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Acid-insoluble lignin in wood and pulp

1. Scope

1.1 This method describes a procedure which can be applied to the determination of acid-insoluble lignin in wood and in all grades of unbleached pulps. In semi-bleached pulp the lignin content should not be less than about 1% to provide a sufficient amount of lignin, about 20 mg, for an accurate weighing. The method is not applicable to bleached pulps containing only small amounts of lignin.

1.2 Some of the lignin dissolves in acid solution during the test and is not included in the test result. In softwoods (coniferous woods) and in sulfate pulps, the amount of soluble lignin is small, about 0.2 to 0.5%. In hardwoods (deciduous woods), non-wood fibers, and in sulfite pulps, the content of soluble lignin is about 3 to 5%. In semi-bleached pulps, soluble lignin could amount to about one-half or more of the total lignin content.

NOTE 1: The acid-soluble lignin can be determined in a solution, after filtering off the insoluble lignin, by a spectrophotometric method based on absorption of ultraviolet radiation. The most often used wavelength is 205 nm (1).

1.3 The total lignin content in pulps can be estimated fairly closely by rapid, indirect methods based on chlorination of the lignin (TAPPI T 253 “Hypo Number of Pulp” - method withdrawn in 1998) or oxidation of the lignin (TAPPI T 236 “Kappa Number of Pulp”).

2. Summary of method

The carbohydrates in wood and pulp are hydrolyzed and solubilized by sulfuric acid; the acid-insoluble lignin is filtered off, dried, and weighed.

3. Significance

Wood contains from about 20 to 30% lignin, removal of which is a main objective of pulping and bleaching processes. Determination of lignin content in wood and pulps provides information for evaluation and application of the

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processes. Hardness, bleachability, and other pulp properties, such as color, are also associated with the lignin content.

4. Definitions

4.1 Lignin represents what is called the “incrusting material” forming a part of the cell wall and middle lamella in wood. It is an aromatic, amorphous substance containing phenolic methoxyl, hydroxyl, and other constituent groups; its chemical structure has not been fully elucidated.

4.2 In this method of determination, lignin (also known as “Klason lignin”) is defined as a wood or pulp constituent insoluble in 72% sulfuric acid.

5. Apparatus

5.1 *Filtration apparatus* (Fig. 1), consisting of a filtering flask, 2000 mL, a filtering crucible, about 30 mL, an adapter, and a siphon tube. Other types of filtration apparatus may also be used.

NOTE 2: Various types of filtering crucibles can be used, provided that the filtration is reasonably fast and all of the lignin is retained on the filter, resulting in a clear filtrate. Glass filtering crucibles with a sintered glass disc of a fine (*F*), or medium (*M*) porosity can be used on wood and on most of the pulps. Lignin in low-yield sulfite pulps forms a fine dispersion, which often clogs the pores of the sintered glass discs and slows the filtration. A disc of a glass fiber paper, fitted in the crucible, facilitates the filtration. Alundum or porous porcelain crucibles, with a mat of glass fibers, may also be used.

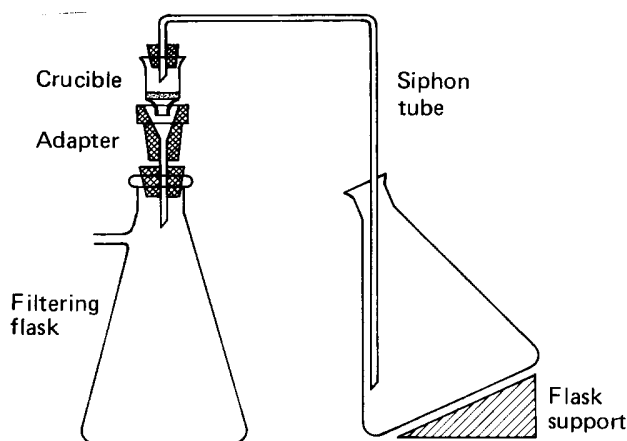


Fig. 1. Lignin filtration apparatus.

5.1.1 Dry the filtering crucibles in an oven at $105 \pm 3^\circ\text{C}$ for about 2 h, cool, and weigh before use.

5.2 *Constant temperature bath*, to maintain a temperature of $20 \pm 1^\circ\text{C}$.

5.3 *Flasks*, Erlenmeyer, 1000 mL, with a mark added at 575 mL volume (for wood specimens; and 2000 mL, with a mark added at 1540 mL volume (for pulp specimens).

5.4 *Reflux condenser* (optional), to be attached to the flask. If used, flasks and condenser should be equipped with ground glass connectors. If ground glass connectors are not available, a rubber stopper may be used.

5.5 *Drying oven*, forced circulation type, maintained at $105 \pm 3^\circ\text{C}$.

5.6 *Hot plate*, electric.

5.7 *Wiley mill*, with a 10 or 20-mesh screen, or a Waring-type blender.

5.8 *Other glassware*: buret, 50 mL; beakers, 100 mL; glass stirring rods.

6. Reagents

6.1 *Sulfuric acid*, 72% H_2SO_4 solution, $24 \pm 0.1N$, sp gr 1.6338 at $20^\circ/4^\circ\text{C}$, prepared as follows:

6.1.1 Carefully pour 665 mL of concentrated H_2SO_4 (95.5 to 96.5%, sp gr 1.84) into 300 mL of water, and after cooling, make up to 1000 mL. Adjust the strength to $24 \pm 0.1N$ by titration with a standard alkali, or by measuring specific gravity. A variation of 0.1% in the strength of acid at this concentration causes a change of 0.0012 in specific gravity.