]_D -35^\circ$, containing only xylose residues.

Periodate Oxidation of Xylan.—(i) *Uptake of periodate.* Dry xylan (B) (361 mg.), (C) (312.4 mg.) was oxidised with 3% sodium metaperiodate (50 ml.) in the dark and the reduction of periodate was measured.¹⁸ The moles of periodate consumed per $C_5H_8O_4$ unit were determined: (B) 0.015 (3 hr.), 0.026 (5 hr.), 0.055 (22 hr.), 0.064 (42 hr.), 0.070 (66 hr.), 0.070 (114 hr. const.). (C) 0.021 (5 hr.), 0.040 (11 hr.), 0.045 (24 hr.), 0.072 (48 hr.), 0.074 (70 hr.), 0.074 (96 hr. const.). The completely oxidised solution (170 hr.), after destruction of excess of periodate with ethylene glycol, was dialysed against distilled water until free from inorganic ions (3 days) and concentrated at 35°/15 mm. to 10–15 c.c. The oxopolysaccharide from (B) (337.8 mg., 98%), isolated from this solution by freeze-drying, was hydrolysed with N-sulphuric acid. The resulting syrup contained xylose¹⁴ (304 mg.) and a trace of glucose.

(ii) *Determination of formic acid released.* Dry xylan (B) (498 mg.) was weighed into a dark stoppered bottle and treated with sodium metaperiodate (50 c.c.; 0.1871M) with continuous agitation. Samples (5 c.c.) were removed at intervals, treated with ethylene glycol (1 c.c.), and titrated with 0.01N-sodium hydroxide from a micro-burette (Methyl Red). The following results (moles $\times 10^{-2}$) of $H-CO_2H$ per $C_5H_8O_4$ unit were obtained: 1.5 hr., 0.37; 3 hr., 0.37; 22 hr., 0.75; 46 hr., 1.1; 94 hr., 1.6; 166 hr., 2.1; 262 hr., 2.3; 298 hr., 2.35; 324 hr., 2.4.

(iii) *Formaldehyde release.* Dry xylan (B) (189.6 mg.) was oxidised with sodium metaperiodate (50 c.c.; 0.025M) and the $\mu g.$ of formaldehyde released measured at intervals:¹⁹ 1 hr., 1.8; 5 hr., 2.6; 23 hr., 5.9; 48 hr., 9.7; 72 hr., 10.6.

Reducing Power of the Xylan.—*Hypoiodite oxidation.* The polysaccharide (B) (100 mg.) in water was treated with 2N-sodium hydroxide (10 c.c.), followed by 0.1N-iodine (10 c.c.), and the reducing power measured as for the esparto grass xylan.⁵ This gave a value of one reducing group per 60 xylose residues.

Methylation of Xylan.—The polysaccharide (B) (8.0 g.) was methylated by sodium hydroxide and methyl sulphate.⁵ After two series of five methylations the product was a white powder (8.7 g.; OMe, 31.5%). This partly methylated material was subjected to four further methylations with methyl iodide and silver oxide. The dried (over P_2O_5 to constant weight) white powder (Found: OMe, 37.2; Calc. for $C_7H_{12}O_4$: OMe, 38.75%) had $[\alpha]_D -61^\circ$ (*c* 1.0 in $CHCl_3$).

Fractionation of the Methylated Xylan.—The methylated xylan (4.2 g.) was fractionated by various mixtures of chloroform and purified light petroleum (b. p. 65–66°). The material started dissolving in 20 : 80 chloroform–light petroleum and was completely soluble in a 35 : 65 mixture. The results are given below:

Fraction	Solvent chloroform– light petroleum	Yield (mg.)	OMe	$[\alpha]_D$
1	0 : 100	0	—	—
2	20 : 80	15	22.1	–26°
3	25 : 75	43	37.0	–61
4	30 : 70	727	37.4	–62
5	35 : 65	2560	37.1	–62

Fractions 3–5 were recombined.

Methylation in Liquid Ammonia.—The above combined fractions 3–5 (3.3 g.) were thoroughly dried and introduced into a flask, fitted with a stirrer, which could be sealed against the atmosphere.⁹ Ammonia (200 c.c.) was condensed in the flask, and metallic sodium (240 mg.) was added with stirring. After 15 min. methyl iodide (1.0 c.c.) was added. Four further additions of sodium and of methyl iodide were made alternately at 15 min. intervals. The ammonia was allowed to evaporate at room temperature (2 hr.) and then at 50° (1 hr.). Sodium iodide and sodium amide were dissolved by the dropwise addition of water (50 c.c.), and the methylated polysaccharide extracted with chloroform (3×100 c.c.). It was precipitated from the dried concentrated chloroform solution as a white powder (2.97 g.) by the addition of ether, and had $[\alpha]_D^{18} -60^\circ$ (*c* 1.0 in $CHCl_3$) and OMe, 37.8%.

Hydrolysis of Methylated Xylan and Separation of Methylated Xyloses.—(a) *By paper chromatography.* The polysaccharide (60 mg.) was treated with 3% methanolic hydrogen chloride (10 c.c.) under reflux for 18 hr., and then hydrolysed with N-hydrochloric acid (10 c.c.) for 18 hr.

¹⁸ Halsall, Hirst, and Jones, *J.*, 1947, 1399, 1427.

at 100°. After neutralisation with silver carbonate the filtrate was de-ionised with Amberlite IR120(H) and IR45(OH) resins. The clear solution was concentrated to a syrup which crystallised completely overnight. Chromatography with butanol-ethanol-water (4:1:5), and development with aniline oxalate, showed three main spots having R_G 0.94, 0.66, and 0.40, identical with 2:3:4-tri-*O*-methylxylose, 2:4-di-*O*-methylxylose, and a mono-*O*-methylxylose respectively. A fourth faint buff-coloured spot, R_G 0.76, was also detected. This is shown later to be due to a trimethyl-glucose.

The methylated pentoses were estimated by alkaline hypoidite after separation on the paper chromatogram:⁵

Sugar	Wt. (mg.)	Molar composition (%)
2:3:4-Tri- <i>O</i> -methylxylose	0.43; 0.42	2.2; 2.1
2:4-Di- <i>O</i> -methylxylose	17.78; 17.66	96.5; 96.6
Mono- <i>O</i> -methylxylose	0.23; 0.21	1.3; 1.2

(b) *By the cellulose column.* Methylated xylan (2.87 g.) was boiled gently under reflux with 3% methanolic hydrogen chloride (300 c.c.), and the rotation observed: $[\alpha]_D -62^\circ$ (initial) $\rightarrow +65^\circ$ (6 hr., constant). After neutralisation with silver carbonate the filtrate was concentrated to a syrup at 35°/15 mm. This was hydrolysed with *N*-hydrochloric acid (300 c.c.) at 95° and then had $\alpha_D +26^\circ$ (18 hr. constant). Neutralisation of the cooled solution with silver carbonate, followed by deionisation of the filtrate with hydrogen sulphide and Amberlite resins IR120 and IR45 and concentration, gave crystals (2.085 g.). The crystals, dissolved in water, were mixed with cellulose powder and the whole was freeze-dried. The freeze-dried material was packed on top of a cellulose column (3.6 × 70 cm.) which was eluted with light petroleum (b. p. 100–120°)-butan-1-ol (7:3) saturated with water. After 9.5 l. had been collected the solvent was changed to butan-1-ol (100 c.c.) and to butan-1-ol half saturated with water in order to elute the monomethylxylose more quickly. After another 1.5 l. had been eluted the column was washed with water. Six fractions were collected. The fractions were weighed after filtration through "Filter Cel," concentration to dryness, dissolution in methanol, filtration, and concentration. The total recovery was 1.813 g. (87%). R_G values were measured in butanol-ethanol-water (4:1:5), and the chromatograms sprayed with aniline oxalate.

Fraction 1 (340–410 c.c.). <5 mg., R_G 1.0 gave a yellow-brown spot identical with 2:3:4:6-tetra-*O*-methylglucose run as a control.

Fraction 2 (600–820 c.c.), Crystalline 2:3:4-tri-*O*-methylxylose (57.4 mg.) gave a mauve spot, R_G 0.94, identical with 2:3:4-tri-*O*-methylxylose. M. p. and mixed m. p. (after recrystallisation from light petroleum) 85–87°, $[\alpha]_D +19.8^\circ$ (*c* 1.1 in H₂O) (Found: OMe, 48.2. Calc. for C₈H₁₆O₅: OMe, 48.4%).

Fraction 3 (2.1–2.29 l.). Crystalline 2:4:6-tri-*O*-methylglucose (15.6 mg. (gave a red brown spot, R_G 0.76, identical with an authentic specimen. M. p. and mixed m. p. 126°, $[\alpha]_D^{15} +108^\circ \rightarrow +70^\circ$ (*c* 1.5 in MeOH).

Fraction 3(a) (2.29–2.35 l.) (268 mg.). Chromatography showed this to be a mixture of 2:4:6-tri-*O*-methylglucose and 2:4-di-*O*-methylxylose.

Fraction 4 (2.35–4.35 l.). Crystalline 2:4-di-*O*-methylxylose (1.433 g.) gave a pink spot, R_G 0.66, identical with an authentic specimen. M. p. and mixed m. p. 116°, $[\alpha]_D^{15} -26^\circ \rightarrow +28^\circ$ (*c* 1.0 in H₂O; 24 hr. constant) (Found: C, 47.44; H, 7.77; OMe, 34.3. Calc. for C₇H₁₄O₅: C, 47.20; H, 7.86; OMe, 34.8%). It gave an X-ray powder photograph identical with that given by authentic material and different from the photograph of 2:3-di-*O*-methylxylose.

Fraction 5 (8.3–9.2 l.). Syrupy 4:6-di-*O*-methylglucose (8.0 mg., contaminated with wax) gave a brown spot, R_G 0.46, when sprayed with *p*-anisidine hydrochloride, as did authentic material, whereas 2:4-di-*O*-methylglucose gave a yellow spot (paper chromatography). Its identity was confirmed by ionophoresis.²⁰ The M_G values of the different dimethylglucoses are: 2:4-di-*O*-methyl, 0.09; 2:6-di-*O*-methyl, 0.05; 3:4-di-*O*-methyl, 0.35; 4:6-di-*O*-methyl, 0.2; 3:6-di-*O*-methyl, 0.65; 2:3-di-*O*-methyl, 0.10.

Fraction 6 (10.72–11.0 l.). Syrupy monomethylxylose (31.5 mg.) had $[\alpha]_D^{15} +19^\circ$ (*c* 3.1 in H₂O) and gave a pink spot, R_G 0.40, with aniline oxalate (Found: OMe, 17.4. Calc. for C₆H₁₂O₅: OMe, 18.9%). Paper chromatography in butan-1-ol-pyridine-water (10:3:3) gave two spots, R_{Rhamnose} 1.19 and 1.10, corresponding to authentic 2- and 4-*O*-methylxylose respectively. Spraying with *p*-anisidine hydrochloride gave a mauve spot with the 2-*O*-methyl

¹⁹ Hough, Powell, and Woods, *J.*, 1956, 4799.

²⁰ Foster, *Chem. and Ind.*, 1952, 1050.

sugar and a reddish-brown spot for the 4-*O*-methyl sugar. Ionophoresis gave two distinct spots, M_G 0.40 for 2-*O*-methylxylose and 0.25 for the 4-*O*-methylxylose.

The water-washings were devoid of carbohydrate material.

Acetylation of Xylan (B).—Xylan (2.4 g.) was acetylated as for esparto grass xylan⁵ except that the pyridine solution was cooled to 0° before addition of acetic anhydride and the product, after thorough washing with water, was isolated by freeze-drying of its suspension in water. A second acetylation gave a white powder (2.75 g.) which was insoluble in methanol, ethanol, acetone, or chloroform, and had $[\alpha]_D -51^\circ$ (c 0.4 in *m*-cresol) (Found: Ac, 39.5. Calc. for $C_9H_{12}O_6$: Ac, 39.8%).

Viscosity Determinations of Xylan and its Derivatives.—The viscosities of xylan in 0.5*N*-sodium hydroxide and its derivatives in *m*-cresol were measured in Ostwald viscometers Nos. 1 and 2 respectively at 20°. The results are tabulated.

	c	Average time of flow (sec.)		η_{sp}/c *
		Solution	Solvent	
Xylan (B)	0.0435	287.8	237.3	4.89
Xylan (C)	0.0381	283.3	237.3	5.01
Acetylated xylan	0.0191	319.4	238.0	17.96
Methylated xylan	0.0316	345.4	238.0	14.29

* c represents concentration of $C_6H_8O_4$ units.

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