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# Thermal Degradation of Wood and Cellulose

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HEAT has two effects upon the properties of wood and cellulose—reversible (*6, 8, 24, 27, 29*) and irreversible. This report deals with the irreversible degradative changes caused by heat.

## CHEMICAL CHANGES

The literature is prolific on the changes that accompany thermal degradation of cellulose. Viscosity changes (*16, 18, 23, 25*) have been reported. Chemical interpretations have been made (*3–5, 7, 13–15, 28*). Goos (*2*) lists 213 compounds that have been identified. Reduction in hygroscopic and swelling characteristics have been reported (*20, 22, 26*). The specific chemical changes that occur during thermal degradation are still obscure.

Data on decomposition products of Douglas fir sawdust and its components are given in Tables I and II.

**Table I. Decomposition Products of Extracted Douglas Fir Sawdust**

(Heated in a stream of air and of nitrogen at 300° C. for 15 hours at atmospheric pressure)

| Heated in | Weight[a] Loss, % | Decomposition Products[a], % | | | | |
|---|---|---|---|---|---|---|
| | | $CO_2$ | $H_2O$ | Tars | Noncondensable volatile[b] | Excess over weight loss |
| Air | 40.0 | 16.0 | 30.6 | 2.8 | [c] | 8.0+ |
| Nitrogen | 47.0 | 7.8 | 31.1 | 5.2 | 2.9 | 0 |

[a] Based on weight of unheated, oven-dry sawdust.
[b] Calculated from difference between original weight loss and weight of other products.
[c] Undeterminable, probably between 0.0 and 3.0.

**Table II. Decomposition Products of Extracted Douglas Fir Sawdust and of Its Isolated Components**

(Heated in both an open system at atmospheric pressure and in a closed system under the developed pressure at 220° C.)

| Materials and Heating Conditions | Heating Time, Hours | Weight Loss[a], % | Decomposition Products, %[a] | | | |
|---|---|---|---|---|---|---|
| | | | $CO_2$ | $H_2O$ | Tars | Noncondensable volatiles |
| Open system | | | | | | |
| Sawdust | 16 | 9.9 | 0.9 | 8.3 | 0.4 | 0.3 |
| α-Cellulose | 16 | 13.9 | 1.4 | 11.8 | 0.0 | 0.7 |
| Hemicellulose | 2 | 23.7 | 7.4 | 14.1 | 0.4 | 1.8 |
| Lignin | 16 | 4.2 | 0.5 | 3.6 | 0.0 | 0.1 |
| Closed system | | | | | | |
| Sawdust | 4 | 15.9 | 3.6 | 12.1 | .. | 0.2 |
| α-Cellulose | 4 | 37.7 | 8.1 | 26.0 | .. | 3.6 |
| Hemicellulose | 0.5 | 43.1 | 17.2 | 22.8 | .. | 3.1 |
| Lignin | 4 | 3.7 | 1.1 | 2.0 | .. | 0.7 |

[a] Based on weight of unheated, oven-dry material.

## KINETICS OF WEIGHT LOSS

MacLean (*9–12*) and Stamm and associates (*13, 20, 21*) have collected extensive data on the weight loss of wood under different heating conditions, for times ranging from 1 minute to 2.4 years (1.25 × $10^6$-fold), and temperatures ranging from 93.5° to 300° C. Hence it seemed worth while to analyze these data from the standpoint of reaction kinetics.

Figures 1 and 2 give the logarithms of the residual weight plotted against the heating time for oven heating and heating beneath the surface of a molten metal. Good straight lines are obtained, except near the origin where there is a slight but definite curvature. The initially high loss in weight is probably due to the loss of residual absorbed water. The linear relationships indicate that the weight loss follows a first-order reaction. Because it is a reaction at the surface of a solid and because so many different reactions are taking place simultaneously, the effective reaction is undoubtedly a heterogeneous one.

Figure 3 gives the plot of the reciprocals of the absolute temperature against log *K* where *K* is the slope of the lines of Figures 1 and 2 per unit of time (hours). All of the data for oven heating fall well on a single straight line, in spite of the fact that the data were obtained by two different groups of investigators using different softwood species (coniferous woods) with different sizes and shapes of specimen. Similar data for the heating of Sitka spruce veneer beneath the surface of a molten metal (*21*) give a straight line with practically the same slope. Heating beneath the surface of a molten metal limits the oxygen available for oxidation to the relatively small amount with the capillary structure of the wood. As the temperature of the wood rises, the air expands and a large part of it bubbles out before it can cause oxidation. Heating in this way therefore permits only limited oxidation. Because of this, the degradation is slower than for the oven heating; hence the displacement of the line to the left.

Table III gives the activation energies for the thermal degradation of wood calculated from the data of Figure 3 and similar data to follow. It also gives the log *K* values at 150° C., so that the displacement of the activation energy curves can be estimated. The log *K* value for heating in air is 0.5 greater than for heating beneath the surface of the molten metal. The former reaction is thus 3.2 times as fast as the latter with respect to weight loss.

Approximate activation energies were calculated from the data of Mitchell, Seborg, and Millett (*13*) for the thermal degradation of Douglas fir sawdust and for the α-cellulose, hemicellulose, and lignin isolated therefrom when heated in an open system. At each temperature the materials were heated for only one period of time. The logarithms of the residual weight–time plots hence were drawn as straight lines through the one point and the origin. Table III gives the activation energies and log *K* at 150° C. It is interesting that the various wood components and the wood itself have very similar activation energies. The log *K* values at 150° C. show that hemicellulose degrades thermally about four times as fast as the wood. The α-cellulose degrades at about the same rate, while the lignin degrades at about half of the rate of the wood. 10-kantainen log

Table III also gives data for the activation energy and the log *K* values at 150° C. for steamed wood calculated from the data of MacLean (*10, 11*) for the same group of softwoods given in Figure 1. The logarithm of the residual weight–time curves similar to those of Figures 1 and 2 showed somewhat more curvature near the origin. This is probably due to the fact that initially green wood was used in these measurements and adsorbed water was lost in the initial stages of heating. Calculations from these data are hence somewhat less accurate than for dry wood, but they do give the order of magnitude of degradation.

ex

ex

$10^{-?}$

10

$10^{-4}$

**THERMAL DEGRADATION OF WOOD**

*is greater under these conditions*

**. . . closed system**

**. . . presence of air**

**. . . steaming conditions**

It is negligible under normal kiln-drying conditions

The calculated activation energy is about half of the value for the heating of wood under dry conditions. The log $K$ value at 150° C. indicates that the degradation is much faster under steaming than under dry heating conditions.

Figure 4 gives the data of Figures 1 and 2 plotted as the logarithm of the heating time against the heating temperature, extrapolated to room temperature. For each weight loss, a good straight line is obtained. These lines tend to converge at the low temperature–long time end. This would be expected on the basis of the Arrhenius equation for a first-order reaction rate, the temperature coefficient of the reaction rate being inversely proportional to the absolute temperature. At 100° C. the reaction rate for oven heating, as indicated by the weight loss, doubles for each 10° C. increase in temperature. At 250° C., it doubles for each 13° to 14° C. rise in temperature. The ratio of temperatures is practically identical with the theoretical value.

As oven heating and heating beneath the surface of a molten metal give almost identical activation energies, their plots in Figure 4 should be parallel and identical when the weight loss due to heating in an oven is 3.2 times that for heating beneath the surface of a molten metal. The values plotted best when the multiple was 3.3.

From Figure 4 it is possible to estimate the degradation of wood in terms of weight loss at normal temperatures over prolonged periods of time. For example, dry wood stored for 355 years at 20° C. should lose 5% of its weight when air circulates freely around it, but only 1.5% if air circulation is excluded. As these weight losses are accompanied by a decrease in volume (*20*), the loss in specific gravity is too small to use as a criterion of the degradation of old timbers due to weight loss.

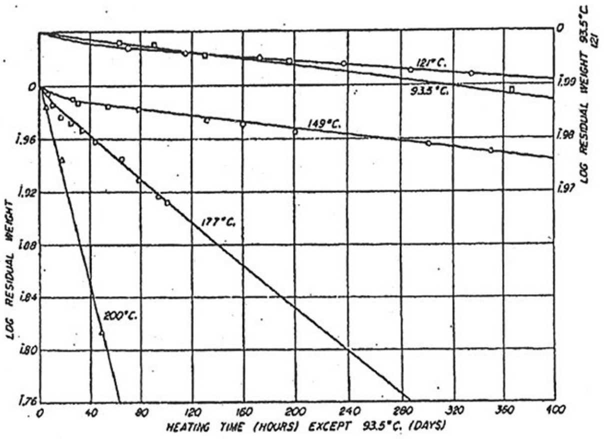

**Figure 1.** Logarithm of residual weight *vs.* time of oven heating

○, □ Average of four specimens each of southern pine, white pine, Douglas fir, and Sitka spruce 6 inches long in fiber direction by 1 inch by 1 inch (*9–12*)

△ Rotary-cut Sitka spruce veneer $^{1}/_{16}$ inch thick, $5^{7}/_{8}$ inches long, and 1 inch wide (*21*)

Figure 5 is a plot similar to that of Figure 4 for steamed wood. If the lines were extrapolated to room temperature, they would give an expected weight loss of 5% if the wood were subjected to saturated water vapor at 20° C. for 1 year. This will be referred to again later.

No extended data on weight loss for heating of cotton or paper pulp were found in the literature. Data are, however, available for strength loss caused by heating.

### KINETICS OF STRENGTH LOSS

Similar kinetic calculations can be made from data for the rate of strength loss. When the logarithm of the residual strength is plotted against the time, straight lines similar to those of Figures 1 and 2 are obtained. Activation energies for both oven- and steam-heated wood were calculated from such plots drawn from data of MacLean (*9, 11*) for the residual modulus of rupture and the residual work to maximum load (area under the stress-strain curves to the point at which rupture occurs in modulus-of-rupture measurements). Table III shows that the activation energies so obtained agree very well with the corresponding values calculated from the weight loss. The log $K$ values in Table III show that the rate of loss of modulus of rupture is about 10 times that for the weight loss under both oven-heating and steaming conditions. The rate of loss of work to maximum load is still greater. Figures 4 and 5 also show the approximately tenfold greater loss in modulus of rupture than of weight.

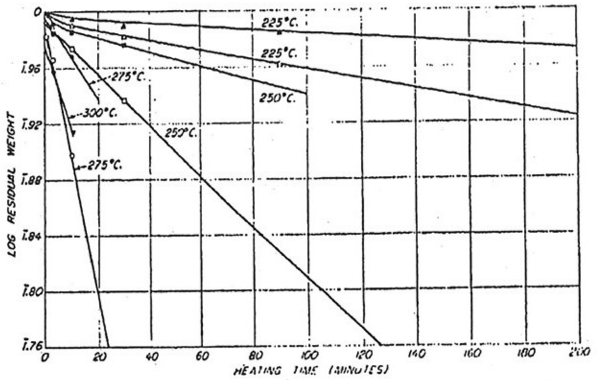

**Figure 2.** Logarithm of residual weight *vs.* time of heating of rotary-cut Sitka spruce veneer $^{1}/_{16}$ inch thick, $5^{7}/_{8}$ inches long, and 1 inch wide (*21*)

Open symbols, oven heated
Shaded symbols, heated beneath surface of molten metal

The graphs show that a loss in modulus of rupture of only about 10% would be expected in 100 years at 20° C. when air circulates freely around the specimens. Under the same conditions, a loss in work to maximum load of about 40% would be expected. In the case of limited access of air, as would prevail for larger timbers, a loss of only 3% in modulus of rupture and 12% in work to maximum load would be expected in 100 years.

Under saturated atmosphere conditions, however, a loss of about 40% in modulus of rupture and 80% in work to maximum load would be expected in 1 year at 20° C. from an extrapolation of the data of Figure 5.

Extrapolation of the logarithm of time-temperature graphs to room temperature gives reasonable weight and strength loss values for dry wood (Figure 4). The values are excessively high, however, for steamed wood. It is obvious that such strength losses do not occur in the heartwood (the dead part) of growing

**Table III. Activation Energies of Wood and Cellulose Calculated from Thermal Degradation Data**

| Material | Heating Condition | Property Loss Measured | Time Range | Temp.-Range, ° C. | Activation Energy, Cal./Mole | Log $K$ at 150° C., Hours |
|---|---|---|---|---|---|---|
| Coniferous wood | | | | | | |
| Southern and white pine, Sitka spruce and Douglas fir sticks and Sitka spruce veneer | Oven | Weight | 1 hr.–2.4 yr. | 93.5–250 | 29,500 | −3.9 |
| Sitka spruce veneer | Under molten metal | Weight | 1 min.–6 days | 167 –300 | 29,800 | −4.4 |
| Sitka spruce sticks | Oven | Modulus of rupture | 1 hr.–188 days | 102 –177 | 26,600 | −2.9 |
| | Oven | Work to max. load | 1 hr.–188 days | 102 –177 | 28,600 | −2.3 |
| Douglas fir sawdust | Oven | Weight | 16 hr.–64 days | 110 –220 | 25,000 | −4.0 |
| α-Cellulose from Douglas fir | Oven | Weight | 16 hr.–64 days | 110 –220 | 26,000 | −4.1 |
| Hemicellulose from Douglas fir | Oven | Weight | 2 hr.–64 days | 110 –220 | 20,700 | −3.4 |
| Lignin from Douglas fir | Oven | Weight | 16 hr.–64 days | 110 –220 | 23,000 | −4.3 |
| Tire cord | | | | | | |
| Cotton | Oven, 40% relative humidity | Breaking strength | 120–1000 hr. | 110 –150 | 30,500 | −2.3 |
| Rayon | | Breaking strength | 80–1600 hr. | 110 –150 | 29,500 | −2.2 |
| Paper | | | | | | |
| 100% rag | Oven | Folding endurance | 72–860 hr. | 60 –120 | 19,100 | −1.2 |
| 50% rag, 50% sulfite | Oven | Folding endurance | 72–860 hr. | 60 –120 | 19,300 | −1.0 |
| 30% rag, 70% sulfite | Oven | Folding endurance | 72–860 hr. | 60 –120 | 19,900 | −0.8 |
| 100% sulfite | Oven | Folding endurance | 72–860 hr. | 60 –120 | 19,700 | 0 |
| Coniferous wood | | | | | | |
| Southern and white pine, Sitka spruce, and Douglas fir sticks | Steam | Weight | 1–200 hr. | 121 –177 | 15,800 | −2.7 |
| Sitka spruce sticks | Steam | Modulus of rupture | 1–32 hr. | 121 –177 | 16,300 | −1.75 |
| | | Work to max. load | 1–32 hr. | 121 –177 | 17,300 | −1.45 |
| Yarn | | | | | | |
| Cotton "purified" | Oven, 95% absolute humidity | Breaking strength | 1.5–6 hr. | 105 –150 | 15,300 | −1.45 |
| Viscose rayon | | Breaking strength | 1.5–6 hr. | 105 –150 | 15,900 | −1.20 |

trees, where the moisture content may exceed the fiber saturation point for hundreds of years. Piling immersed in water has also retained sufficient strength to be serviceable for periods up to 50 years. It thus appears unsafe to extrapolate the steaming data to room temperature, as was done for heating under dry conditions. This is not surprising, as the concentration of water vapor present drops off very rapidly with a decrease in temperature. Temperature would thus not only affect the reaction rate in the normal way, but also the vapor concentration.

The degradative effect due to kiln drying of softwoods can be estimated by considering the drying conditions to be made up of a wet period and a dry period. Of course, in reality there is a gradual transition from one to the other. For example, consider a kiln load of a softwood heated for 2 days at 160° F. (71.1° C.) and 5 days at 200° F. (93.3° C.) and that the wood is very wet for only the first 2 days. The loss in modulus of rupture occurring during the 2 days while free moisture is still present would be only about 2.5% and the loss in work to maximum load would be about 5%, even on the basis of the simple extrapolation of the data of Figure 5 which has been shown to give high values. The loss during the 5-day period where the moisture content is low would be about 0.5% for the modulus of rupture and 2.0% for the work to maximum load.

Hardwoods (broad-leaf species) are dried at lower temperatures but for longer periods of time to avoid mechanical degradation. A typical long schedule might be 5 days at 130° F. (54.4° C.) for the first wet period and 20 days at 160° F. (71.1° C.) for the dry period. If for the moment the thermal degradation of hardwoods is assumed to be the same as for softwoods, then the loss of modulus of rupture during the wet period under the most severe direct extrapolation conditions would be 2.5% and the loss in work to maximum load would be 5.0%. During the dry period the loss in modulus of rupture would be only 0.1% and in the work to maximum load, 0.4%. Hardwoods, however, show a greater thermal degradation than softwoods, presumably because of the higher hemicellulose and lower lignin contents and hydrolytic effects due to the fact that greater amounts of acetic acid are formed. The values should not be more than twice as great. Even if the values are doubled, they would not be seriously high.

Conrad, Tripp, and Trinidad (*1*) give data for the loss in breaking strength of both cotton and rayon tire cord that occurs upon oven heating at 40% relative humidity. Table III shows that the activation energies and the log $K$ values are very similar.

Wiegerink (*29*) gives data for the thermal degradation of various yarns when heated at high, low, and intermediate absolute humidities. He unfortunately carried the degradation sufficiently far to calculate activation energies with any degree of accuracy only in the case of heating at high absolute humidities. Values calculated from these data for a "purified" cotton and a viscose rayon are given in Table III. The values are similar to those for wood heated in steam, and the log $K$ values at 150° C. are also in reasonably good agreement with the values for wood determined from strength test data.

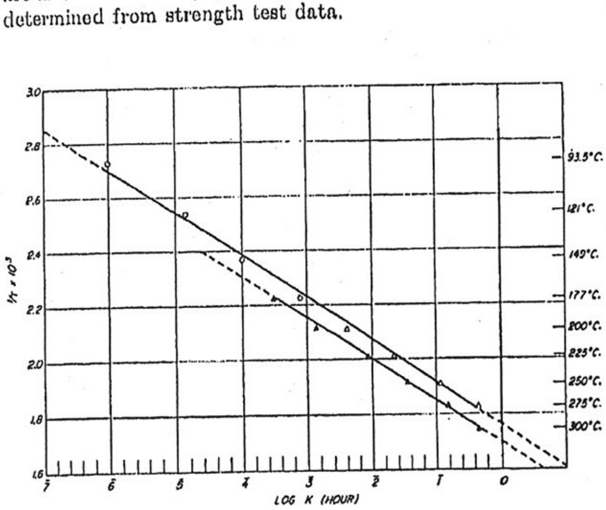

Figure 3. Reciprocal of absolute temperature *vs.* logarithm of $K$

$K$ Slope of straight lines of Figures 1 and 2 per unit of time in hours
○, △ Heated in oven
▲ Heated beneath surface of molten metal
○ (*9–12*)
△, ▲ (*21*)

Rasch (*17*) gives data for the loss in folding endurance of paper made from 100% rag and 100% sulfite pulp and mixtures thereof when heated in an oven for 72 hours at four different temperatures. Activation energies were calculated from linear plots of the logarithm of the residual folding endurance plotted through the single time point and the origin. The values are given in Table III. Activation energies for the four papers are practically the same, but the log $K$ values at 150° C. increase in going from 100% rag paper to 100% sulfite paper, indicating that the sulfite paper is about 12.5 times more sensitive to heat degradation than the paper made from rag stock.

$e^{-2,7} \approx$

$e^{-1,75} \approx$

$e^{-1,45} \approx$

$R = 8,$

$T \approx 150°$

$E_a = 1000$

$= 800$

$\frac{E_a}{RT} \approx 2$

$E_a \approx 100$

$\approx 400$

$\frac{E_a}{RT} \approx 1$

$e^{-1} \approx$

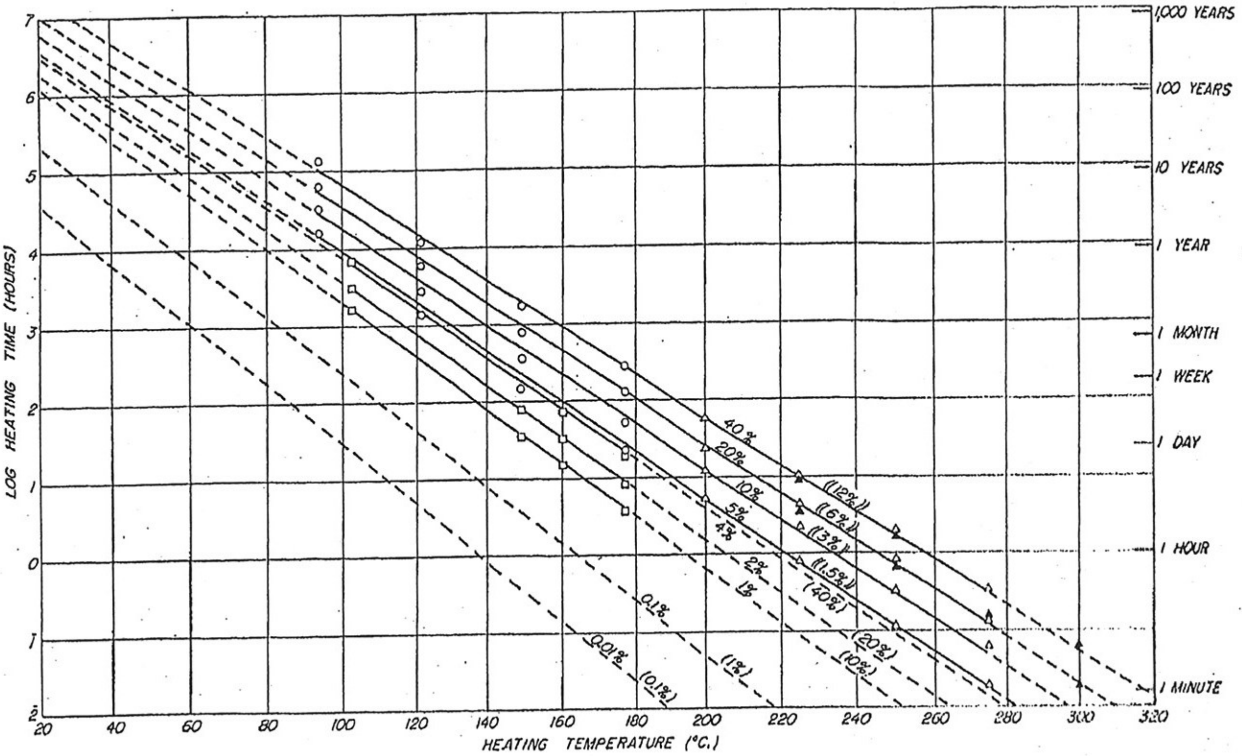

**Figure 4.** Logarithm of heating time *vs.* temperature to attain various degrees of degradation of wood

Open symbols, heating in oven
Shaded symbols, heating beneath surface of molten metal
○ Weight loss (*9–12*)
△, ▲ Weight loss (*21*)
□ Modulus of rupture loss (*9–12*)
No parentheses, weight loss on oven heating
Double parentheses, weight loss on heating beneath surface of molten metal
Single parentheses, modulus of rupture loss on oven heating

Figure 6 gives the data of Rasch (*17*) plotted as the log of time against the temperature for different losses in folding endurance extrapolated to room temperature. Richter (*19*) determined the loss in folding endurance of similar rag paper at 38° and 100° C. These values are also plotted in Figure 6. The observed losses in folding endurance are very close to the values predicted by the straight lines. It thus appears possible by such a plot to estimate the loss of folding endurance with time at room temperatures. Such a plot would be needed for each type of purity of paper. In the case of this particular paper, it would be expected to lose 24% of its folding endurance on storing with free access of dry air at 20° C. for 1.4 years. Degradation due to storage in large rolls should, of course, be appreciably less.

## CONCLUSIONS

Thermal degradation of wood is greater in the presence of air than in its absence, because of oxidation by atmospheric oxygen.

It is greater in a closed system, where acids formed build up in concentration and hence can catalyze hydrolysis, as compared to a system where the volatile products can escape but air cannot enter.

Thermal degradation for all the materials tested follows a first-order reaction.

Thermal degradation is greater under steaming than under dry heating conditions. The activation energies for the degradation reactions are about half as large as under dry heating conditions.

The activation energies for the thermal degradation of dry wood are similar to the values for the major components of wood. Hemicellulose from Douglas fir, however, degrades about four times as fast as the wood itself and α-cellulose isolated from the wood. Isolated lignin, on the other hand, degrades only half as fast as the wood.

Activation energies calculated from weight loss and strength

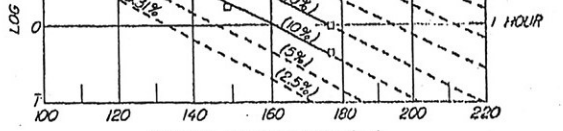

**Figure 5.** Logarithm of heating time in steam *vs.* temperature to attain various degrees of degradation of wood

○ Data of MacLean for loss of weight of softwood specimens similar to those of Figure 1
□ Data of MacLean for loss of modulus of rupture of Sitka spruce specimens similar to those of Figure 1
No parentheses, weight loss
Parentheses, modulus of rupture loss

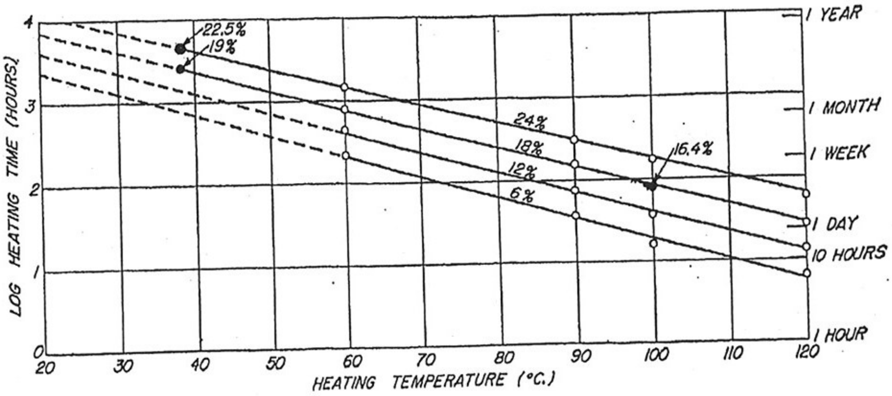

Figure 6. Logarithm of heating time in oven *vs.* temperature to attain various losses in folding endurance of 100% rag paper

○ **Data of Rasch**
● **Data of Richter**

loss data are similar. Under both dry heating and steaming conditions, the loss in modulus of rupture is about 10 times the loss in weight, and the loss in work to maximum load is still greater.

Plots of the logarithm of the heating time against the heating temperature to attain different degrees of degradation under dry heating conditions when extrapolated to room temperature give an idea of the rate of natural degradation of dry wood. Only the work to maximum load for softwoods is sufficiently affected to be readily detected after 100 years' storage at room temperature under dry conditions. This extrapolation procedure cannot be applied to wood exposed to a moisture-saturated atmosphere.

The thermal degradation of wood under normal kiln-drying conditions is fortunately negligible.

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# Correction

In the article entitled, "Densities of Ternary System Nitric Acid-Dinitrogen Tetroxide-Water" [Robert W. Sprague and Ethel Kaufman, IND. ENG. CHEM. 47, 458 (1955)], the values of calculated density and error in Table I (page 460) for white acids correspond to the following third-order expression for $\alpha$:

$$\alpha = [-0.23313 + 2.94588\,R - 21.06723\,R^2 + 78.76576\,R^3]\,O$$

Corrected Table I, corresponding to the second-order expression for $\alpha$, is presented herewith.

**Author's Note:** The interpolation formula presented in the paper has been found awkward to use, and, in addition, the interpolated value depends on the interval $\rho_0 - \rho_2$ chosen. A simpler method (and possibly a more accurate method) is to use a correction for the density calculated from the general equation in the region of interpolation (less than 4.5% $N_2O_4$); the correction is obtained by assuming the error to vary linearly along a line of constant water from zero at 4.5% $N_2O_4$ to the value shown in Table I above at 0% $N_2O_4$. For example, at 35° C. for the composition 1% $N_2O_4$, 4% $H_2O$, 95% $HNO_3$ the calculated density is 1.4753; the error from the above Table I is 0.002; the correction is $\left(\frac{4.5 - 1}{4.5}\right)(-0.002) = -0.00155$; then the corrected density is 1.4738.

Table I. Error Table for Calculated Values of Density at 35° C. of White Acids, No Dinitrogen Tetroxide

| % $H_2O$ | Density, Calculated | Density, Observed | Error |
|---|---|---|---|
| 0 | 1.4830 | 1.4830 | 0.00 |
| 2 | 1.4764 | 1.4729 | 0.0035 |
| 4 | 1.4699 | 1.4679 | 0.0020 |
| 6 | 1.4633 | 1.4641 | −0.0008 |
| 8 | 1.4568 | 1.4599 | −0.0031 |
| 10 | 1.4503 | 1.4552 | −0.0049 |
| 12 | 1.4437 | 1.4502 | −0.0065 |
| 14 | 1.4372 | 1.4450 | −0.0078 |
| 16 | 1.4306 | 1.4397 | −0.0091 |
| 18 | 1.4241 | 1.4337 | −0.0096 |
| 20 | 1.4175 | 1.4268 | −0.0093 |

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