Alpha, Beta, and Gamma Cellulose.

The determination of alpha, beta, and gamma cellulose* in cellulose obtained from wood by the chlorination method or directly on pulps, is a measure of the resistance of the cellulose or pulp to the action of 17·5-per-cent. sodium-hydroxide solution, known as Mercer's solution. In cases where the alpha-cellulose content of a cellulose or pulp alone is desired, or when the alpha cellulose is not difficult to filter and wash, the gravimetric method may be used. However, when data on all three constituents is desired the work may be facilitated by using the volumetric method of Bray and Andrews.†

Gravimetric Method for Alpha Cellulose.

One gramme of oven-dry cellulose obtained by the above method, or of the sample of pulp, is weighed into a beaker, triturated with 25 c.c. of mercerizing liquid (17·5-per-cent. sodium-hydroxide solution)‡ until the mass is homogeneous, and allowed to stand for thirty minutes. The contents of the beaker are filtered by suction through either a tared alundum crucible (porosity R.A. 98), a Gooch crucible, or a fritted-glass Jena crucible (No. 3, porosity 5 to 7). After the insoluble cellulose or alpha cellulose is sucked practically dry, it is loosened with a glass rod, washed with 4-per-cent. sodium-hydroxide solution (50 c.c.), and then with cold distilled water (approximately 300 c.c.). The alpha cellulose is next treated with approximately 75 c.c., hot dilute (10 per cent.) acetic acid, again washed with hot distilled water (300 c.c.), dried at 105° C., cooled in a desiccator, and weighed as alpha cellulose.§

Lignin.

The lignin,‖ or non-carbohydrate content of wood-substance or pulp, is obtained by a modification of the method of Ost and Wilkening¶ employed in the hydrolysis of cellulose.

Approximately 2 grm. of air-dry wood (sawdust passed through a 60- and held on an 80-mesh wire sieve), or shredded pulp, are weighed into a tared alundum crucible (porosity R.A. 98). The crucible is dried in the oven for two hours at 105° C., cooled, and weighed in a stoppered weighing-bottle. It is then extracted with ether in a Soxhlet apparatus for three to four hours as described under the ether-soluble determination. In the case of pulps the extraction operation may be dispensed with, and the samples may be weighed and dried in weighing-bottles directly. The dried material is transferred from the crucibles to weighing-bottles, divided into fine particles, and treated with 40 c.c. of 72-per-cent.** ($\pm$ 0·1 per cent.) sulphuric acid by weight, the strength of which has been previously determined by titration with a standard sodium-hydroxide solution. The hydrolysis is allowed to proceed for sixteen hours at room-temperature, with frequent stirring at the beginning of the operation. The resulting solution is transferred to a 2-litre Erlenmeyer flask and diluted to 1,570 c.c. with distilled water which makes a concentration of H_2SO_4 exactly 3 per cent., covered with a watch-glass, and boiled for two hours, adding distilled water from time to time to keep the volume constant. The suspended particles of lignin are then filtered on the tared alundum crucible used in the beginning of the determination, washed thoroughly with hot distilled water (500 c.c.), dried for two hours and a half at 105° C., cooled in a desiccator, and weighed as lignin.

Ether-soluble Material.

This determination is a measure of the waxes, fats, resins, &c. Approximately 2 grm. of the sawdust or shredded pulp are weighed in a tared alundum crucible (porosity R.A. 98) and stoppered weighing-bottle. The crucible containing the material is placed alongside the bottle and heated for two hours or to constant weight at 105° C. The crucible is placed in the stoppered bottle, cooled in a desiccator over concentrated H_2SO_4, and weighed as oven-dry material. The crucible is placed in a Soxhlet apparatus, and the sawdust is extracted with ethyl ether for three to four hours, dried at 105° C., cooled, and weighed. The extracted sawdust or pulp may be used for the lignin determination. The amount of material extracted may be determined either by weighing the residue after evaporation of the solvent, or by determining the loss in weight of sawdust due to extraction. The latter procedure was followed in these tests.

Alcohol-benzene - soluble Material.

In addition to the substances removed by ethyl ether, possibly some of the so-called wood-gums are dissolved by extraction with a mixture of 67 per cent. benzene and 33 per cent. alcohol. The

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* Cross and Bevan, "Researches on Cellulose," III (1905–10), 23; "Papermaking" (1916), 97. Schwalbe, "Chemie der Cellulose" (1911), 637; Jentjen Zeit Kunstoffe, 1, 165 (1911); Mag. Jahr., 57, 426 (1911); Zts. Aug. Chem., 24, 1341 (1911). Opfermann, "Die Chemische Untersuchung Pflanzliche Rohstoffe und der daraus abschiedenen Zellstoffe," translated by C. J. West), Paper 8, 19 (1921). Bronnert, Die Chem. Under ect. Berlin (1920). Schwalbe and Sieber, "Die Chemische Betriebs-Kintrolle in der Zellstoffe and Papier Industrie," 151. Bray and Andrews, J. Ind. & Eng. Chem., 15, No. 4, 377 (April, 1923); Cellulose Chemie 4, 115 (1923); Zelstoff und Papier, 3, 5, 109 (May, 1923); Papermakers' Monthly J., London, 61, 6 (1923). Sherrard and Blanco, J. Ind. & Eng. Chem., 14, 64 (1922). Th. Beutzen, Paper Trade J. 83, No. 22, 48 (1926).

† Bray and Andrews, *loc. cit.*

‡ Add 825 c.c. of distilled water to 175 grm. of C.P. sodium hydroxide; cool and titrate a sample with normal sulphuric acid to methyl orange and phenolphthalein end points. In this manner the solution may be adjusted so that the actual NaOH content is 17·5 per cent.

§ In all cases the crucibles were placed in stoppered weighing-bottles when cooled in a desiccator containing H_2SO_4 or when weighed.

‖ Klason, Paper Industry, 4, 262 (May, 1922); *ibid.*

¶ Cross and Bevan, "Researches on Cellulose," III, 39 (1905–10); Chemiker Zeit., 461 (1910).

** Becker, Papier-fabr., xvii, 1325. P. Klason, Svenk. Pap. Tid., 129 (1916).