# CELL-WALL DENSITY OF DOUGLAS-FIR BY TWO OPTOMETRIC METHODS<sup>1</sup>

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#### ABSTRACT

The change in cell-wall density from the water-swollen (1.0 g/cm<sup>3</sup>) to the oven-dry (1.43 g/cm<sup>3</sup>) condition is a function of the percent of shrinkage in the cell wall. Cell-wall density obtained optometrically by the Dual-Linear measuring micrometer and the dot-grid eyepiece compared favorably with densities reported for mercury porisimeter or picnometric techniques. Both optometric techniques are nondestructive and offer advantages over destructive techniques. Void volumes in the dry cell wall were calculated to be approximately 3.5%, fiber saturation point for extractive-free cell wall about 35%. Average interfibrillar spacing in the water-swollen condition was 20 Å for both earlywood and latewood and in the dry wall; spacing was 4.7 Å for earlywood and 8.3 Å for latewood.

Keywords: Specific gravity, cell-wall density, cell-wall area, cell-wall shrinkage, optometric measurements, Douglas-fir.

#### INTRODUCTION

This paper compares two nondestructive procedures for estimating cell-wall density of Douglas-fir in both the green and oven-dry condition: the Dual-Linear measuring micrometer and the dot-grid eyepiece. Estimates from these procedures allow the cell-wall void volume and the spacing between microfibrils within the wall to be derived in both the green and oven-dry conditions. These estimates also provide a basis to approximate dry wall density and void volume, both important in chemical bulking treatments, chemical modification, permeability studies, shrinkage, growth responses to silvicultural treatments, and heritability studies. Proportionate cross-sectional wall area was estimated directly from microtomed surfaces of extracted solid wood specimens of isolated but intact earlywood and latewood.

## LITERATURE REVIEW

Cell-wall density in the water-swollen, extractive-free condition is 1.53 g/cm<sup>3</sup> as determined picnometrically in water (Stamm 1929) and 1.46 g/cm<sup>3</sup> by helium in the oven-dry condition (Stamm and Hanson 1937). This range allows for a maximum of  $(1/1.46 - 1/1.53)1/1.46 = (1 - 1.46/1.53) \times 100 = 4.6\%$  void in the dry-wall volume uncorrected for absorption of water. Numerous reviews have been made concerning the controversy over both the cell-wall density in the dry state and the percent of voids in the dry cell wall (Berlyn 1964, 1970; Frey-Wyssling 1968; Kellogg and Wangaard 1969; Rollins and Tripp 1961; Stamm 1967b; and Wilfong 1964, 1966). Values for cell-wall density in the "near" or oven-dry

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condition as derived by some optometric method have been reported over the range of 0.71–1.325 (Besley 1964; Jayme and Krouse 1963; McIntosh 1965; Tsoumis 1964; Yiannos 1964; Harris 1969), which would indicate considerable pore space in the dry wall. These low values have been criticized (Feist and Tarkow 1967; Stamm 1967b; Weatherwax and Tarkow 1968b; Wilfong 1966) because they did not adjust values to account for the moisture content of the specimens. Values published for picnometric methods range from 1.42–1.61 g/cm<sup>3</sup> (Stamm 1964; Stamm and Seborg 1935; Wilfong 1966), while values published for mercury porosimeter methods range from 1.32–1.45 g/cm<sup>3</sup> (Stayton and Hart 1965; Stone et al. 1966). Other criticism that has been levied against published optometric methods for determining dry cell-wall density has been related to micrometric procedures that bulk, skew, or otherwise damage the wood sections used for these determinations (Boutelje 1962; Kellogg and Wangaard 1969).

In a review of published techniques, Smith (1967) categorized those by which the amount and distribution of cell wall can be determined: (1) test-point (dotgrid) method, (2) line integration method, and (3) cut-out-and-weigh (from photomicrographs) and planimetric methods. Of all the methods and techniques reviewed, only the one by Smith and Miller (1964) did not require either thin sectioning or some time-consuming preparatory methodology.

The technique for using the Dual-Linear measuring micrometer was initially described by Smith (1965). By modeling and utilizing various lumen configurations and "average" cell dimensions, she demonstrated that the estimated proportional cell-wall area gave excellent correlation (r = 0.997) with specific gravity determined by the maximum moisture method. She assumed a cell-wall substance density of 1.53 g/cm<sup>3</sup> (Stamm 1964), and for two independent sets of Douglas-fir specimens found cell-wall specific gravity to be 0.99 and 0.98 for the earlywood sets and 0.97 and 0.95 for the latewood sets in the water-swollen condition.

Using a dot-grid eyepiece with a microscope, Wangaard (1969) derived the proportionate area of cell wall from microtomed sections mounted in water. He published values for cell-wall density in the water-swollen condition as 0.999 for loblolly pine and 0.988 for spruce pine, but made no differentiation between earlywood and latewood.

The concept of dot-grid sampling was first used in mensurational techniques (Bryan 1943), subsequently to obtain the proportion of tissue types (Jayme and Krause 1963; Ladell 1959), fiber wall area as a percentage of total wood wall area (Kellogg and Ifju 1962), and the proportion of wall of various tissue types on a total dry wall weight basis (Chudnoff and Tischler 1963).

To determine comparability between the Dual-Linear measuring micrometer and the dot-grid eyepiece, Quirk (1975) developed two procedures for using the dot-grid eyepiece (random fields versus random passes) to yield a mean within specified limits of sampling error. Using the random pass technique, Quirk and Smith (1975) demonstrated that specific gravity of the water-swollen cell wall estimated from the proportional cell-wall area was highly correlated (r = 0.9976for Dual-Linear and r = 0.9991 for dot-grid) with specific gravity determined by the maximum moisture method. Cell-wall density for earlywood by Dual-Linear was 1.032 and 0.994 for dot-grid and 0.993 for latewood by both techniques. On the basis of a paired "t" test, the differences between earlywood and latewood were not significant.

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After it had been established that either the Dual-Linear or dot-grid technique gives comparable results (r = 0.9967) for estimating specific gravity in the waterswollen condition compared to specific gravity by the maximum moisture method, the two techniques were utilized for estimating specific gravity in the oven-dry condition (Quirk 1984). Cell-wall area values measured by the two techniques were highly correlated (r = 0.9987) for the oven-dry condition. Regressions were established for the estimated specific gravity and measurements of cell-wall area in the oven-dry condition. Forcing the regressions through the origin allows the slope of the regression to be an indicator of the cell-wall density (Quirk 1984).

Frey-Wyssling (1968) demonstrated that the basic microfibrils in the wood cell wall are 36 Å thick. Using these findings, Berlyn (1970) published a procedure for calculating interelementary fibrillar spacing within the dry cell wall, assuming that the diameters of the free space are relatively uniform, and arrived at a value of 1.7 Å.

A direct approach to "seeing" or measuring pore structure in the dry cell wall with the electron microscope has been unsuccessful. If interstices on the order of 1.7 Å are representative of the microcapillary structure, then these pore sizes are beyond the limit of resolution of a microscope, and necessitate indirect methods of estimating pore volumes based on statistical probabilities. Furthermore, interstices of this size are beyond the penetrating capacity of the measuring media because of their molecular structure and, therefore one would expect density estimates to be less than 1.53 g/cm<sup>3</sup> in the dry cell wall.

Berlyn's calculations of 1.7 Å spacing as well as evidence from Stamm (1950), Stone (1964, 1966), Stone et al. (1966), Stone and Scallan (1967), and Weatherwax and Tarkow (1968a) indicate that the density of the dry cell wall is essentially that of the wood substance—approximately 1.5 g/cm<sup>3</sup>—which would compute as  $(1 - 1.50/1.53) \times 100$  or 2% voids.

Therefore, it was considered necessary to maintain an assumption that the density of wood substance in the extracted condition is 1.53 g/cm<sup>3</sup> in water, and that the cell wall contains a finite number of 36 Å microfibrils. When completely water-swollen, the interfibrillar space is filled with water and the specific volume is 0.6536 cm<sup>3</sup>/g of solids. Upon drying, water lost from the interfibril space causes shrinkage; but the wall contains the same number of fibrillar elements and the density of the solids component remains at 1.53 g/cm<sup>3</sup>. In this way, integrity of the morphological structure is maintained and does not preempt the probable existence of microcapillaries in the dry cell wall. Data for spacing for this study are computed on this basis from both the Dual-Linear and the dot-grid techniques.

## EXPERIMENTAL PROCEDURE

The same specimens were used as in this author's previous study (Quirk 1975) with the exception of one earlywood specimen that was damaged and replaced. A total of six earlywood and six latewood specimens were measured to derive the amount of cell-wall area in tracheid walls only and also for whole wood (tracheids plus rays). The coefficient of correlation between cell-wall area as derived by both techniques was r = 0.9967 for the water-swollen condition and r = 0.9987 for the oven-dry condition. There was no significant difference in specific gravity of the dry wall determined by either technique. The average

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		Proportional tracheid wall area			Proportional wood wall area						
Specifi		Specific Dual-Line	Linear	iear Zeiss dot-grid		Dual-Linear		Zeiss dot-grid		Specific	
B		gravity	Green	Dry	Green	Dry	Green	Dry	Green	Dry	gravity
Specimen	Green	$(D_t)$	(D' <sub>0</sub> )	$(Z_1)$	$(Z_{t'})$	(D <sub>w</sub> )	(D' <sub>w</sub> )	$(Z_w)$	(Z'w)	Dry	
					M	m <sup>2</sup>					
				EA	RLYWC	OD					
1	0.3488	0.3828	0.2853	0.3466	0.2937	0.3742	0.2794	0.3459	0.2929	0.3928	
2	0.3492	0.3542	0.2541	0.3623	0.2699	0.3460	0.2488	0.3614	0.2690	0.3754	
3	0.2800	0.2666	0.1972	0.2943	0.1918	0.2574	0.1906	0.2933	0.1908	0.3040	
4	0.2760	0.2688	0.1995	0.2736	0.2050	0.2598	0.1925	0.2728	0.2041	0.3020	
5	0.2317	0.2301	0.1669	0.2328	0.1913	0.2243	0.1628	0.2323	0.1907	0.2406	
6	0.2198	0.2124	0.1558	0.2139	0.1785	0.2062	0.1513	0.2134	0.1780	0.2354	
Average	0.2842	0.2858	0.2098	0.2873	0.2217	0.2780	0.2042	0.2865	0.2209	0.3084	
				L	ATEWO	DD					
1	0.9538	0.9447	0.8836	0.9410	0.8742	0.9411	0.8785	0.9392	0.8724	1.3292	
2	0.9053	0.8934	0.8890	0.9025	0.8866	0.8879	0.8833	0.8998	0.8839	1.1807	
3	0.8266	0.8609	0.8338	0.8440	0.8108	0.8498	0.8204	0.8414	0.8082	1.1783	
4	0.7978	0.8452	0.8073	0.7974	0.7937	0.8388	0.7922	0.7945	0.7908	1.0874	
5	0.6949	0.6854	0.6718	0.7056	0.6408	0.6755	0.6607	0.7032	0.6384	0.9124	
6	0.6498	0.7067	0.6161	0.6645	0.6439	0.6978	0.6074	0.6480	0.6274	0.8754	
Average	0.8047	0.8227	0.7836	0.8092	0.7750	0.8143	0.7738	0.8043	0.7702	1.0939	

TABLE 1. Average measurement data for earlywood and latewood of Douglas-fir.

specific gravity of earlywood in the dry condition was  $0.308 \text{ g/cm}^3$ , and that for latewood in the dry condition was  $1.094 \text{ g/cm}^3$ .

Data on the proportionate amount of tracheid wall and tracheid wall plus ray wall (whole wood) are listed in Table 1 (from the prior study).

Cell-wall density (P) in the water-swollen or oven-dry condition was calculated from the relationship

$$P = G/\% \text{ cell-wall area}$$
(1)

where G is bulk specific gravity, green volume basis. Actual specific gravity was measured by the maximum moisture method. The relationship of actual specific gravity to estimated cell-wall area that has been derived by both measuring techniques in the water-swollen condition are given by the regression equations in Table 2. Specific gravity in the oven-dry condition (G') was estimated by measuring the coefficient of shrinkage (S) from the green to the oven-dry condition using the relationship

$$G' = G/(1 - S)$$
 (2)

where G is bulk specific gravity, green volume basis. The coefficient of shrinkage (S) is the change in the proportionate amount of cell-wall area per  $mm^2$  due to the radial and tangential movement from drying (Quirk 1984). The relationship between estimated cell-wall area and specific gravity in the dry condition is given by the regression equations in Table 2. From the relationship of Eq. (1), estimates of average cell-wall density can be made using the slope of the regression. The regression equations noted in Table 2 were forced through 0, yielding values listed

	Through data	Through origin
	TRACHEIDS ONLY	
Water-swollen		
Dual-Linear	$y = 0.0057 + 0.972x; r \approx 0.9963; S_{yx} = 0.0244$	$y = 0.9803x; r = 0.9993; S_{yx} = 0.0246$
Dot-grid	$y = -0.0042 + 1.0009x; r = 0.9994; S_{yx} = 0.0098$	$y = 0.9948x; r = 0.9987; S_{yx} = 0.0156$
Dual-Linear on dot-grid	$y = -0.0066 + 1.023x; r \approx 0.9967; S_{yx} = 0.0235$	For service proceeding of the process of the service of the ser
Oven-dry		
Dual-Linear	$y = 0.0193 + 1.373x; r = 0.9959; S_{yx} = 0.0405$	$y = 1.401x; r = 0.9989; S_{yx} = 0.0399$
Dot-grid	$y = -0.0081 + 1.423x; r = 0.9969; S_{yx} = 0.0330$	$y = 1.411x$ ; $r = 0.9987$ ; $S_{yx} = 0.0431$
Dual-Linear on dot-grid	$y = -0.0198 + 1.0364x; r \approx 0.9987; S_{yx} = 0.0156$	
	WHOLE WOOD	
	(Tracheids, Rays, and Resin Ducts)	
Water-swollen		
Dual-Linear	$y = 0.0132 + 0.9727x; r = 0.9967; S_{yx} = 0.0243$	$y = 0.9919x; r = 0.9993; S_{yx} = 0.0240$
Dot-grid	$y = -0.0041 + 1.006x; r \approx 0.9995; S_{yx} = 0.0094$	$y = 0.9998x; r = 0.9998; S_{yx} = 0.0092$
Oven-dry		
Dual-Linear	$y = 0.0077 + 1.431x; r \approx 0.9953; S_{yx} = 0.0436$	$y = 1.419x; r = 0.9988; S_{yx} = 0.0418$
Dot-grid	$v = 0.0254 + 1.382x; r = 0.9962; S_{m} = 0.0392$	$v = 1.420x$ ; $r = 0.9989$ ; $S_{m} = 0.0398$

TABLE 2. Regression of specific gravity and ratio of wall to cross-sectional area.

in Table 2. This procedure was done because some of the Y intercept values were plus or minus.

Cell-wall density was calculated for each of the specimens from the data in Table 1 for tracheids only and whole wood (tracheids + rays). The numerical average for the six specimens of earlywood and the six specimens of latewood is listed in Table 3, rather than values obtained from calculation of the "average specific gravity" and "average proportional wall area" values.

Specific gravity of the completely swollen cell wall at or above fiber saturation can be calculated from the equation (Stamm 1964)

$$Pm = \frac{Psm}{1 + Psm \cdot M}$$
(3)

where Psm is the specific gravity of the dry cell-wall substance determined by water displacement. M was equated to the computed void volume in the cell wall (VVcw) in the water-swollen condition.

It is assumed that the specific gravity of the cell wall, Pm, at any intermediate moisture content between the fiber saturation point and the oven-dry condition can be computed from the equation (Stamm 1964)

$$Pm = \frac{Pso}{1 + \frac{Pso(M)}{Pw}}$$
(4)

where Pso is the true specific gravity of the dry cell-wall substance determined by helium displacement or 1.46 g/cm<sup>3</sup> (Stamm and Hanson 1937). M is the absorbed moisture content (below saturation of the fiber) in grams per gram of dry wood substance or its equivalent, the void volume in the cell wall below fiber saturation point, and Pw is the average specific gravity of the absorbed water. Reviews by Wangaard (1969), and Kellogg and Wangaard (1969), demonstrate a more acceptable value for density of absorbed water to be 1.014 g/cm<sup>3</sup>, as calculated by Weatherwax and Tarkow (1968), rather than 1.113 g/cm<sup>3</sup> (Stamm 1929, 1950). For computational use, Pw was taken to be 1.014. Computational M was equated to the computed void volume in the dry cell wall (VVcw).

If the measured volume is greater than the volume of the cell-wall substance, the extra volume must be due to voids. Estimates of void volume in the cell wall in this VVcw were computed from the reciprocal of density—the specific volume (Vs) which for the swollen condition is 1/1.53 = 0.6536 cm<sup>3</sup>/g and for the ovendry condition is 1/1.46 = 0.6849 cm<sup>3</sup>/g of solids. The average values derived by both Dual-Linear and dot-grid techniques are listed in Table 2 for the six early-wood and six latewood specimens.

For computational use the relationship D = 1.53(1 - VVcw) was rearranged to adjust for density of absorbed water at 1.014 or 0.0052 cm<sup>3</sup>/g as

$$VVcw = \left[ \left( \frac{1}{D} + 0.0052 \right) - \frac{1}{1.53} \right]$$
 (5)

The adjusted specific volume minus 0.6536 (water swollen) or 0.6849 (oven-dry) yielded the values for VVcw in Table 3.

Estimates of the fiber saturation point were based on the relationship (VVcw/

	Cell-wall density (P)	Specific volume (Vs)	Void volume in cell wall (VVcw)	Percent of void volume in cell wall	Adjusted <sup>1</sup> cell-wall density (P)
	g/cm <sup>3</sup>	cm <sup>3</sup> /g	cm <sup>3</sup> /g	Pct	g/cm <sup>3</sup>
		TRACHEID	S—GREEN		
Earlywood	0.9922	1.0085	0.3606	35.48	0.987
Latewood	0.9932	1.0070	0.3586	35.42	0.988
Annual ring	0.9923	1.0077	0.3593	35.47	0.987
Regression	0.9948	1.0052	0.3568	35.31	0.990
		TRACHEI	DS—DRY		
Earlywood	1.394	0.7216	0.0732	9.55	1.384
Latewood	1.410	0.7107	0.0623	8.52	1.400
Annual ring	1.399	0.7149	0.0665	9.23	1.388
Regression	1.411	0.7087	0.0603	8.44	1.401
	TR	ACHEIDS AND	RAYS-GREEN		
Earlywood	0.995	1.0060	0.3576	35.32	0.990
Latewood	1.000	1.0002	0.3519	34.98	0.995
Annual ring	0.9965	1.0035	0.3551	35.20	0.991
Regression	0.9998	1.0002	0.3518	34.99	0.995
	Т	RACHEIDS AN	D RAYS-DRY		
Earlywood	1.399	0.7191	0.0615	9.23	1.389
Latewood	1,420	0.7057	0.0573	7.90	1.409
Annual ring	1.405	0.7117	0.0633	8.83	1.395
Regression	1,420	0.7042	0.0560	7.89	1.409

TABLE 3a. Cell-wall density (P) and void volume in cell wall (VVcw) for earlywood and latewood in the water-swollen and oven-dry condition by dot-grid eyepiece.

<sup>1</sup> Adjusted volume for density of water Pw = 1.014 or 0.0052 cm<sup>3</sup>/g.

Vs)  $\times$  100 or the percent of void volume in the cell wall. Average values for earlywood and latewood specimens are listed in Table 3. The individual measurements for earlywood and latewood cell-wall densities cannot be directly compared to the estimates derived from the regressions. Therefore, the average values for cell-wall density of tracheids and tracheids plus rays (whole wood) for both the water-swollen and oven-dry conditions were weighed by the corresponding ring volume occupied (earlywood 70% and latewood 30%). Average values are listed in Table 3 as "Annual ring."

### **RESULTS AND DISCUSSION**

In the water-swollen condition, there was little (0.5%) difference in cell-wall density between earlywood and latewood by the dot-grid technique (Table 3a). This finding was directly in support of Lange's (1958) results, that the average earlywood cell-wall density is 2% less than that of latewood. The opposite is true for the Dual-Linear technique that indicated earlywood to be approximately 3% denser than latewood (Table 3b).

In the oven-dry condition, there was little (1.0%) difference in cell-wall density between earlywood and latewood by the dot-grid technique. However, Dual-Linear technique results showed earlywood to be 6% more dense than latewood (Table 3b).

	Cell-wall density (P)	Specific volume (Vs)	Void volume in cell wall (VVcw)	Percent of void volume in cell wall	Adjusted cell-wall density (P)
	g/cm <sup>3</sup>	$cm^3/g$	cm <sup>3</sup> /g	Pct	$g/cm^3$
		TRACHEID	S—GREEN		
Earlywood	1.0026	0.9995	0.3511	34.80	0.9974
Latewood	0.9767	1.0253	0.3769	36.38	0.9718
Annual ring	0.9948	1.0052	0.3568	35.31	0.9897
Regression	0.9803	1.0201	0.3717	36.25	0.9753
		TRACHEI	DS—DRY		
Earlywood	1.4770	0.6780	0.0296	4.20	1.4657
Latewood	1.3953	0.7180	0.0695	9.46	1.3852
Annual ring	1.4525	0.6885	0.0401	5.78	1.4415
Regression	1.401	0.7138	0.0654	9.09	1.3910
	TR	ACHEIDS ANI	RAYS-GREEN	r	
Earlywood	1.0317	0.9717	0.3233	32.92	1.0262
Latewood	0.9871	1.0145	0.3660	35.81	0.9820
Annual ring	1.0183	0.9820	0.3336	33.79	1.0130
Regression	0.9919	1.0082	0.3598	35.50	0.9868
	Т	RACHEIDS AN	D RAYS-DRY		
Earlywood	1.5187	0.6596	0.0112	0.20	1.5068
Latewood	1.4135	0.7086	0.0602	8.28	1.4032
Annual ring	1.4871	0.6724	0.0240	3.54	1.4758
Regression	1.419	0.7047	0.0563	7.93	1.4086

TABLE 3b. Cell-wall density (P) and void volume in cell wall (VVcw) for earlywood and latewood in green and oven-dry condition by Dual-Linear.

When weighting the fractional components of the annual ring, estimates of the "average" cell-wall density were slightly higher by Dual-Linear and slightly lower by dot-grid compared to those obtained from forcing the regressions through the origin. Cell-wall density estimates of latewood alone by either technique were nearly identical with the regression estimates.

In the oven-dry condition, cell-wall density derived by measurement using either the Dual-Linear or the dot-grid technique demonstrated the same pattern as found in the water-swollen condition. Earlywood was slightly less dense than latewood by the dot-grid technique for either tracheids alone or whole wood. On the other hand, latewood was more dense than earlywood by the Dual-Linear technique for either tracheids alone or whole wood (Table 3). The value for whole wood derived by regression was 1.42 by either technique and is perhaps more realistic compared to accepted values published in the literature. The values derived in this study from the microtomed smooth surfaces of separated but intact blocks of all earlywood or of all latewood cells in the extractive-free condition are good approximations similar to those attained by porisimeter or picnometrically.

The average values for calculated specific volume in the water-swollen and oven-dry condition derived from Dual-Linear and dot-grid methods for both earlywood and latewood are listed in Table 4. Shrinkage was computed as the change TABLE 4. Specific volume (Vs) and shrinkage.

		Earlywood		Late	wood	Regression	
		Z	DL	Z	DL	Z	DL
		TR	ACHEIDS	1			
Water-swollen specific volume (VsG)	cm <sup>3</sup> /g	1.0085	0.9995	1.0070	1.0253	1.0052	1.0201
Oven-dry specific volume (VsD)	cm <sup>3</sup> /g	0.7216	0.6780	0.7057	0.7180	0.7087	0.7138
Shrinkage $(VsG - VsD)/VsG \times 100$	pct	28.45	32.16	29.92	29.97	29.50	30,02
Wall area shrinkage from prior study	pct	28.03	31.85	29.67	30.08	_	
		TRACHE	IDS AND	RAYS			
Water-swollen specific volume (VsG)	cm <sup>3</sup> /g	1.0060	0.9717	1.0002	1.0145	1.0002	1.0082
Oven-dry specific volume (VsD)	cm <sup>3</sup> /g	0.7191	0.6596	0.7957	0.7086	0.7042	0.7047
Shrinkage $(VsG - VsD)/VsG \times 100$	pct	28.52	32.12	29.44	30.15	29.59	30.10
Wall area shrinkage from prior study	pct	28.39	32.07	29.43	30.03	_	

in volume and is shown compared to total wall area shrinkage values derived from the prior study on shrinkage of cell wall in Douglas-fir (Quirk 1984). The data would indicate that the final cell-wall density value in the oven-dry condition is a direct function of the percent shrinkage that occurs as the cell wall loses moisture.

The individual values of cell-wall density for the six earlywood and the six latewood specimens were plotted against the percent shrinkage that occurred in the total wall area and are shown in Fig. 1 for tracheids only and Fig. 2 for tracheids plus rays (whole wood). Coefficients of correlation of 0.97 were found.

From the spread of the data points, it is obvious that both earlywood and latewood samples varied considerably in the percent shrinkage and subsequent final estimated wall density.

Estimates of cell-wall density can be attained with better precision with either the Dual-Linear micrometer or the dot-grid ocular eyepiece in conjunction with an incident illuminating microscope than can be attained with cut sections. The wood specimen is left fully intact, and the sampling field is optically superimposed upon the wood image.

Determinations of proportional wall area are more time-consuming with the dot-grid eyepiece, yet most laboratories are not equipped with a Dual-Linear micrometer. The dot-grid eyepiece, which is readily available and of small cost, is just as useful in obtaining proportional data.

Multiple regressions as used here are limited to predicting the specific gravity or cell-wall density of the wood under the given set of test conditions for which they were developed. Therefore it would be inadvisable to apply such equations, developed on the basis of a restricted sample, to the species in general. Never-



FIG. 1. Cell-wall density of tracheids only of earlywood and of latewood with percent shrinkage occurring in tracheid wall area.

theless, this comparison demonstrates that from a few sample specimens one first can ascertain the variance and from that the degree of sampling for a desired precision; one therefore can develop new predictive regressions for a new set of test conditions.



FIG. 2. Cell-wall density of whole wood (tracheids plus rays) of earlywood and of latewood with percent shrinkage occurring in whole-wood wall area.

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As mentioned earlier, most controversy in the literature concerns estimating or measuring void volumes in the cell wall (VVcw) and the size of the interfibrillar spacings, especially in the near or oven-dry condition.

Total wall volume less the specific volume of solids gives estimates of the void volume in the cell wall (VVcw) at the time and conditions of measurement. From the relationship D = 1.53(1 - VVcw), it can be shown that if the value for cell-wall density in the water-swollen condition is taken as unity or at least closest to the density of water at 4 C, estimates of the VVcw would be 34.6%. In this study the derived values in Table 3 indicated that a fiber saturation point estimated from the void volume in the water-swollen cell wall would be 32.9% for earlywood and 35.8% for latewood by the Dual-Linear technique. The estimated fiber saturation point is 35.3% for earlywood and 35.0% for latewood by the dot-grid technique. These values are similar to a fiber saturation point of 35% for loblolly pine, and 36% for spruce pine found by Wangaard (1969), and are in keeping with other data (Feist and Tarkow 1967; Nearn 1955; Stamm 1971; Stone and Scallan 1967; Wangaard and Granados 1967) indicating either that removal of extractives increases the fiber saturation point of the wood or that the true fiber saturation point is lowered because of the bulking action of extractives.

Data for this study by both the Dual-Linear and the dot-grid computed on the basis of 1.53 g/cm<sup>3</sup> would indicate a 7.93% void volume (VVcw) in the dry wall by Dual-Linear and a 7.89% void volume (VVcw) in the dry wall by dot-grid (Table 3). If one were to use the density value of 1.46 g/cm<sup>3</sup> (helium) and adjust the values in Table 3, however, the adjusted percentages would be 1 - 1.41/1.46 = 3.4%, more in keeping with expected published values.

Using the formula of Berlyn (1970), the spacing between microfibrils within the water-swollen cell wall computes to 20 Å for both earlywood and for latewood. The average interfibrillar spacings between elementary fibrils in the dry wall compute as 4.7 Å in earlywood and 8.3 Å for latewood, by either of the two techniques.

## SUMMARY

Dry cell-wall density (1.42 g/cm<sup>3</sup>) determined by optometric methods for estimating wall volume was equivalent to values obtained by mercury porosimeter and picnometric methods using nonswelling immersion media.

Void volume estimates of the cell wall in the water-swollen condition gave good estimates of the fiber saturation point.

In addition, data indicated that cell-wall density from the water-swollen to the oven-dry condition is governed by the percentage shrinkage that takes place in the cell walls or that cell-wall density is cell-wall substance plus void volume. Finally, the two methods demonstrate that anatomic parameters of any intact wood specimen can be estimated under an incident illuminating microscope at any test condition to derive cell-wall area, cell-wall density, and specific gravity, which are important to pulping, permeability, gluing, veneering, or particleboard studies as well as to those studies involving strength-structure relationships.

## REFERENCES

BERLYN, G. P. 1964. Recent advances in wood anatomy: The cell walls of secondary xylem. For. Prod. J. 14(10):467–476.

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—. 1970. Ultrastructural and molecular concepts of cell-wall formation. Wood Fiber 2(3):196–227.

- BESLEY, L. 1964. The significance of fiber geometry and distribution in assessing pulpwood quality. Tappi 47(11):183A-184A.
- BOUTELJE, J. 1962. On shrinkage and change in microscopic void volume during drying as calculated from measurements on microtome cross-sections of Swedish Pine (*Pinus sylvestris* L.). Svensk Paperstid. 65:209–215.

BRYAN, M. M. 1943. Area determination with the modified acreage grid. J. For. 41(10):764-766.

- CHRISTENSEN, G. N., AND H. F. A. HERGT. 1968. The apparent density of wood in non-swelling liquids. Holzforschung 22(6):165–170.
- CHUDNOFF, M., AND K. TISCHLER. 1963. Fiber morphology of *Eucalyptus camaldulensis* Dehn. and the relation of wood anatomy to certain physical and mechanical properties. Israel Forest. Assoc., Ilanoth. La-Yaaran. Supplement 1. pp. 3–23.
- FEIST, W. C., AND H. TARKOW. 1967. Polymer exclusion in wood substance, a new procedure for measuring fiber saturation points. For. Prod. J. 17(10):65–68.

FREY-WYSSLING, A. 1968. The ultrastructure of wood. Wood Sci. Technol. 2:73-83.

- HARRIS, J. M. 1969. The use of beta rays in determining wood properties. Part 4, Estimating the thickness of tracheid cell walls. N.Z. J. Sci. 12(2):432–442.
- JAYME, G., AND T. KRAUSE. 1963. Über die Packungsdichte der Zellwäande in Laubhölzern. Holz Roh- Werkst. 21(1):14–19.

KELLOGG, R. M., AND G. IFJU. 1962. Influence of specific gravity and certain other factors on the tensile properties of wood. For. Prod. J. 12(10):463–470.

- —, AND F. F. WANGAARD. 1969. Variations in the cell-wall density of wood. Wood Fiber 1(3): 180–204.
- LADELL, J. I. 1959. A method of measuring the amount and distribution of cell wall material in transverse microscope sections of wood. J. Inst. Wood Sci. 3:43–46.
- LANGE, P. W. 1958. The distribution of the chemical constituents throughout the cell wall. Pages 147–186 in F. Bolam, ed., Fundamentals of paper-making fibers. Trans. Symp. held at Cambridge in 1957.
- MCINTOSH, D. C. 1965. Wall structure of loblolly pine summerwood holocellulose fibers in relation to individual fiber strength. Pages 561–571 in W. A. Côté, ed., Cellular ultrastructure of woody plants. Syracuse Univ. Press, Syracuse, NY.
- NEARN, W. T. 1955. Effect of water soluble extractives on the volumetric shrinkage and equilibrium moisture content of eleven tropical and domestic woods. Penn. State Univ., Agric. Exp. Stn. Bull. No. 598.
- QUIRK, J. T. 1975. Dot-grid integrating eyepiece: Two sampling techniques for estimating cell wall areas. Wood Sci. 8(2)88-91.
- ——. 1984. Shrinkage and related properties of cell walls of Douglas-fir. Wood Fiber Sci. 16(1): 115–133.

—, AND D. M. SMITH. 1975. Comparison of Dual-Linear and dot-grid eyepiece methods for estimating wood properties of Douglas-fir. Wood Sci. 8(2):92–96.

- ROLLINS, MARY L., AND VERNE W. TRIPP. 1961. The architecture of cellulose: A microscopic review. For. Prod. J. 11(11):493–504.
- SMITH, D. M. 1965. Rapid measurement of tracheid cross-sectional dimensions of conifers: Its application to specific gravity determinations. For. Prod. J. 15(8):325–334.
- ——. 1967. Microscopic methods for determining cross-sectional cell dimensions. USDA For. Serv. Res. Pap. FPL 79, For. Prod. Lab., Madison, WI.
- —, AND R. B. MILLER. 1964. Methods of measuring and estimating tracheid wall thickness of redwood (*Sequoia sempervirens* (D. Don) Endl.). Tappi 47:599–604.
- STAMM, A. J. 1929. Density of wood substance and absorption by wood. J. Phys. Chem. 33:398.
- -----. 1950. Specific volume of native cellulose. Textile Res. J. 20(9):631-636.
- ------. 1964. Wood and cellulose science. Ronald Press Co., New York, NY.
- \_\_\_\_\_. 1967a. History of two phases of wood physics. Wood Sci. and Tech. 1:186-190.
- . 1967b. Review of nine methods for determining the fiber saturation points of wood and wood products. Wood Sci. 4(2):114–128.
- 1971. Review of nine methods for determining the fiber saturation points of wood and wood products. Wood Sci. 4(2):114–128.

#### WOOD AND FIBER SCIENCE, APRIL 1984, V. 16(2)

—, AND L. A. HANSEN. 1937. Bonding force of cellulosic materials for water, from specific volume and thermal data. J. Phys. Chem. 41(7):107–116.

------, AND R. M. SEBORG. 1935. Absorption compression of cellulose and wood. J. Phys. Chem. 39:132.

STAYTON, C. L., AND C. A. HART. 1965. Determining pore size distribution in softwoods with a mercury porosimeter. For. Prod. J. 15(10):435–440.

STONE, J. E. 1964. The porous structure of wood and fibers. Pulp Pap. Mag. Can. 65(1):T3-T12.

— 1966. The cell wall. Trend Mag. Activities of the Pulp and Paper Institute of Canada, No. 7:4–9. Spring.

, AND A. M. SCALLAN. 1967. The effect of component removal upon the porous structure of the cell wall of wood. II. Swelling in water and the fiber saturation point. Tappi 50(10):496– 501.

—, —, AND G. M. A. ABERSON. 1966. The wall density of native cellulose fibers. Pulp Pap. Mag. Can. 67(5):T263–268.

TRENDELENBURG, R. 1939. Über Fasersättigungsfeuchtigkeit, Schwindmass, und Raumdichtezahl wichtiger Holzarten. Holz Roh- Werkst. 2(1):12–17.

TSOUMIS, G. 1964. Microscopic measurement of the amount of cell wall substance in wood and its relationship to specific gravity. Tappi 47(11):675–677.

WANGAARD, F. F. 1969. Cell-wall density of wood with particular reference to the southern pines. Wood Sci. 1(4):222–226.

——, AND L. A. GRANADOS. 1967. The effect of extractives on water-vapor sorption of wood. Wood Sci. Technol. 1:253–277.

WEATHERWAX, R. C., AND H. TARKOW. 1968a. Cell-wall density of dry wood. For. Prod. J. 18(2): 83-85.

------, AND -------. 1968b. Density of wood substance. Importance of penetration and adsorption compression of the displacement fluid. For. Prod. J. 18(7):44-46.

WILFONG, J. G. 1964. Improved techniques for determining the specific gravity of wood substance. M.S. Thesis, Univ. North Carolina at Raleigh.

----. 1966. Specific gravity of wood substance. For. Prod. J. 16(1):55-61.

YIANNOS, P. N. 1964. The apparent cell-wall density of wood and pulp fibers. Tappi 47(8):468-471.