

Basics of Carbohydrate analysis by high performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD) of wood/fibers

Sample preparation of pulp and acid hydrolysis for carbohydrate analysis is based on method described in Tappi T-249.¹ The monomeric sugar content of the hydrolysed pulp was determined by HPAEC-PAD analysis (Figure 1).² Deionized water was used in all of the steps.

The air-dried pulp was ground to pass through a 40 mesh screen. 0.1750 g of each ground sample was weighed into a 50 ml tube. Standard sugars were also weighed in a 50.00 ml tube in order to put them through the same hydrolysis procedures as described for the pulp samples. The typical weights of standard sugars are presented in Table 1.



Figure 1. Photograph of high performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD).

Table 1. Typical weights of standard sugars for carbohydrate content analysis of pulp.

Standard sugar	Weight (g)
Glucose	0.1800
Xylose	0.0360
Arabinose	0.0100
Mannose	0.0200
Galactose	0.0100

¹ Tappi test methods. (1992-1993). T-249. Carbohydrate composition of extractive-free wood and wood pulp by gas-liquid chromatography.

² Davis, M. W. A rapid modified method for compositional carbohydrate analysis of lignocellulosics by high pH anion exchange chromatography with pulsed amperometric detection (HPAEC/PAD). *J. of Wood Chem. Technol.* 1998, 18(2), 235-252.

1.50 ml of 72% sulfuric acid solution was added to each sample. The sample was stirred with a glass rod until wet. The glass rod was left in the sample and stirred occasionally throughout the primary hydrolysis process. The tube was placed into the Digibloc set at 30.0 °C. After 1 hour, the sample was diluted with 42.00 ml water. A watch glass was placed on the top of the tube and put into the autoclave for 1 hour on the 121°C setting. This was the secondary hydrolysis step. The solution was then diluted to 50.00 ml with water and filtered through glass filters. As for the pulp sample, 1.00 ml of the solution was pipetted and transferred to a 25.00 ml volumetric flask. Also, 1.00 ml of 1,00 mg/ml fucose was added and additional water was added to the volumetric flask. 5 standard solutions were prepared by the same procedure. Typical dilutions were composed of 1.00, 0.75, 0.50, 0.25, and 0.10 ml aliquots of stock each and 1.00 ml of 1.00 mg/ml fucose stock brought up to 25.00 ml. The diluted solution was transferred to a 0.50 ml vial and placed in the auto sampler for carbohydrate content analysis by HPAEC. The output screen of the carbohydrate content for a sample is shown in Figure 2. The results of sugar content ($\mu\text{g/ml}$) could be obtained at the same time and for each sample, it was conducted in duplicate and the error was within $\pm 2\%$ of the average value.

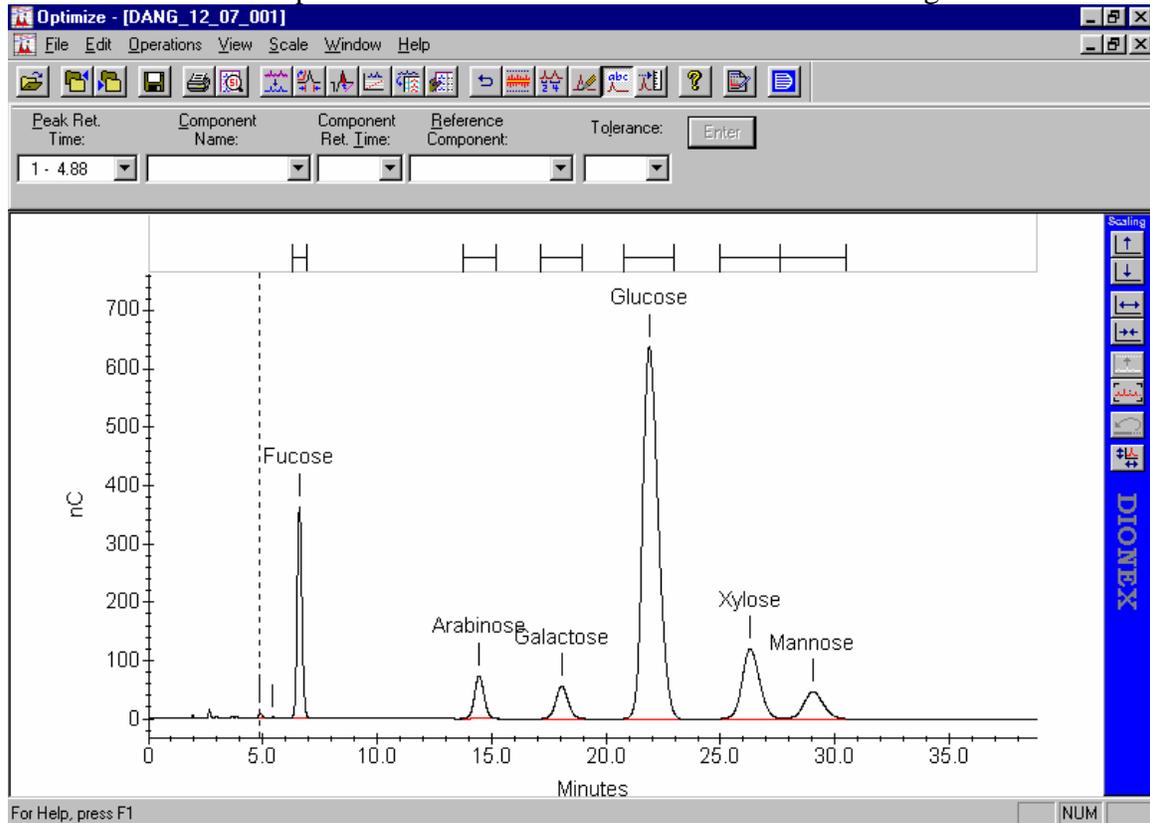


Figure 2. The output screen of HPAEC for carbohydrate content analysis of pulp.