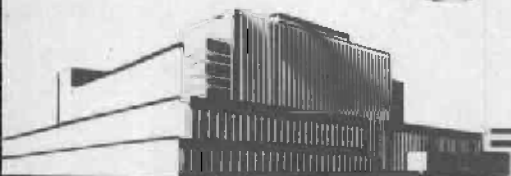


A COMPARISON OF TWO METHODS FOR DETERMINING THE SPECIFIC GRAVITY OF SMALL SAMPLES OF SECOND-GROWTH DOUGLAS-FIR

Report No. 2033

September 1955



FOREST PRODUCTS LABORATORY
MADISON 5, WISCONSIN

UNITED STATES DEPARTMENT OF AGRICULTURE
FOREST SERVICE

In Cooperation with the University of Wisconsin

A COMPARISON OF TWO METHODS FOR DETERMINING THE SPECIFIC
GRAVITY OF SMALL SAMPLES OF SECOND-GROWTH DOUGLAS-FIR¹

By

DIANA M. SMITH, Technologist

Forest Products Laboratory,² Forest Service
U. S. Department of Agriculture

Summary

A comparison between the maximum-moisture and water-immersion methods of determining the specific gravity of small wood samples showed an average difference of less than 1 percent of the mean water-immersion specific gravity. The maximum-moisture method assumes a constant for the density of cell wall substance, but the nature of the formula used is such that reasonable latitude in the assumed value for this cell wall constant contributes little error to the determination of the specific gravity of small wood samples. From the standpoint of technique, the maximum-moisture method is definitely superior, since it eliminates the determination of the weight of the sample in water. Because of its simplicity, the maximum-moisture method is well suited to large-scale growth-quality studies.

Introduction

The importance of specific gravity as an indication of wood quality is well known (4).² Precise methods of determining the specific gravity of small wood samples, such as increment cores, are essential in growth-quality studies of forest trees when it is not feasible to cut the trees. In recent years, therefore, considerable interest has been aroused in methods of determining the specific gravity of exceedingly small wood samples.

¹This report constituted part of a thesis submitted in partial fulfillment of the requirements for the degree of Master of Science at the State University of New York College of Forestry, Syracuse, N. Y.

²Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

³Underlined numbers in parentheses refer to the literature cited at the end of this report.

Specific gravity, as measured for growth-quality studies, is usually based on green volume, and is defined as the ratio of the weight of the oven-dry sample to the weight of a volume of water equal to the volume of the green sample. Methods of specific gravity determination that will give the necessary accuracy on this basis are limited. The Jolly balance method described by Paul and Baudendistel (5) for increment cores gives an approximate value only. Vintila (12) described a technique for obtaining the specific gravity of small samples ranging in volume from 200 to 1,000 cubic millimeters by the standard water-immersion method. This method requires the determination of three weights for each sample, from which the specific gravity is calculated: 1, the soaked weight in air; 2, the weight when held submerged in water; and 3, the oven-dry weight. Keylwerth (2) developed a maximum-moisture technique, based on the relationship between the specific gravity and the maximum moisture content of wood, that compared favorably with duplicate determinations using the technique described by Vintila when applied to Douglas-fir samples 1,000 to 8,000 cubic millimeters in volume, and containing several growth rings. The latter technique has the advantage of requiring only two determinations: the weight of the completely water-saturated sample, and its oven-dry weight. From these two weights, together with a constant that represents the density of cell wall substance, the specific gravity is calculated on the green volume basis.

It is known that for coniferous woods such as Douglas-fir, which have distinct springwood and summerwood zones in the annual rings, the chemical composition of the wood substance in the two zones differs with respect to the proportion of cellulose to lignin, and also with respect to extraneous substances, such as pitch and extractives (1, 6). It has been questioned whether, in critical work, the assumption of a constant for the specific gravity of cell wall substance is justifiable and, if so, to what extent the assumption affects the results.

The work here reported was done to compare the specific gravity values obtained for the same samples of Douglas-fir by the water-immersion and maximum-moisture methods. The samples consisted of springwood, summerwood, or whole annual rings, and ranged in volume from 110 to 1,600 cubic millimeters.

Material and Procedures

The 96 individual annual rings sampled in this study were selected at random from strength test specimens of second-growth Douglas-fir (Pseudotsuga menziesii (Mirb.) Franco). The wood was taken from 16 log sections collected from four mill ponds in the States of Washington and Oregon for research on the growth-strength relations of wide-ringed Douglas-fir (8). The width of the selected annual rings ranged from 0.094 to 0.471 inch, averaging 0.261 inch, and the percentage of summerwood in the annual rings ranged from 15.9 to 62.2 percent, averaging 33.1 percent.

Preparation of Test Specimens

The random rings were first isolated and then divided into three parts, A, B, and C, each measuring 1/2 inch in both the tangential and longitudinal directions (fig. 1). These subdivisions of the ring were dissected and used separately for determining the specific gravity of the complete ring, and of the springwood and summerwood. Sample B was used to determine the specific gravity, percentage of summerwood, and ring width of the complete annual ring. Since both springwood and summerwood could not be cut from the same piece without some loss, only springwood was taken from sample A and only summerwood from sample C. For example, to secure the summerwood portion of a particular ring, the springwood zones of the selected ring and of the subsequent ring were split off in the tangential planes, so that part of the springwood was left on each side of the summerwood. The remaining springwood was then carefully removed with a scalpel. Owing to the difficulties in visually establishing accurate boundaries between springwood and summerwood, the boundary was first determined under the microscope using Mork's definition (3) which reads:

"All tracheids in which the common wall between two cell cavities multiplied by 2 is equal to or greater than the width of the lumen are considered as summerwood; those in which this value is less than the width of a lumen are considered as springwood (all measurements being made in the radial direction)."

After dissection, the samples were examined under the microscope to make sure that only the designated portion of the ring was included.

A total of 96 samples of springwood and 96 samples of summerwood were obtained, which corresponded with the 96 samples comprising a section of 1 complete annual ring, giving a total of 288 samples that ranged in volume from 110 to 1,600 cubic millimeters.

The samples were placed in distilled water at room temperature in a vacuum flask, and a vacuum was applied intermittently until they had absorbed water to a constant weight. In practice, this period was found to be 7 to 10 days. To insure complete saturation, a vacuum was applied intermittently over a period of 15 days.

To obtain the soaked and oven-dry weights of the samples, an automatic balance (fig. 2) reading to 0.01 milligram was used under controlled temperature and humidity. Weighing bottles were used to avoid loss or gain in moisture, which could contribute considerable error to the green or oven-dry weight of the samples.

Determination of Specific Gravity
by the Water-Immersion Method

The green volume of the samples was determined by Vintila's method (12). The saturated wood samples in water and the weighing bottles were allowed to reach equilibrium temperature under controlled conditions. To obtain the soaked weight of a sample in air, the weight of the weighing bottle was first recorded. The saturated sample, from which the surface water had been removed with a damp cloth, was placed in the bottle and the total weight recorded. The difference between the weight of the weighing bottle and that of the bottle and sample gave the weight of the saturated wood sample in air. To prevent the sample from drying out, it was reimmersed in distilled water until the next determination was made.

A bridge was then placed over the single pan of the balance to support a beaker of distilled water containing two drops of a wetting agent.⁴ This bridge eliminated the beaker and water from consideration in the weighings. A piece of fine wire 0.01 inch in diameter with a weight and a short needle point at one end was suspended from the beam with the weight and needle point submerged in the water (fig. 3).

When an automatic balance is used, weights up to 10 milligrams are determined from the beam deflection. Consequently, samples of different weights are not ordinarily immersed to a uniform depth. Provision had to be made, therefore, for raising or lowering the water level in the container to insure uniform depth of immersion. A water-level indicator, consisting of a piece of fine, rigid wire with a sharp point, was attached to the weight as shown in figure 2. An eye dropper was used to adjust the water level in the beaker so that the indicator point barely touched the surface of the water, as seen from the reflection at the surface of the water through the glass sides of the beaker. The weight of the hydrostatic apparatus was recorded. The sample was then placed on the needle point and lowered into the glass beaker, and the water level again was adjusted until the indicator point barely touched the surface of the water. The weight of the hydrostatic apparatus with the sample was recorded, and the difference in the two weights gave the weight in grams of the soaked sample in water. Since 1 gram is the weight of 1 cubic centimeter of water, the volume (V_F) of the sample in cubic centimeters was obtained from the difference in — grams of its weight in air and its weight when held submerged in water.

To obtain their oven-dry weight, the samples were dried in a vacuum oven for approximately 18 hours at 60° C. and a vacuum of 25 inches of mercury so that an essentially constant weight was obtained. They were then transferred rapidly to a desiccator containing calcium chloride, and allowed to reach equilibrium in a temperature-controlled room before the oven-dry weight (m_o) was determined.

⁴—1.0 gram of Nacconol NR (National Analine Division, Allied Chemical and Dye Corporation, New York 6, N. Y.) in 100 cubic centimeters of distilled water.

The specific gravity on the basis of green volume was calculated from the formula:

$$G_f = \frac{m_o}{m_m - m_w} = \frac{m_o}{V_f} \quad (1)$$

where G_f is the specific gravity based on green volume, m_o is the oven-dry weight of the sample in grams, m_m is the saturated weight of the sample in grams, m_w is the weight in grams of the saturated sample when held submerged in water, and V_f is the weight in grams of a volume of water equal to the volume of the sample obtained by the above method.

Determination of Specific Gravity by the Maximum-Moisture Method

The maximum-moisture method for obtaining specific gravity on the basis of soaked volume is extremely simple and straightforward, and requires only two determinations: the weight of the completely water-saturated wood sample and its oven-dry weight. These weights were determined previously in the method for obtaining the specific gravity by water immersion. The specific gravity of the samples (7) was calculated by substitution in the formula:

$$G_f = \frac{1}{\frac{m_m - m_o}{m_o} + \frac{1}{G_{so}}}$$

or

(2)

$$G_f = \frac{1}{\frac{M_{max}}{G_{so}} + \frac{1}{G_{so}}}$$

where m_m is the weight in grams of the completely saturated sample, G_{so} is the specific gravity of dry cell wall substance, and M_{max} is the maximum moisture content in grams of water per gram of oven-dry wood. The average value of 1.53 obtained by Stamm (10) was substituted for G_{so} in the above formula.

Discussion of Results

A comparison was made of the specific gravity values for the same samples obtained by the water-immersion and maximum-moisture methods. There were 96 comparisons of specific gravity of the complete rings, the springwood,

and the summerwood samples. The average specific gravity values obtained by the two methods, together with the standard deviations and the coefficients of variation, are given in table 1. The specific gravity values obtained by the water-immersion method are consistently larger, by a small amount, for each set of 96 comparisons. The differences for individual samples being compared ranged from -0.005 to 0.018, and only 18 negative differences occurred in 288 pairs.

The test used to determine the significance of differences in specific gravity values obtained by the two methods was:

$$t = \frac{\bar{d}}{s_d / \sqrt{n}} \text{ for } n - 1 \text{ degrees of freedom}$$

where \bar{d} is the mean difference between pairs of specific gravity determinations, s_d is the standard deviation calculated from the differences, and n is the number of pairs (9). The t -test showed a highly significant difference at the 1 percent level for each set of comparisons (table 2). The t -test does not give a true picture of this comparison, however, since a consistent difference in specific gravity will yield a significant t -value. Therefore, the specific gravity values for the individual test specimens were plotted with the water-immersion values on the abscissa and the maximum-moisture values on the ordinate (fig. 4). The 45° line in the figure represents the theoretically identical values for the two methods of determining specific gravity. It is evident from figure 4 that the differences in the specific gravity values obtained by the two methods increase with the actual specific gravity of the test specimens. This trend also becomes apparent from a consideration of the mean differences between the specific gravity values obtained by the two methods for the annual ring components. If the mean differences (table 2) are expressed as a percentage of the mean specific gravity by the water-immersion method (table 1), the following percentages are obtained (table 2):

- (1) 0.359 percent for complete annual rings
- (2) 0.153 percent for springwood
- (3) 0.935 percent for summerwood

While a discrepancy between methods of less than 1 percent of the mean is unimportant for all practical purposes, an explanation of the consistency in the differences requires a better understanding of the limitations inherent in the techniques as they are applied in the respective formulas. For this purpose it is necessary to reduce the formulas to a comparable basis.

If G_{max} denotes the specific gravity obtained by the maximum-moisture method, $G_{\text{H}_2\text{O}}$ denotes the specific gravity obtained by the water-immersion method, and the primes denote the experimental values, then from formula (1) page 5:

$$G_{H_2O} = \frac{m_o'}{m_m' - m_w'}$$

$$= \frac{1}{\frac{m_m'}{m_o'} - \frac{m_w'}{m_o'}}$$

But $m_w' = m_w \pm \alpha$, where α is the experimental error associated with m_w , the true saturated weight of the sample when held submerged in water.

$$m_w = m_m - V = m_m - [m_m - m_o + \frac{m_o}{G_{so}}] = m_o - \frac{m_o}{G_{so}}$$

That is, the true volume of the sample, V , is made up of two parts: the volume of water in the sample $= (m_m - m_o)$ and the volume of the dry cell wall substance $= \frac{m_o}{G_{so}}$.

Therefore

$$G_{H_2O} = \frac{1}{\frac{m_m'}{m_o'} - \frac{m_o - \frac{m_o}{G_{so}} \pm \alpha}{m_o'}}$$

(3)

$$G_{H_2O} = \frac{1}{\frac{m_m'}{m_o'} - \frac{m_o}{m_o'} + \frac{m_o}{G_{so}(m_o')} \mp \frac{\alpha}{m_o'}}$$

The formula used for calculating specific gravity by the maximum-moisture method, page 5, was:

$$G_{M_{max}} = \frac{1}{\frac{m_m' - m_o'}{m_o'} + \frac{1}{G_{so}'}}$$

If the assumed value, G_{so}' is in error by an amount β , then

$$\frac{1}{G_{so}'} = \frac{1}{G_{so} \pm \beta} = \frac{1}{G_{so}} \mp \frac{\beta}{G_{so}^2} + \frac{\beta^2}{G_{so}^3} \mp \dots$$

From Stamm's work on the density of cell wall substance (10) it is known that G_{so} is approximately equal to 1.53 and the maximum error involved is approximately ± 0.03 . Values of $\frac{\beta^2}{G_{so}^3}$ and higher powers, therefore, are not expected to contribute any appreciable error to the assumption that

$$\frac{1}{G_{so}'} = \frac{1}{G_{so} \pm \beta} = \frac{1}{G_{so}} \mp \frac{\beta}{G_{so}^2}$$

Therefore

$$G_{M_{max}} = \frac{1}{\frac{m_m}{m_o'} - 1 + \frac{1}{G_{so}} \mp \frac{\beta}{G_{so}^2}} \quad (4)$$

For values of $G_{H_2O} = G_{M_{max}}$ it follows from equations (3) and (4) that

$$-\frac{m_o}{m_o'} + \frac{m_o}{G_{so}(m_o')} \mp \frac{\alpha}{m_o} = -1 + \frac{1}{G_{so}} \mp \frac{\beta}{G_{so}^2} \quad (5)$$

It is now necessary to consider the experimental error involved in the determination of m_o' . Since every precaution was taken to reduce this error to a minimum by drying the samples in a vacuum oven to an essentially constant weight, it seems reasonable to assume that $m_o = m_o'$. Therefore equation (5) reduces to:

$$\mp \frac{\alpha}{m_o} = \mp \frac{\beta}{G_{so}^2}$$

If, experimentally, identical values for specific gravity were obtained by the two methods, and provided $m_o' = m_o$, it would be correct to assume that the error β in the assumed specific gravity of cell wall substance had the following relationship to the error α involved in determining the soaked weight of the sample when held submerged in water:

$$\beta = \frac{G_{so}^2}{m_o'} \alpha$$

The average values of m_o' determined experimentally were 0.16283 gram for springwood, 0.19158 gram for summerwood, and 0.36704 gram for complete annual rings. If G_{so} is approximately equal to 1.53, values of β 6 to 14

times as large as α would be expected if identical specific gravity values were obtained by the two methods. However, identical values for specific gravity by the two methods were not obtained experimentally (table 1), and moreover, the water-immersion values were consistently larger than those obtained by the maximum-moisture method. Therefore, the above relationship did not hold true for the experimental values.

To account for the differences in average specific gravity values obtained by the two methods it was found that if it is assumed that $\beta = 0$, α would have the following average values: 0.00092 gram for springwood, 0.00242 gram for summerwood, and 0.00325 gram for complete annual rings.

These values were calculated as follows:

$$\frac{1}{G_{H_2O}} - \frac{1}{G_{M_{max}}} = \frac{\alpha}{m_o}$$

Conversely, if it is now assumed that $\alpha = 0$, it is possible to estimate β from the average specific gravity values obtained by the water-immersion method (table 1) and the average maximum-moisture values given in table 3. The observed differences in specific gravity would be accounted for by values of β having the following magnitudes: 0.013 for springwood ($G_{so} = 1.543$), 0.030 for summerwood ($G_{so} = 1.560$), and 0.021 for complete annual rings ($G_{so} = 1.551$).

These values were calculated by substitution in the following formula and solving for G_{so} :

$$G_{H_2O} = \frac{1}{M_{max} + \frac{1}{G_{so}}}$$

Then

$$\beta = G_{so} - 1.53$$

The estimated magnitudes of α , based on the assumption that $\beta = 0$, cannot be explained satisfactorily since α includes all possible sources of error in obtaining the soaked weight of the test specimens when held submerged in water, including surface tension on the suspension wire and the possibility of bubbles adhering to the submerged portion of the apparatus and specimens. The magnitude of surface tension, for instance, cannot be estimated since the suspension wire was not chemically clean and a wetting agent was used in the water in which the samples were submerged.

The second assumption, that $\alpha = 0$ and that the differences in specific gravity values obtained by the two methods may be ascribed to the error in

the assumed specific gravity of cell wall substance, is more easily explained since the estimated values of β are in agreement with the known chemical composition of cell wall substance. Trendelenburg (11) states that springwood has more lignin than summerwood, and that "the fiber with a high lignin content has a lower specific weight than the cellulose-rich fiber." This trend is shown in the estimated values for the specific gravity of cell wall substance based on the assumption that $\alpha = 0$. The range of these estimated values -- 1.50 to 1.56 -- lies within the range of Stamm's determinations of the specific gravity of cell wall substance for all species by the water displacement method (10). Accordingly, the specific gravity values obtained by the maximum-moisture method for the 96 samples of the annual ring components were recalculated using the appropriate estimated values for G_{so} . Table 4 gives an analysis of the results that shows that the agreement between the two methods is entirely satisfactory when these new values of G_{so} are used.

The initial problem, whether the assumption of a constant for the specific gravity of cell wall substance is justifiable in calculating specific gravity by the maximum-moisture method, and if so, to what extent the assumption affects the results, can now be answered. A further study of equation (2) shows that since G_f is obtained by taking the reciprocal of a sum, $\frac{M_{max}}{G_{so}}$ plus the reciprocal of the specific gravity of cell wall substance, a small error in the assumed specific gravity of cell wall substance will result in an even smaller error in the calculated value for G_f . This is especially true for springwood and for complete annual rings, where the values for $\frac{M_{max}}{G_{so}}$ are considerably larger than $\frac{1}{G_{so}}$. It was found that when 1.53 was substituted for G_{so} in formula (2), the mean differences in the specific gravity values obtained by the 2 methods were less than 1 percent of the mean specific gravity. Therefore there can be reasonable latitude in the assumed value for the specific gravity of cell wall substance without contributing any appreciable error to the calculated value for specific gravity by the maximum-moisture method.

Summary and Conclusions

Comparisons showed that the maximum-moisture method gave consistently lower specific gravity values than the water-immersion method when the former method was based on the assumption of a constant, 1.53, for the density of cell wall substance. The average difference between the two methods amounted to less than 1 percent of the mean specific gravity, and appeared to increase with the specific gravity of the test specimens.

To investigate the consistency of the discrepancy between methods, the respective formulas were compared by reducing them to similar terms and

examining the possible sources of error. Since the true specific gravity of the samples was not known, the discrepancies in specific gravity values by the two methods were first attributed to the possible errors involved in determining the soaked weight of the submerged samples by the water-immersion method. The errors were then estimated, and although they were consistently positive, they were not consistent in their magnitudes and did not appear to be related to specimen size. When the discrepancies were attributed to the possible errors involved in the assumption of a constant for the specific gravity of cell wall substance, and the errors necessary to produce the observed discrepancies were estimated, they appeared to be related to the known differences in chemical composition of springwood and summerwood and lay within the range of values for the specific gravity of cell wall substance for all woods tested by Stamm. Further work to determine the specific gravity of cell wall substance of the annual ring components by a method such as the picnometric method used by Stamm (10) is indicated if the maximum-moisture method is to be used in critical work where a discrepancy up to 1 percent of the mean water-immersion specific gravity is important.

It should be emphasized, however, that from the practical point of view the maximum-moisture method, when applied to samples ranging in volume from 100 to 1,600 cubic millimeters, is entirely satisfactory in spite of the assumption of a constant for the specific gravity of cell wall substance. The nature of the formula used in the maximum-moisture method is such that reasonable latitude in the assumed value for this cell wall constant will contribute little error to the determination of the specific gravity of small wood samples.

From the standpoint of technique, there is no doubt that the maximum-moisture method is superior to the water-immersion method. It eliminates the need for determining the weight of the sample in water, which is the least accurate and most time-consuming determination involved in the water-immersion method. Because of its simplicity, the maximum-moisture method is well suited to large-scale growth-quality studies where specific gravity is required on the green volume basis. The development of a more rapid means of obtaining water-saturation of the test specimens will still further increase the utility of the method.

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Table 1.--The means, standard deviations, and coefficients of variation of specific gravity determined by two methods for individual annual rings of second-growth Douglas-fir and their springwood and summerwood portions

Statistic	Water-immersion method	Maximum-moisture method
Complete rings		
Number of comparisons	96	96
Mean specific gravity	0.4122	0.4107
Standard deviation	0.06725	0.06690
Coefficient of variation (percent)	16.31	16.29
Springwood		
Number of comparisons	96	96
Mean specific gravity	0.2662	0.2658
Standard deviation	0.02752	0.02745
Coefficient of variation (percent)	10.34	10.33
Summerwood		
Number of comparisons	96	96
Mean specific gravity	0.7423	0.7354
Standard deviation	0.06484	0.06352
Coefficient of variation (percent)	8.74	8.64

Table 2.--Comparison of the water-immersion and the maximum-moisture methods for determining the specific gravity of small second-growth Douglas-fir samples

Portion of growth ring	: Number : of : compar- : isons	: Mean : difference : between : methods	: Variances of : differences : between : methods	: t^1 : values	: Mean difference : as a percentage : of the mean : specific gravity ²
Complete rings	: 96	: 0.0014792	: 0.000001136	: 13.60	: 0.359
Springwood	: 96	: .0004062	: .000001023	: 3.94	: .153
Summerwood	: 96	: .0069375	: .000024859	: 13.63	: .935

¹ $t_{0.01} = 2.63$ for 95 degrees of freedom.

²Mean specific gravity as obtained by the water-immersion method.

Table 3.--Average maximum moisture content¹
of complete rings of second-
growth Douglas-fir, and their
springwood and summerwood portions

Portion of	:	Average
annual ring	:	maximum moisture content
-----		-----
	:	<u>Grams per gram</u>
Complete ring	:	1.7813
Springwood	:	3.1086
Summerwood	:	.7062

¹Average maximum moisture content, \bar{M}_{\max} , expressed in grams of water per gram of oven-dry wood, obtained by substitution in the formula:

$$\bar{M}_{\max} = \frac{1}{\bar{G}_f} - \frac{1}{1.53}$$

where \bar{G}_f is the average of 96 specific gravity values obtained by the maximum-moisture method using $G_{s0} = 1.53$.

Table 4.--Comparison of the differences between adjusted¹ specific gravity values obtained by the maximum-moisture method with duplicate values obtained by the water-immersion method

Portion of growth ring	Number of comparisons	Mean difference between methods	Variances of differences between methods	t ² values	Mean difference as a percentage of the mean specific gravity ²
Complete rings	96	-0.0000333	0.00000092	-0.340	0.008
Springwood	96	.0000281	.00000069	.332	.011
Summerwood	96	-.0002073	.00001808	-.478	.028

¹Specific gravity values adjusted according to the estimated specific gravity of cell wall substance: 1.543, 1.560, and 1.551, for springwood, summerwood, and complete annual rings, respectively.

²t_{0.05} = 1.986 for 95 degrees of freedom.

³Mean specific gravity as obtained by the water-immersion method.

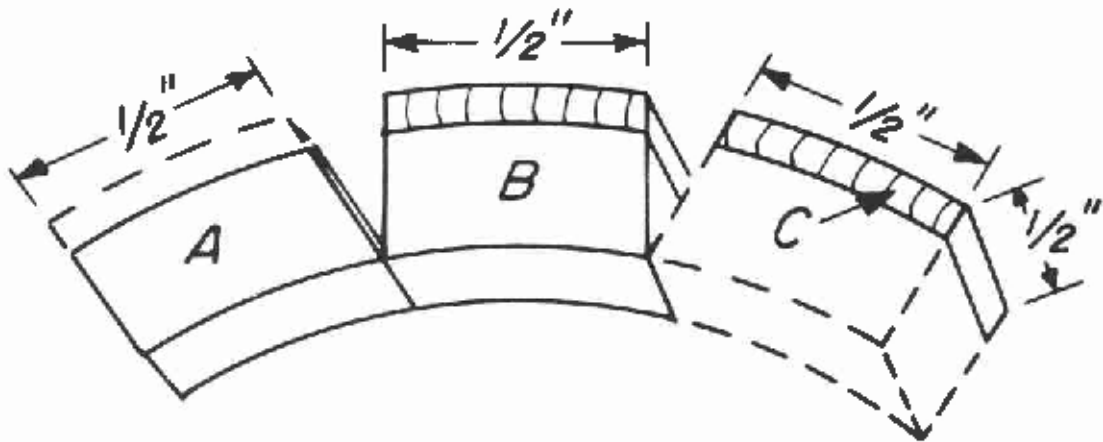


Figure 1.--Isolated growth ring separated into three specific gravity samples: A, springwood; B, complete annual ring; and C, summerwood.

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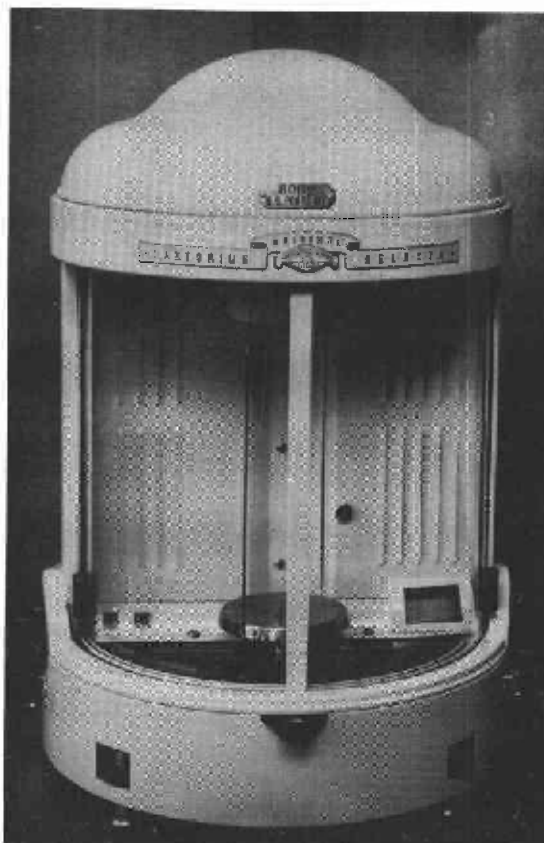
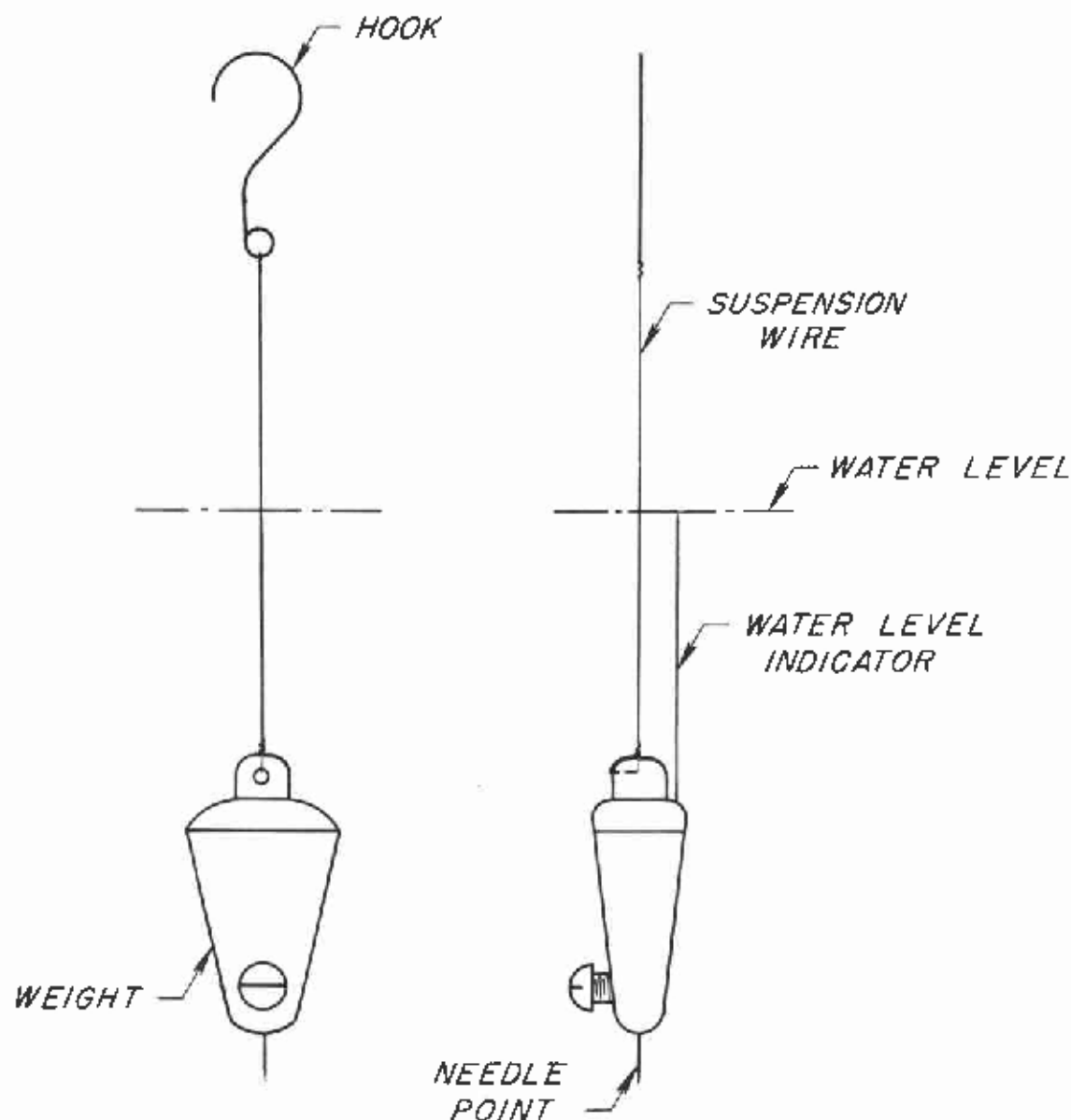


Figure 2. --Automatic balance used to obtain soaked and oven-dry weights of the samples.

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Figure 3. --Hydrostatic apparatus for obtaining weight of the sample in water. The apparatus, which is suspended from the beam of the balance, consists of a weight with a needle point to which the sample is attached. A water-level indicator, consisting of a fine rigid wire with needle point, is attached to the weight.

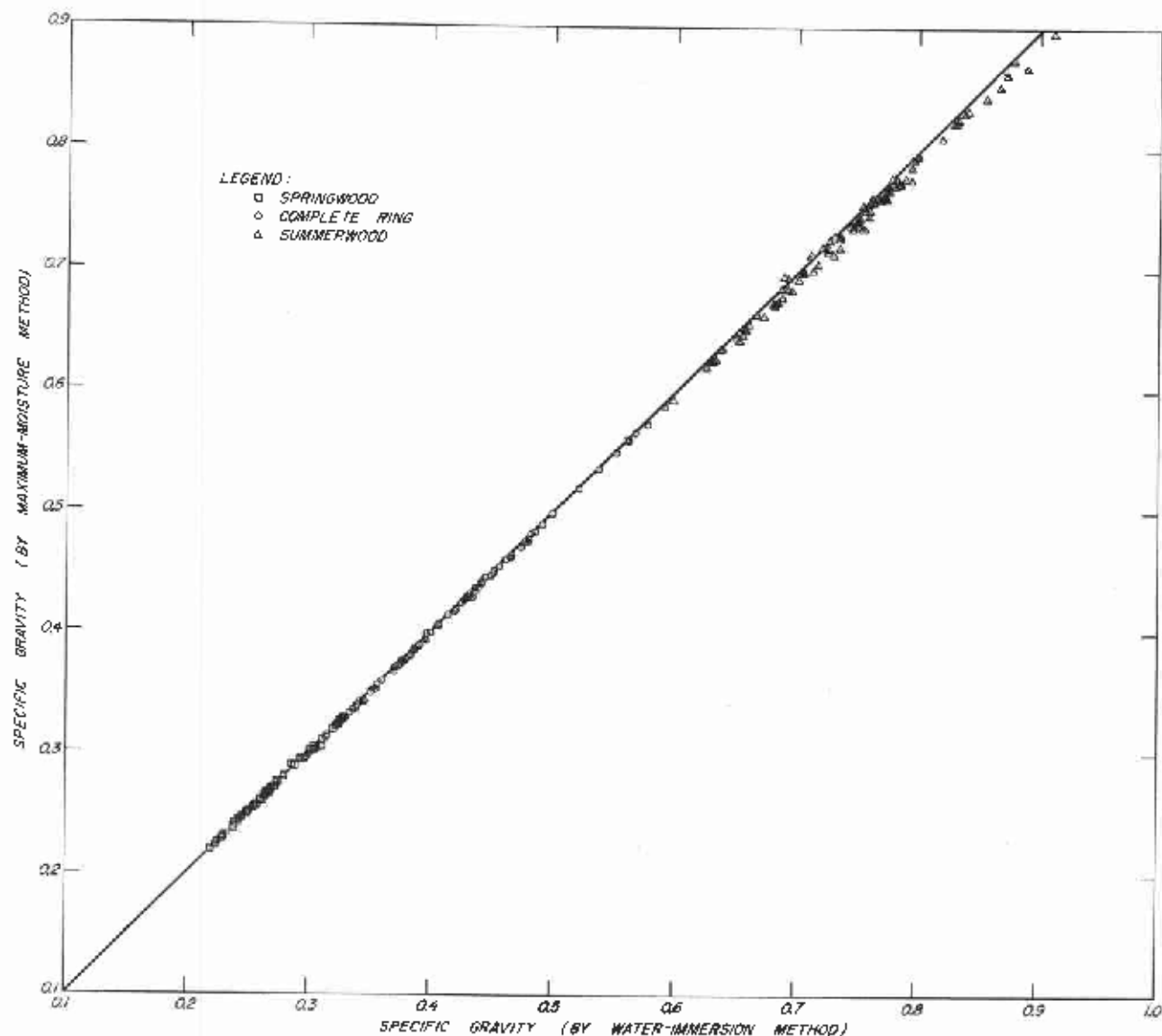


Figure 4.--Comparison of specific gravity values obtained by the maximum-moisture and water-immersion methods. The specific gravity of the cell wall substance was assumed to be 1.53 for the maximum moisture method.

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