SALT MIGRATION AND SHRINKAGE IN FIRE-RETARDANT-TREATED LUMBER*

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INTRODUCTION

There are definite indications of increased interest in the use of fire-retardant-treated (FRT) lumber. Consequently more attention should be given to both processing techniques and the effects of this type of chemical treatment upon the physical properties of the material.

The Ottawa Laboratory, FPRB conducted drying studies on 2" thick white pine that was treated with fire-retardant salts to retentions of about 3 to 10.5 lbs. per cubic foot (3). They found that a chemical deposit appeared on the surface of the wood from some of the charges; the higher the chemical retention the greater the surface deposit. They also found that no deposit occurred at slow rates of drying and that the heaviest deposits occurred when the lumber was dried quickly.

In a more recent publication, Brazier and Laidlaw (1) report that strength losses of between 10 and 30 percent have been reported for FRT lumber that was kiln dried at temperatures of about 65°C. They point out that the extent to which each strength property is affected may depend "not only on the treatment and any temperature effect but also on the type of timber treated and the amount and distribution of the absorbed salt."

A review of the literature revealed very little information on the shrinkage behavior of FRT lumber. There is considerable information on the bulking effect of water-soluble salts, but an absence of shrinkage values for the treated lumber.

There is a need for considerable research on FRT lumber. Strength losses due to treatment and processing may indeed be greater than the 10 percent reduction in design stresses that is normally employed. As pointed out above, there is also evidence that the water-soluble salts do migrate to the surface of the lumber during drying and it is further recognized that the pattern of salt distribution, which is at least in part a result of migration during drying, may in fact significantly influence the final strength properties. In light of these kinds of

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concerns, it was decided to conduct the research that is being reported on in this paper.[1]

EXPERIMENTAL OBJECTIVES

1. To describe the migration of a fire-retardant salt in softwood structural lumber during kiln drying.
2. To quantify the effect of drying rate upon the migration of the salt.
3. To compare the shrinkage of fire-retardant-treated lumber with that of untreated lumber under room temperature drying conditions.

EXPERIMENTAL MATERIALS AND PROCEDURES

Sample preparation

The lumber employed in the study was obtained from a pressure treating company located in Minneapolis. A total of 256 pieces of S4S kiln dried coast type hem-fir 2" x 6" were delivered to our laboratory. Forty-eight of the boards were 12' long and the remaining 208 were 10' long. The lumber was NLGA grade stamped No. 1 and 2.

The study involved a total of four kiln runs. Twelve of the 12' long boards were allocated to each kiln run along with 52 of the 10' boards.

The 12' long boards were processed as shown in Figure 1. Initially a 3" end trim was taken. Following this a 15" piece was removed for eventual vacuum-pressure treatment with plain water and use in the shrinkage study. Next came a 1" cross section for determination of specific gravity and moisture content prior to salt treatment. The remainder of the board was then returned to the company and treated by their standard commercial vacuum-pressure process.

Following return of the boards to our laboratory each board was weighed and further processed as shown in Figure 1. A 2" end trim was taken to nullify the end grain effect on salt pickup. Next came a 15" salt treated section for use in the shrinkage study and following this a 2" cross section for use in the before-drying salt analysis. The remaining 8'8" board was end-coated with phenolic resin plus an interspersed layer of aluminum foil, and measured in width and thickness prior to kiln drying.

Following drying the board was end trimmed before cutting the 2" cross section that was used for the after-drying salt analysis. Finally, the board was weighed and again measured in width and thickness.

The 10' boards allocated to each kiln run were processed as follows: A moisture content and specific gravity section was taken from each board after end trimming. Next a 15" length was removed for use in developing the procedure for the vacuum-pressure treatment to be carried out in the lab. The shortened boards were then weighed

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[1] The authors greatly appreciate the suggestions of Carlton Holmes of the U. S. F. P. L., Madison, Wisconsin regarding needed research in the area of drying FRT lumber.

and returned to the company for commercial treatment along with the 48 boards described earlier.[2] Upon return to the lab each board was weighed and end-coated prior to kiln drying. At the end of kiln drying each of these boards was also weighed.

Kiln schedules

Table 1 summarizes the kiln drying conditions. The slow kiln schedule employed a maximum dry bulb temperature of 140°F. The "fast" schedule raised the d. b. t. in two steps to a final value of 160°F. The other principal difference in the two schedules was the lower relative humidity conditions employed in the "fast" schedule.

All runs employed 3/4" stickers with an air velocity of 5 to 6 hundred f. p. m. A load cell was placed under the front beam of the kiln car. The weight determined by the load cell was displayed on a digital readout panel meter, allowing an estimate to be made of the current average moisture content of the entire charge. The estimated ovendry weight of the charge was based upon the measured volume of the lumber and its average specific gravity. The estimated salt content of the charge was based upon the weight of solution picked up plus its stated concentration.

Salt analysis

Figure 2 illustrates how each salt analysis section was subdivided. About 3/4" was trimmed off each end of the cross section and the remainder was reduced to slices, with each surface and intermediate slice being 7/32" thick and the core slice 15/32" thick. The two surface slices were combined for salt analysis as were the two intermediate slices.

The slices were reduced to slivers of about rectangular cross section through the use of a wood chisel. These slivers were fed into a floor model hammermill and the output was then put through a bench mounted hammermill.

The hammermilled samples were stored in open French bottles at 9% E. M. C. conditions until three consecutive weighings established that constant weight was achieved. Samples were not analyzed for salt content until they attained constant weight.

The procedure required for salt analysis was more complicated than what was originally envisioned. The first approach envisioned was to ovendry the material, extract it, and then ovendry the extracted sample to obtain the salt content by weight difference. We quickly discovered that this approach was inadequate since it was impossible to obtain a constant ovendry weight for the salt treated material. There was an apparent effect of the salt at the ovendrying temperature that resulted in a breakdown of the wood substance and a continuous loss in sample weight. In addition, the use of water to extract the salt from the hammermilled samples caused the removal of water-soluble extractives. This also confounded the attempt to

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[2] The commercial treatment was atypical. Three hours into the eight hour pressure cycle the pump broke down. As a substitute for the remaining five hours of pressure treatment, the lumber was allowed to soak for two days in the solution.

arrive at a salt quantity through the principle of weight differences.

The fire retardant salt used in this study consists of 60% ammonium sulfate [$(NH_4)_2SO_4$] by weight. Consequently we decided to utilize the standard quantitative procedure for the gravimetric determination of sulfate in a soluble salt mixture. Inherent in electing this option to evaluate total salt migration is the assumption the other soluble salts have the same migration properties as the ammonium sulfate.

The salt analysis proceeded as follows: The equilibrated sample, normally in the range of 15 to 20 grams, was weighed to the nearest .0001 gram. It was then put in a 600 ml beaker and 400 ml of reverse osmosis distilled water was added. The beaker was allowed to sit at room temperature for one hour and its contents were stirred for 30 seconds every 10 minutes. One hundred ml of supernatant was collected by filtration through No. 1 Whatman filter paper.

Precisely 50 ml of the supernatant was pipetted into a 250 ml beaker and 50 ml of distilled water was added. In accordance with the requirements of this analytical procedure, 2 ml of 5 N HCl was added to insure an acidic medium. One hundred ml of .0721 N $BaCl_2$ was pipetted into a 100 ml beaker. The beaker containing the fire-retardant salt solution, and the $BaCl_2$ beaker were then heated to boiling on a hot plate. The contents of the two beakers were added together and this solution was stirred for 3 minutes and then left undisturbed for 15 minutes to permit the $BaSO_4$ precipitate to settle. Several drops of $BaCl_2$ solution were added to insure that all the sulfate ions had precipitated as $BaSO_4$.

The $BaSO_4$ precipitate was vacuum filtered into a sintered porcelain crucible, and the crucible was washed three times with distilled water. Several drops of water were collected from the third wash, to which were added a drop of 3% $AgNO_3$. The production of a milky precipitate indicated the need for additional washing to completely remove excess $BaCl_2$ from the crucible. The crucible was then fired in a 600°C muffle furnace for one hour and then transferred to a desiccator to cool for 20 minutes before weighing it to the nearest .0001 gram.

The amount of ammonium sulfate present in the 50 ml sample was determined as follows:

$$\text{Weight of } (NH_4)\, SO_4 = \frac{132.08 \text{ grams/Mole}}{233.40 \text{ grams/Mole}} \times \text{Wt. of } BaSO_4$$

This weight of ammonium sulfate was multiplied by the factor of 8 to obtain the total amount that was extracted from the sample.

Collection of shrinkage data

The samples employed in the shrinkage study were board sections 15" in length. The salt treated samples were cut from the boards immediately after they were returned from the commercial treater. End-matched pieces had been cut from the boards prior to commercial treatment, and these sections were vacuum-pressure treated with city water at the laboratory.

There were 48 sets of these end-matched samples, all of which were end-coated with two layers of phenolic resin plus an interspersed layer of aluminum foil. The 48 sets were placed on racks in a walk-in environment room maintained at $80^{\circ}F$. The samples were allowed to equilibrate with relative humidities of 80%, 64%, and 36%. At each equilibration all 96 samples were weighed and measured in both width and thickness. Equilibration was determined by randomly selecting 4 controls and 4 salt treated sections for periodic weighings. Equilibration was defined as the condition in which none of the 8 samples deviated more than $\pm .1$ gram in three successive weighings spaced five days apart.

The moisture content of each control sample was estimated for each equilibration. This was accomplished by ovendrying each control sample after completing the equilibration at 26% relative humidity.

RESULTS AND DISCUSSION

Analysis of salt data

The results of the salt analysis are summarized in Figure 3, where the weight of $(NH_4) SO_4$ as a percent of the total weight of the sample is plotted for shell, intermediate, and core slices for before and after kiln drying.

The shell slices show a pronounced increase in salt concentration following kiln drying while the core slices show a decrease. Both of these features are to be expected with a movement of the salts from the interior toward the surface during the drying operation.

The intermediate slices remained more closely the same for before and after drying than did either the surface or core slices. This could be logically explained on the basis that the salt which migrated from the intermediate slices into the shell slices was replaced by the salt that migrated from the core slice toward the surface. Consequently the bar graphs are interpreted to mean that a migration of the salt from the interior toward the surface did in fact occur during kiln drying.

There is, however, an obvious difficulty with the bar graphs. It is apparent that a reasonable material balance is not possible. There is over a one percent increase for the shell after kiln drying. If all of this is due to migration there should be a correspondingly large decrease in average salt concentration for the intermediate and core slices, perhaps especially for the core slices. The data in Figure 3 suggests that salt was manufactured during kiln drying. This, of course, is not reasonable. It is also doubtful that the problem can be explained solely on the basis of variation resulting from the necessity of evaluating salt concentration for before and after drying on different cross sections.

We subjected our procedure for salt analysis to critical review and isolated a factor that may be responsible for confounding the salt analysis results. The slices for before kiln drying were processed through the hammermills without the benefit of drying. The slices for after drying, on the other hand, had moisture contents in accordance with the final board values achieved in the kiln. We suspect that when the wet, tough material was hammermilled it did not break down in the same way as did the kiln dried material. Conceivably the before-drying fibers remained more intact, perhaps individually or in bundles with the salt more encapsulated. As a consequence the standardized water extraction process may not have been capable of completely extracting the salt from the before-drying sample, which would suggest the manufacture of salt during drying. We are currently examining this possibility.

In retrospect it is obvious that equilibrating the before-and-after-drying slices to the same moisture content before hammermilling would have been preferable. In addition it might be advisable to also subject the before-drying slices to an amount of heating approximately equivalent to what the after-drying slices endured during kiln drying.

The salt analysis data was obtained from a total of 12 boards; six from kiln run No. 3 and six from kiln run No. 4. Due to technical problems with the load cell in runs No. 1 and 2, the lumber was over dried. Consequently the use of data from these two runs in this report is quite minimal.

A statistical test for a possible difference in salt migration for runs 3 and 4 was performed. This was non-significant. We do not consider this totally conclusive, however, in view of the difficulty described earlier in regard to running a material balance on salt for before and after drying. Therefore the possible influence of drying rate upon the extent of salt migration may exist but gone undetected in this experiment.

Analysis of shrinkage data

Figures 4 and 5 summarize the shrinkage data that was obtained with the 15" long boards at a temperature of 80°F. The shrinkage values are plotted as a function of the average moisture content of the untreated samples, i. e., the samples that were vacuum-pressure impregnated with plain water. It should also be pointed out that both the radial and tangential shrinkage data were obtained in the thickness direction of the board. This means that the radial shrinkage data was obtained on 19 pairs of end-matched flatsawn boards, and the tangential shrinkage data came from the 15 end-matched pairs of quartersawn boards. This was done in order to minimize the effect of drying stresses and sets upon the shrinkage results. The measuring points were also far enough removed from the edge of the board to avoid the influence of the edge-drying effect upon the measurement.

Both figures show a significant reduction in shrinkage due to the salt treatment, and both treated and untreated show a straight line relationship between shrinkage and moisture content. The reduction for both the radial and tangential directions is almost 2% shrinkage over the range of moisture content investigated.

At the highest E. M. C. condition of about 17%, there was a definite swelling effect for the treated pieces in both the radial and tangential directions. This illustrates the high bulking efficiency of the salts as the pieces were slowly dried at 80°F. The shrinkage for the parent boards that were dried in the kiln are indicated by the K plot in each figure. The shrinkage reductions in kiln drying were less than what was obtained at 80°F. This is no doubt explained by the fact that at 80°F and the 5 weeks required for equilibration to the 83% relative humidity, there was an extended duration for salt solution to be present in the cell lumens and for salt to diffuse from the lumen into the cell walls.

The significant reduction in shrinkage during kiln drying of over one percent indicates the opportunity for the use of more severe drying conditions on salt treated lumber. This benefit may be partially offset, however, by the fact that there is a greater tendency for degrade to develop in the drying of treated lumber than in the drying of comparable green lumber (2).

Moisture content of FRT boards

Figure 6 gives the distribution of the estimated moisture content of 40 boards following commercial treatment. The moisture contents are based on the water to wood weight for each board, i. e., the estimated salt weight has been subtracted out of the board weight that was obtained at the end of drying. Due to the occurrence of some drying between treatment and the obtaining of board weights, the absolute moisture contents may be somewhat less than what could be expected. The distribution pattern of these moisture contents, however, is probably typical for commercially treated hem-fir.

A significant feature is that almost 50% of the boards had after-treatment moisture contents in the 30 to 50% range. This is well below the range of 90 to 120% moisture content that McKay (2) gives for coastal western hemlock treated with waterborne wood preservatives, but perhaps this is due to the much higher salt concentration for the fire-retardant-salt solution.

Reference to Table 2, which relates treated moisture content to end-of-drying moisture content for these 48 boards, shows the potential for ovendrying this category. Even in run 3, which was dried to an average charge moisture content of 14%, this category averaged about 7%. No attempt was made to quantify degrade in this study, but casual observation illustrated the tendency for significant cupping and checking in the ovendried boards. Possibly such boards would also experience a greater-than-average reduction in strength due to kiln drying.

Further inspection of Table 2 suggests the desirability of segregating the treated pieces for kiln drying. It seems apparent that if severe overdrying of the low moisture content pieces is to be avoided, the charge should not be dried to an overall average of less than 15%. Under this condition, boards with an initial moisture content up to about 100% should meet the 19% requirement. Boards greatly above 100% probably would not meet the requirement. Perhaps a light and heavy segregation is in order, and it would be relatively easy to do this strictly on a board weight basis. This is certainly not perfect because of concurrent specific gravity variation, but it would go a long way toward solving the problems of overdried lumber and uneconomical kiln drying. Due to the increased use of FRT lumber and the critical requirement of high product quality, improved kiln drying procedures are in order.

CONCLUSIONS

This study provides evidence that fire retardant salts migrate from the interior of the piece of lumber toward the surface during kiln drying. However, due to experimental difficulties it was not possible to precisely quantify the amount of migration or to definitely establish if the amount of migration was influenced by the rate of drying.

A comparison of the shrinkages of FRT boards and control boards showed that treated boards shrunk about one-half as much as the control boards under room temperature drying conditions. The reduction in shrinkage was about one-half as large for the matched treated boards dried in the kiln.

There is a large variation in the after-treatment moisture content of a hem-fir sort. This variation complicates the kiln drying since in attempting to dry all boards in the charge to 19% or less, the lower moisture content boards are severely overdried with accompanying warp and degrade. Sorting of the treated boards into light and heavy segregations for more efficient kiln drying is suggested.

Much additional research should be done on the important parameters of kiln drying FRT lumber. In particular, an improved experimental procedure should be developed for analyzing the amount of salt migration and its interaction with the strength properties of FRT lumber. Also, kiln drying procedures should be developed that utilize a simple sorting procedure for FRT lumber so as to improve drying efficiency and minimize its deleterious effect upon the strength and final grade of the lumber.

LITERATURE CITED

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Table 1. Kiln Schedules Employed

Slow Kiln Schedule

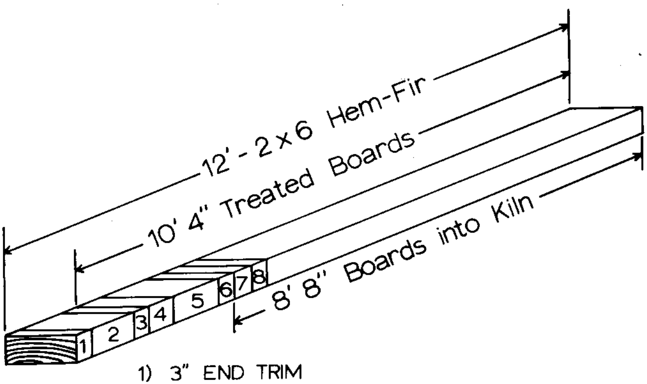
Factor	Moisture Content		
	Green to 30%	30% to 20%	20% to Final
Dry Bulb Temperature	140°F	140°F	140°F
Wet Bulb Temperature	125°F	115°F	100°F
Relative Humidity	64%	54%	25%
Equilibrium Moisture Content	9.6%	8.0%	4.1%

Fast Kiln Schedule

Factor	Green to 30%	30% to 20%	20% to Final
Dry Bulb Temperature	140°F	150°F	160°F
Wet Bulb Temperature	110°F	100°F	110°F
Relative Humidity	38%	28%	21%
Equilibrium Moisture Content	5.8%	4.2%	3.2%

Table 2. Summary of moisture content data by kiln run

	Run 1		Run 2		Run 3		Run 4	
Board	Treated M.C.	Dried M.C.	Treated M.C.	Dried M.C.	Treated M.C.	Dried M.C.	Treated M.C.	Dried M.C.
1	30	9	30	1	33	10	36	7
2	35	6	30	0	40	4	37	8
3	38	0	31	1	43	6	39	4
4	44	10	38	5	45	7	41	7
5	46	13	47	5	50	6	44	7
6	46	5	48	5	56	15	51	12
7	51	7	49	7	74	11	63	13
8	59	12	50	2	94	11	65	13
9	70	7	61	4	94	15	70	10
10	110	1	78	5	117	21	76	2
11	119	13	79	4	118	24	126	16
12	122	15	159	11	171	39	165	25
Averages	64	8	58	4	78	14	68	10
Standard Deviation	34	5	36	3	42	10	40	6



1) 3" END TRIM
2) 15" WATER-TREATED SHRINKAGE SAMPLE
3) 1" SPECIFIC GRAVITY & M.C. SECTION
4) 2" END TRIM
5) 15" SALT-TREATED SHRINKAGE SAMPLE
6) 2" BEFORE-DRYING SALT ANALYSIS SAMPLE
7) 2" END TRIM
8) 2" AFTER-DRYING SALT ANALYSIS SAMPLE

Figure 1. Processing procedure for each of the twelve 12' - 2" x 6" boards allocated to a kiln run.

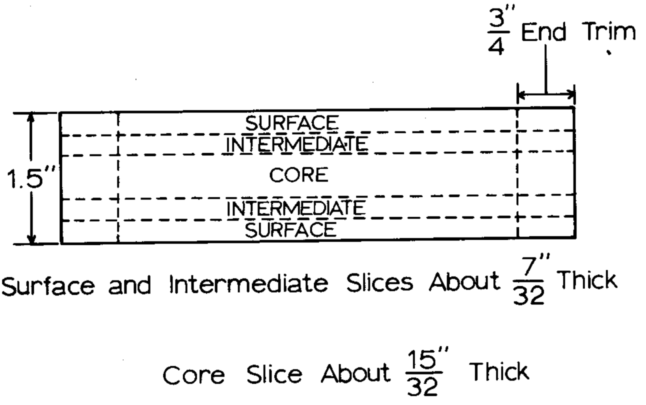


Figure 2. Preparation of salt analysis slices from cross sections cut from treated boards before and after drying.

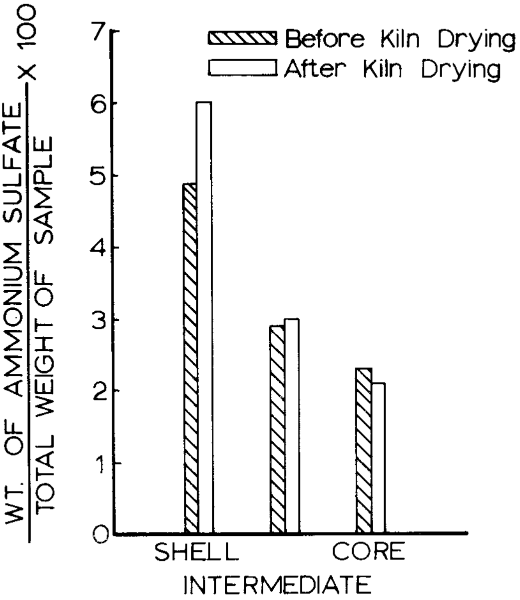


Figure 3. Salt concentrations in the shell, intermediate and core slices for before and after drying. The values plotted are the average for 12 boards.

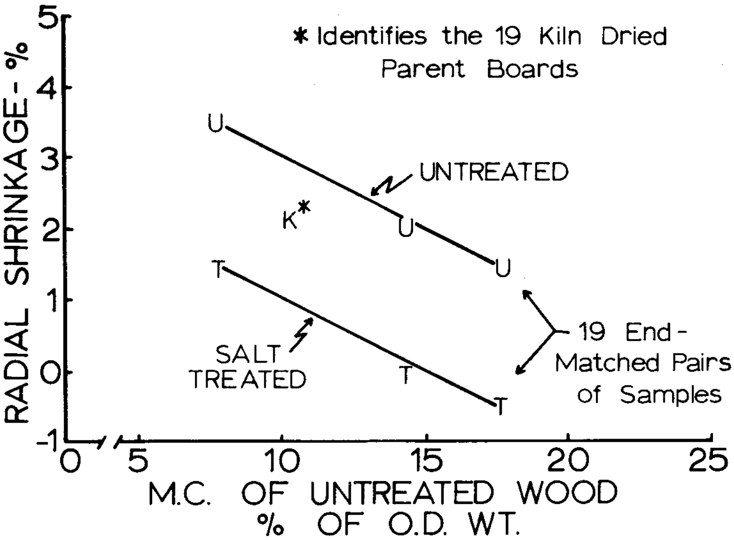


Figure 4. The effect of fire-retardant-salt treatment upon the radial shrinkage of hem-fir 2" x 6" boards. The end-matched pairs were equilibrated to 3 different EMC values at a temperature of 80°F.

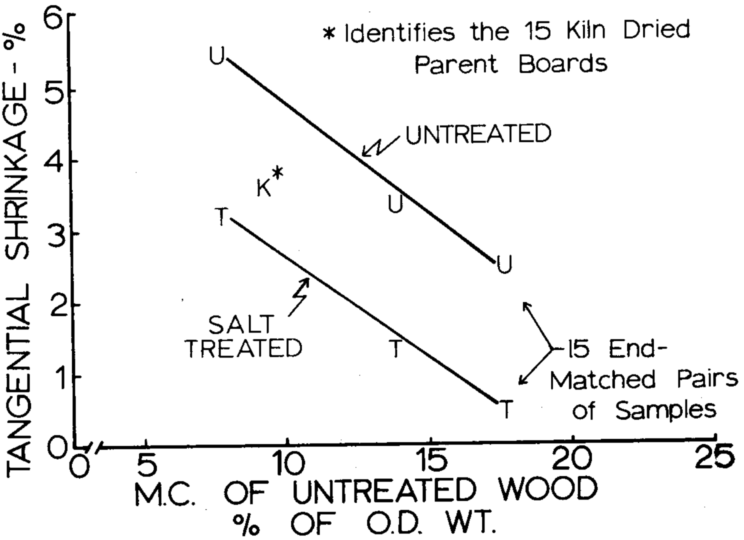


Figure 5. The effect of fire-retardant-salt treatment upon the tangential shrinkage of hem-fir 2'' x 6'' boards. The end-matched pairs were equilibrated to three different EMC values at a temperature of 80°F.

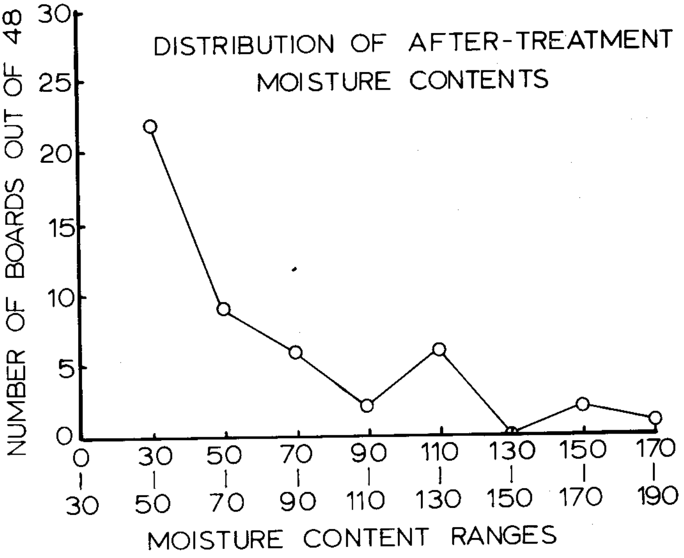


Figure 6. Distribution of after-treatment moisture contents.