Notes

Column chromatography of polysaccharides in the presence of urea

The separation of polysaccharides by ion exchange chromatography has been used successfully in certain cases¹ but has lacked any general applicability. This has been because ionic interactions are largely obscured by molecular sieving effects and by association due to hydrogen bonding. A recent paper² has reported that these secondary binding forces may be eliminated by the addition of urea to the eluting system. We now wish to report that the use of such urea solutions offers considerable promise in the chromatographic separation of polysaccharides.

Black spruce (*Picea mariana*) is known to contain 3-O-methyl-L-rhamnose³ in small amounts and, in common with other coniferous woods, alkaline extraction of a chlorite holocellulose yields a mixture of polysaccharides containing predominantly arabinomethoxyglucuronoxylans and galactoglucomannans. Attempted resolutions of such a mixture on a DEAE-cellulose column (OH⁻ form) using water and aqueous ammonium acetate (2N) did not result in any clear-cut separation but gave approximately equal amounts of the xylan and mannan polymers in the aqueous eluate. When the eluting solvents used were 7 M urea and ammonium acetate in 7 M urea the fraction eluted with 7 M urea was free of xylose and on hydrolysis gave a galactose:glucose:mannose:3-O-methyl-rhamnose ratio of 1.0:2.2:2.5:3.8. The remaining fractions eluted with 2 N ammonium acetate in 7 M urea contained mainly xylose polymers. The 3-O-methyl-rhamnose was readily identified by its chromatographic behaviour³ in ethyl acetate-acetic acid-formic acid-water (18:3:1:4) and ethyl acetate-pyridine-water (9:2:2).

These results illustrate the application of this method to polysaccharides and show that the elution pattern is markedly changed in the presence of urea. They also show that the 3-O-methyl-rhamnose is associated with a mannan rather than a xylan polymer. It is likely that the polysaccharide containing the 3-O-methyl-rhamnose has a low degree of polymerization resulting in its separation from the other polymers². It is hoped to study this point later.

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