

estimation in wood and wood-pulps, it is possible to determine the amount of chlorine required for the isolation of the cellulose. The data thus obtained, especially in the case of pulps, furnishes a measure of their relative bleachability.

Method of Estimation.

Approximately 2 grammes of air-dry wood or pulp are weighed in a tared alundum crucible, placed in a weighing-bottle, and dried in the oven for two hours and a half at 105° C. The crucible is placed alongside the bottle during drying, after which it is again placed in the bottle, stoppered, cooled in a desiccator, and reweighed to obtain the weight of oven-dry material.

In the case of wood the crucible, with contents, are extracted three to four hours in a Soxhlet extractor with a mixture of 67 per cent. of benzene and 33 per cent. alcohol. This extraction may be dispensed with in the case of pulps. After evaporating the solvent the sawdust is thoroughly washed with hot water, using the suction-pump. The moist material is then transferred to a Jena glass crucible (size 35 c.c., No. 3, porosity 5 to 7), which is equipped with a fritted-glass bottom. Suction is applied first at the bottom of the crucible until the excess moisture is removed, and then at the top. This removes the water from the fritted-glass plate and evenly distributes the remaining moisture through the entire sample. The crucible is water-jacketed and connected between the gas burette and Hemphel gas-pipette of the Roe* apparatus by means of two rubber stoppers, through which passes a capillary glass tubing. A measured quantity of chlorine gas is passed from the burette up through the material in the crucible and over into the Hemphel gas-pipette as fast as possible. During the first chlorination samples of wood absorb approximately 230 c.c. of the chlorine gas at room-temperature and atmospheric pressure; this necessitates refilling the burette, which may be done quickly, as it is connected to a chlorine-tank with a three-way stop-cock. The first chlorination treatment requires from three to four minutes, after which the crucible is removed from the apparatus and the material washed with approximately 50 c.c. of distilled water, and successively with 50 c.c. of approximately 3-per-cent. SO₂ water, 50 c.c. water, then 50 c.c. of freshly prepared 2-per-cent. normal sodium-sulphite solution. The material is transferred to a 250 c.c. Pyrex beaker, using a pointed glass rod, the last traces of material being washed out of the crucible with 100 c.c. of a 2-per-cent. sodium-sulphite solution. The beaker is covered with a watch-glass and placed in a boiling-water bath for thirty minutes. The fibres are again transferred to the glass crucible and washed with about 250 c.c. of hot distilled water. The above procedure is never sufficient to remove all the lignin, so that the treatment with chlorine and subsequent treatments as outlined above are repeated until the fibres are practically a uniform white or show only a very faint tinge of pink upon addition of the sodium-sulphite solution. The second and following treatments with chlorine should not require more than two or three minutes.†

After all the lignin has been removed the fibrous residue is thoroughly washed in an alundum crucible (porosity 98) successively with hot water, dilute acetic acid, 500 c.c. hot water, 50 c.c. of 95-per-cent. alcohol, and finally with 50 c.c. of ether. It is then dried for two hours at 105° C., again placed in the original stoppered weighing-bottle, cooled in a desiccator, and weighed as cellulose.

CHLORINE-CONSUMPTION.

This value is a measure of the total chlorine gas required in the isolation of the cellulose, and in the case of pulps indicates their relative bleachabilities. The percentage of chlorine consumed is obtained by summing up all the values of the several chlorination treatments after correcting the results to 0° C. and 760 mm. pressure, taking into consideration the vapour-pressure of chlorine gas at the chlorination temperature over calcium-chloride‡ solution saturated with chlorine gas, using the following formula:—

$$V_0 = \frac{V_t (P - a)}{760 (1 + 0.00367t)}$$

where V_0 = volume under standard conditions of temperature and pressure, a = vapour-pressure of chlorine gas over calcium-chloride solution saturated with chlorine gas, and t is the temperature of chlorination.

$t^{\circ}\text{C.}$	A.	$t^{\circ}\text{C.}$	760 (1 + 0.00367 t).
24.5	17.3	24.5	828.36
25	18	25	829.76
26	19.4	26	832.57
27	20.8	27	835.40
28	22]	28	838.12
29	23.5	29	840.96
30	25	30	843.66
31	26.4	31	846.48
32	29	32	849.30
		33	852.04
		34	854.90
		35	857.67

The weight of 1 c.c. of chlorine gas at standard temperature and pressure = 0.0031675 gm.

$$\text{Percentage chlorine consumed} = \frac{V_0 \times 0.0031675 \times 100}{\text{Oven-dry weight of sample.}}$$

* R. B. Roe, *loc. cit.* The Roe apparatus consists of a water-jacketed gas-burette, a chlorination-chamber, and a Hemphel gas-pipette used as a reservoir.

† Prolonged-action chlorine gas, together with the hydrochloric acid formed in the secondary reactions, hydrolyzes the cellulose, gives low yields, and causes varying amounts of alpha cellulose.—Paper Trade J., 76, No. 8, 47 (22nd February, 1923).

‡ Calcium-chloride solution for gas-burette and Hemphel gas-pipette: Saturate a solution with calcium chloride at room temperature and bubble chlorine gas in until saturated and filter for use.