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## The Variability of Extinction Coefficient with Respect to Wood Species

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THE VARIABILITY OF EXTINCTION COEFFICIENT  
WITH RESPECT TO WOOD SPECIES

By

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A Thesis Submitted in  
Partial Fulfillment of the  
Course Requirements for  
The Bachelor of Science Degree

Western Michigan University

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## ABSTRACT

The variability of extinction coefficient with respect to wood species is an important aspect to ultra-violet determination of lignin both in cooking liquor and in the wood itself. The precision with which the abundance of a solution can be measured, and therefore the lignin concentration is dependent upon the extinction coefficient.

The objective of this investigation was to analyze various woods for variability and possibly determine overall extinction coefficient averages for woods or groups of woods (hardwood/softwood) that will be more representative than currently used extinction coefficients.

The results contained within indicate that the extinction coefficients for lignin samples in solution are affected by pulping conditions. It is also clear that the wood species is a factor to consider when using ultra-violet determinations of lignin samples. There is a small but significant deviation in the extinction coefficients for differing wood species.

Key words: Ultra-violet, lignin, extinction coefficients, absorption, and spectrophotometric.

## TABLE OF CONTENTS

	Page
INTRODUCTION . . . . .	1
DISCUSSION . . . . .	2
Basic Lignin Structure . . . . .	2
Use of U.V. Spectra . . . . .	3
Beer-Lambert Law . . . . .	4
Internal Workings . . . . .	4
EXPERIMENTAL PROCEDURE . . . . .	6
Procedure . . . . .	6
DISCUSSION OF DATA . . . . .	9
Black Liquor Data . . . . .	9
Kappa Numbers . . . . .	9
Absorption Data . . . . .	10
CONCLUSIONS . . . . .	14
SUGGESTIONS FOR FURTHER WORK . . . . .	15
APPENDIX I . . . . .	16
APPENDIX II . . . . .	17
APPENDIX III . . . . .	18
APPENDIX IV . . . . .	19
APPENDIX V . . . . .	21
REFERENCES . . . . .	22

## INTRODUCTION

The use of extinction coefficients is an inherent part of any experiment that utilizes an ultra-violet spectrophotometer. The accuracy of a spectrophotometer depends upon several physical and calculated quantities. The physical variables included the length of the test cell, quality of the filters, and the substances being measured.

The calculated quantities include standard extinction coefficients and absorbancy, the former being the more important of the two. This is because absorbancies are calculated from extinction coefficients by means of an integrating graphing systems or from a mathematical approach.

The precision which extinction coefficients are measured will affect the absorbance of a particular solution. The use of an extinction coefficient where a different wood species or mix of wood species was used for the calibration could result in a loss of accuracy.

The calculation of an exact extinction coefficient is necessary to increase the accuracy and precision of an ultraviolet determination on lignin in a sample.

## DISCUSSION

### Basic Lignin Structure

Lignin is a polymer compound of phenylpropane units linked together by as many as ten different bond types.<sup>1</sup>

The three basic lignin precursors are p-coumaryl, coniferyl, and sinapyl alcohols<sup>2</sup> (See Figure 1).

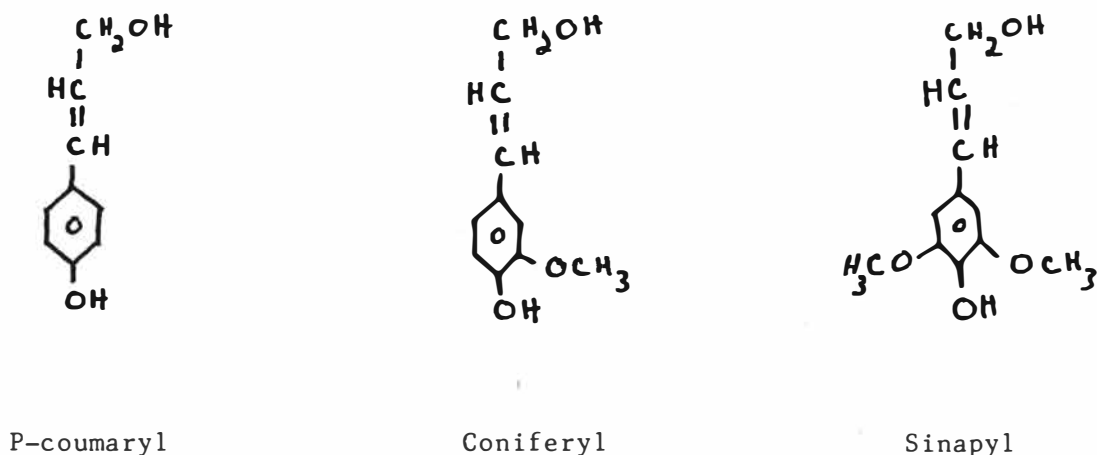


Figure 1

From acid hydrolysis studies it has been determined that of the ten bond types linking the phenyl propane units, several dominate<sup>2</sup> (See TABLE I).

% Each of Different Types of Bonds in Lignin

Bond Type	%
Arylglycerol- $\beta$ -arylether	48
Glyceraldehyde-2aryl ether	2
Non cyclic-benzyl aryl ether	6-8
Phenyl coumaran	6-12
Structures condensed in 2 or 6 position	2.5-3
Biphenyl	9.5-11
Diaryl ether	3.5-4
1,2, $\beta$ -Diaryl propane	7
$\beta$ , $\beta$ - linked structures	2

TABLE I

During pulping these bonds are broken in different proportions depending upon the severity of the cook and liquor concentrations. The cook will also be affected by bond type distribution throughout the fiber,<sup>3</sup> the more internalized bond type being broken less.

The resulting liquor is complicated further by the formation of two different residues in hardwoods (syringal and guaiacyl), where as softwoods have only one<sup>3</sup>.

It becomes obvious that the haphazard use of extinction coefficient not derived for a specific study can result in erroneous data. This can be construed from the fact that differing concentrations of residues or different wood species will cause different absorbances.

One study used four technical lignins: organosolv lignin from spruce, lignin sulfonates from spruce and beech, sulfate lignin from pine, and five analytical lignins: MWL spruce, MWL beech, TFA lignin spruce, ethanol-lignin spruce, and sulfuric extractal. These were examined and it was determined that the difference in ultraviolet spectra could not be attributed in any case specifically to the kind of lignin isolation.<sup>4</sup> This means that the residues are the absorbing fractions of the solution and the different pulping chemicals have little affect on where bonds are broken.

#### Use of U.V. Spectra

The use of ultraviolet absorption spectra as a tool in lignin investigations was studied by Kleinert and Joyce in 1957.<sup>5</sup> They found that the Beer and Lambert Law was found to be valid for low concentration solutions of lignin. They also found that the extinction coefficient does decline with increasing wavelength.<sup>5</sup> This was attributed to the strong influence of groups substituted on to the ring structure.

Later Kleinert and Joyce showed that characteristic absorption maxima in the short ultraviolet wavelength range was attributed to the resonating aromatic units.<sup>6</sup>

Finally, Kleinert and Joyce demonstrated that a spectrophotometer could be used for control of bisulphite cooking by continuous absorption measurement of the liquor circulated through a commercial digest.<sup>7</sup> From this it can be seen that ultraviolet spectrophotometry is useful as a scientific tool and also has applications as an industrial control system.

### Beer-Lambert Law

The ultraviolet absorption spectrum is an useful tool because it remains valid for concentrations at high liquor dilution.<sup>7</sup>

The basis for the Beer-Lambert Law states that the absorbance of a solution is proportional to the length of the test cell times the concentration of species in solution times a proportionality constant called the extinction coefficient.

Symbolically represented:

$$A = I_0/I = E C l$$

where A = absorbance

E = extinction coefficient l/g cm

C = concentration g/l

l = length of cell cm

The Beer-Lambert Law also requires the use of a monochromatic light source.

### Internal Workings

In the machine a light source, usually a tungsten filament, is directed through a series of collating filters. These filters allow only a monochromatic wavelength of light to pass through. By means of changing the filter speeds the wavelength may be increased. Therefore, an ultraviolet spectrophotometer can have a continuously increasing monochromatic light source.



After the light passes through the filter, it is split by a mirror or prism. One half of the light beam passes through a test cell and the absorbance is detected by a photo cell on the other side. The other one half beam passes through a reference cell where the absorbance due to the solvent is registered and electronically removed from the absorbance for the test cell.

The actual nature of  $\epsilon$  (E), the extinction coefficient is theoretical. It has the necessary units of liters/(cm g). E does have a specific number for a specific dilution which is dependent upon the chemical reactivity of the species in solution. The objective is then to find how E varies with different wood species at short wavelength (205 nanometers) where maximum absorption occurs.

## EXPERIMENTAL PROCEDURE

### Procedure

Eight species of wood were collected. Four hardwoods and four softwoods were collected. Below is a listing of the species.

Hardwoods:	Burr Oak	Softwoods:	Red Spruce
	Rock Elm		East Red Cedar
	Weeping Willow		North Pine
	Paper Birch		Balsam Fir

Each of the species collected was considered suitable or nearly suitable for conventional pulping.

Initially, samples of the woods were dried in an oven to determine percent moisture. This was done to calculate the water contribution from the wood during pulping.

It was thought that a raw or undercook and an overcook could possibly show that differing severity of cook would also affect the extinction coefficient. Therefore, softwoods were cooked at the following conditions:

Undercook: 165<sup>o</sup>C and 15% Active Alkali  
Overcook: 175<sup>o</sup>C and 22% Active Alkali

Likewise, hardwoods were cooked at:

Undercook: 155<sup>o</sup>C and 12% Active Alkali  
Overcook: 165<sup>o</sup>C and 22% Active Alkali

The white liquor used for pulping contained 98.27 g/l active alkali and 17% sulfidity. Dilution was calculated and combined with air dry chips and distilled water to form a 5:1 liquor to wood ratio. Approximate amount of wood used was 100 grams. All cooks were made in the oil bath and bomb system. All cooks were done at temperature for two hours.

The resulting black liquor was tested for available alkali, sodium sulfide and approximate active alkali. This data and discussion follow the Experimental Procedure.

The pulp was washed and dried. Kappa numbers of the pulp were later tested for and calculated. This data appears in Appendix 5.

Testing procedures followed PAPR 203 Lab procedures and T-236-05-70 for blade liquor and Kappa numbers respectively.

The extraction of the lignin from the black liquor required the precipitation of carbonates first. This was accomplished by the addition of 100 milliliters of 10%  $\text{BaCl}_2$  to a 50 milliliter aliquot of black liquor. This was volumetrically diluted to 250 milliliters and mixed vigorously. The precipitated carbonate was removed by centrifuging.

About 250 milliliters of the carbonate-free black liquor was transferred to a 500 milliliter beaker and heated to between 70 and 80°C. The pH of the liquor was then adjusted to 2-3 with the addition of 4  $\text{NH}_2\text{SO}_4$ .

This resulted in the precipitation of the lignosulfonate as a fine white material capable of being filtered from solution. The heating of the black liquor was necessary to make the precipitated lignin filterable. At room temperatures the precipitated lignin is still only a slimy colloid. At temperatures just below the melting point of the material, the lignin is collectable by filtration.

The material was collected on a Buchner. The resulting filter cake was washed with dilute acid and water. The sample was then dried at temperatures less than its melting point. This was between 70°C and 80°C.

Normal yield for 250 milliliters of carbonate-free black liquor is .25-.40 gram.

Samples of approximately .1 gram were dissolved in a 500 milliliter volu-

metric flasks with 16% NaOH that was previously heated to 65°C. The hot NaOH dissolved the lignin sample quickly and effectively. The use of .1 gram in 500 milliliters is approximately a dilution of 1 in 4000.

Testing of the lignin samples was done in silica cells on loan from Dr. B. Anderson of Western Michigan University's Chemistry Department.

The finding of a workable machine perhaps presented the most difficulty. Only one of four ultra-violet spectrophotometers was functional in the Chemistry Department. This machine had a 30% stray light. This is roughly equivalent to a 30% error in quantitative values at low wave lengths.

Finally, a machine that was operative was located in the Bio-Medical Department at W.M.U. Located in Wood Hall, the machine did not possess the wavelength scanning and recording equipment. Therefore, the choice of a single wavelength in the test area (205-210mm) was done. The value of 207mm was taken.

Testing was done on a Gilford Ultra-Violet Spectrophotometer. Two runs from each sample were done. This data was recorded and appears in the discussion.

Chemicals other than those discussed were prepared by the simple act of dissolving or diluting a given quantity to the desired strength.

## DISCUSSION OF DATA

### Black Liquor Data

Data collected for available alkali, sodium sulfide and approximate active alkali shows that the desired undercooking and overcooking was accomplished.

### Black Liquor Tests (g/l as Na<sub>2</sub>O) Average Values

	Available Alkali	Sodium Sulfide	Approximate Active Alkali
Hardwood			
Raw	9.14	1.28	9.78
Over	23.34	3.90	25.29
Softwood			
Raw	8.38	3.02	9.89
Over	19.86	3.60	21.65

On the average the values for all tests on hardwoods and softwoods were two to three times greater for the overcooked bombs. The low values for the undercooked bombs indicate the consumption of a high percentage of pulping chemicals. The excess of chemicals for the overcooked bombs indicates the extreme degree to which pulping went.

### Kappa Numbers

Kappa numbers reflect this also.

### Kappa Numbers

Hardwoods	Rawcook	Overcook	Δ in Kappa
Burr Oak	80.80	56.42	24.40
Rock Elm	67.80	48.90	18.90
Weeping Willow	71.05	44.10	26.75
Paper Birch	55.10	38.45	16.65

Kappa Numbers  
(Cont.)

Softwoods	Rawcook	Overcook	in Kappa
East Red Cedar	59.87	78.70	-18.80
Red Spruce	55.15	70.15	-15.00
North Pine	65.74	34.00	31.74
Balsam Fir	60.31	48.52	11.80

The drop in Kappa number from raw to overcooks indicates greater delignification. This was expected.

There are two values whose change was negative. This apparent mistake may be due to leaky bombs or attributable to instrument and/or operator error.

Absorption Data

Below is a listing of the final calculated extinction coefficients. Raw data and sample calculations appear in Appendix V.

North Pine	1/cm.g
raw(r)	.34
over (o)	5.436
Balsam Fir	
raw	9.758
over	3.42
Red Spruce	
raw	8.804
over	6.398
East Red Cedar	
raw	5.569
over	3.880
Weeping Willow	
raw	3.840
over	3.653
Paper Birch	
raw	.943
over	1.173
Rock Elm	
raw	1.183
over	2.108
Burr Oak	
raw	2.698
over	2.777

Literature states that extinction coefficient values ranging from 4.0-15.5 l/cm.g are to be expected.<sup>3</sup> While some values are low, they are not too far out of the acceptable range.

A sample from Westvaco (Indulin AT) was also tested. Because of the nature of the lignin, it absorbed too strongly, even at low concentrations to provide a readable absorbance. The data, as it stands, is not representative of anything. The species must be taken as paired points. This allows for the determination for cooking degree and differences due to wood species.

It is evident from a statistical determination of a single one standard deviation that the two different levels in cooking did affect the absorbant properties of the lignin in solution. The standard deviation calculated for the groups of points utilized the difference between pairs. This resulted in a standard deviation of  $\sigma = 2.385$  which indicates a difference in the absorption due to different levels of cooking. Therefore the extinction coefficient of a lignin sample in solution is affected by the severity of the pulping conditions. This can be seen in the following graph.

The next step was to correct the pairs for the differences due to the variation in cooking strength. The generation of an average difference was done by averaging each group of points (undercooked and overcooked). The resulting average undercooked value ( $\bar{x}_r$ ) minus the average overcooked value ( $\bar{x}_o$ ) resulted in an average difference. This difference was then subtracted from each point in the undercooked set to form a new group, free from variations due to the cooking conditions.

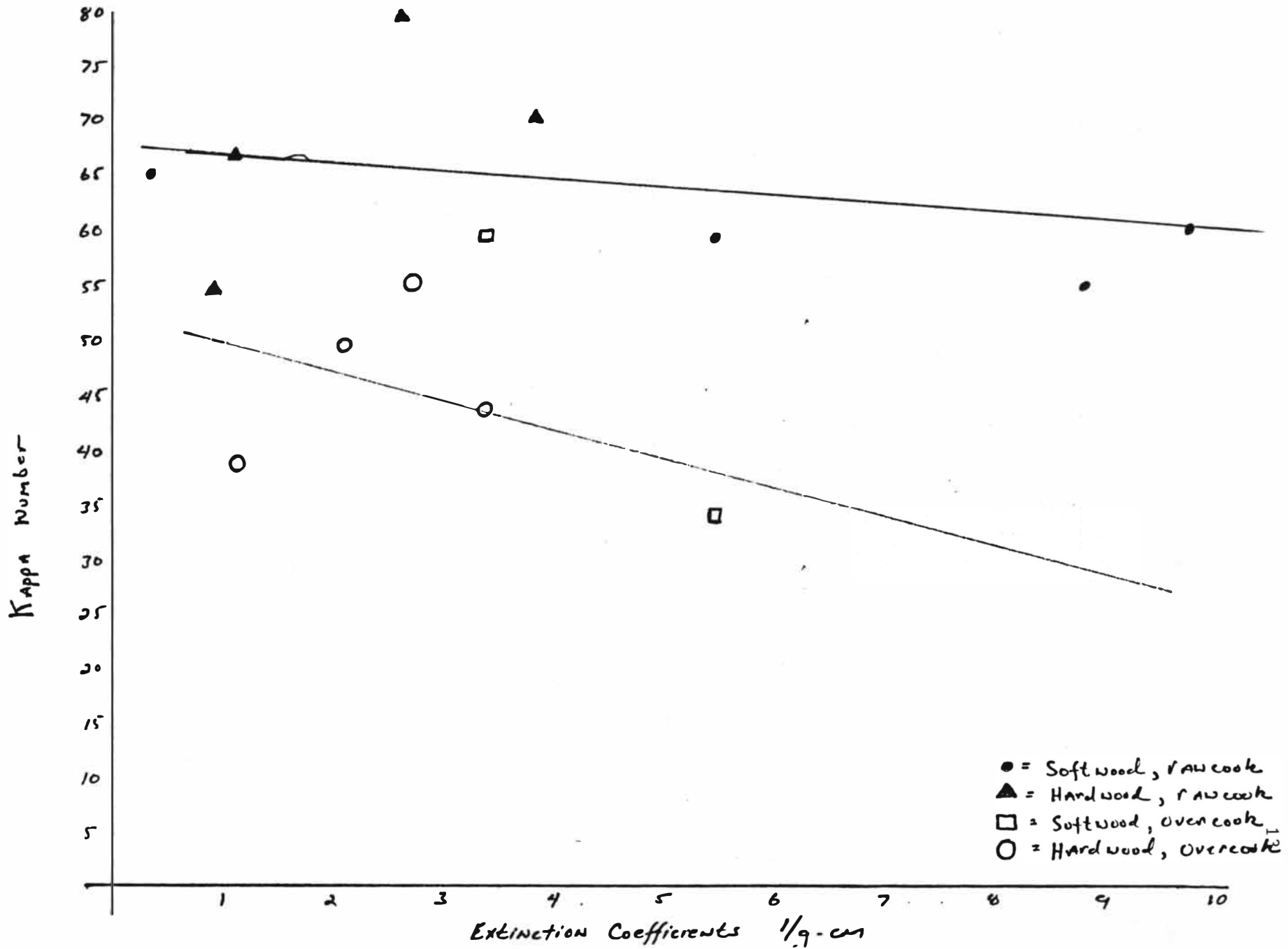
Examination of the new undercooked points and the original overcooked points could now reveal variations due to the differences involved with the wood species.

The calculated standard deviations for the new group and the original

overcooked set yielded a standard deviation of  $\sigma = 2.158$ , and a variance of  $S_o^2 = 4.66$ . These values indicate a significant variation in extinction coefficients for different woods.



Extinction Coefficients vs. Kappa No.



## CONCLUSIONS

The extent of this variation is large enough to consider when evaluating data from other ultra-violet determinations where the variation in extinction coefficient was not taken into account.

## SUGGESTIONS FOR FURTHER WORK

The extent of the variation in extinction coefficients for wood species was not quantifiable. That is the amount of variation was not calculable. This was because of the statistically small data set and the unavailability of an ultra-violet machine that scanned. The former problem was due more to time and other physical restrictions. The latter was a problem because in all probability the choice of 207mm as the test wavelength did not represent the entire group. The interest in the absorption lies at the peak absorbance. Peak shifts are not uncommon when dealing with organic substances. Therefore, any further work on a topic similar to the one contained in this paper should utilize a scanning type of machine and contain a statistically large group of points.

## APPENDIX I

### ANALYSIS OF WHITE ALKALINE COOKING LIQUOR

g/l Active Alkali	98.27 g/l as $\text{Na}_2\text{O}$
% Sulfidity	17%
g/l NaOH	26.3 g/l as $\text{Na}_2\text{O}$
g/l $\text{Na}_2\text{S}$	5.40 g/l as $\text{Na}_2\text{O}$

APPENDIX II

CHARGE CALCULATIONS PER 100 GRAMS OVEN DRIED WOOD

5:1 RATIO

$$15\% \text{ cook } \frac{1000 \times 15\%}{98.27} = 156.2 \text{ ml lignin to charge}$$

$$25\% \text{ cook } \frac{1000 \times 25\%}{98.27} = 254.4 \text{ ml lignin to charge}$$

North Pine - Air Dry Weight: 114g - O.D. Weight: 100g

15% 333.4 ml H<sub>2</sub>O added

25% 231.5 ml H<sub>2</sub>O added

East Red Cedar - Air Dry Weight: 120g - O.D. Weight: 100g

15% 327.4 ml H<sub>2</sub>O added

25% 225.0 ml H<sub>2</sub>O added

Red Spruce - Air Dry Weight: 125g - O.D. Weight: 100g

15% 322.4 ml H<sub>2</sub>O added

25% 220.6 ml H<sub>2</sub>O added

Balsam Fir - Air Dry Weight: 113g - O.D. Weight: 100g

15% 334.4 ml H<sub>2</sub>O added

25% 263.2 ml H<sub>2</sub>O added

$$12\% \text{ cook } \frac{1000 \times 12\%}{98.27} = 122.0 \text{ ml lignin to charge}$$

$$22\% \text{ cook } \frac{1000 \times 22\%}{98.27} = 223.8 \text{ ml lignin to charge}$$

Burr Oak - Air Dry Weight: 113g - O.D. Weight: 100g

12% 365.0 ml H<sub>2</sub>O added

22% 263.2 ml H<sub>2</sub>O added

Rock Elm - Air Dry Weight: 113g - O.D. Weight: 100g

12% 365.0 ml H<sub>2</sub>O added

22% 263.2 ml H<sub>2</sub>O added

Weeping Willow - Air Dry Weight: 113g - O.D. Weight: 100g

12% 365.0 ml H<sub>2</sub>O added

22% 263.2 ml H<sub>2</sub>O added

Paper Birch - Air Dry Weight: 114g - O.D. Weight: 100g

12% 364.0 ml H<sub>2</sub>O added

22% 262.2 ml H<sub>2</sub>O added

All bombs cooked two hours at temperature.

APPENDIX III

BLACK LIQUOR ANALYSIS

Hardwood - Undercooked (155°C = 12%)

	<u>Available Alkali</u>	<u>Sodium Sulfide</u>	<u>Approximate Active Alkali</u>
Burr Oak	6.625	.75	7.000
Rock Elm	7.05	1.25	7.675
Weeping Willow	6.00	1.62	6.81
Paper Birch	16.9	1.525	17.66

Hardwood - Overcooked (165°C + 22%)

	<u>Available Alkali</u>	<u>Sodium Sulfide</u>	<u>Approximate Active Alkali</u>
Burr Oak	23.7	4.025	25.7125
Rock Elm	23.65	4.05	25.675
Weeping Willow	21.1	3.775	22.9875
Paper Birch	24.9	3.8	26.8

Softwood - Undercooked (165°C + 15%)

	<u>Available Alkali</u>	<u>Sodium Sulfide</u>	<u>Approximate Active Alkali</u>
East Red Cedar	5.7	2.225	6.8125
Red Spruce	7.4	1.9	8.35
Northern Pine	11.8	4.1	13.85
Balsam Fir	8.65	3.85	10.575

Softwood - Overcooked (175°C + 25%)

	<u>Available Alkali</u>	<u>Sodium Sulfide</u>	<u>Approximate Active Alkali</u>
East Red Cedar	19.75	2.575	21.0375
Red Spruce	18.1	2.65	19.425
Northern Pine	22.7	4.65	25.025
Balsam fir	18.9	4.475	21.1375

APPENDIX IV

KAPPA NUMBERS - Blank 57.7 ml

Hardwoods - Undercooked (155°C + 12%)

			<u>"P"</u>	<u>Corrected</u>
Burr Oak	$\frac{(57.7 - 12.0)^2}{(.98)(1.258)}$	= 74.14	(1.090)	= 80.8
Rock Elm	$\frac{(57.7 - 17.4)^2}{(.98)(1.293)}$	= 63.607	(1.006)	= 67.80
Weeping Willow	$\frac{(57.7 - 10.2)^2}{(.98)(1.498)}$	= 64.712	(1.098)	= 71.054
Paper Birch	$\frac{(57.7 - 17.3)^2}{(.98)(1.598)}$	= 51.595	(1.068)	= 55.104

Hardwoods - Overcooked (165°C + 22%)

			<u>"P"</u>	<u>Corrected</u>
Burr Oak	$\frac{(57.7 - 26.9)^2}{(.98)(1.143)}$	= 54.993	(1.026)	= 56.423
Rock Elm	$\frac{(57.7 - 27.85)^2}{(.98)(1.273)}$	= 47.854	(1.022)	= 48.9069
Weeping Willow	$\frac{(57.7 - 34.4)^2}{(.98)(1.074)}$	= 44.275	(.994)	= 44.0090
Paper Birch	$\frac{(57.7 - 32.3)^2}{(.98)(1.351)}$	= 38.369	(1.002)	= 38.446

Softwoods - Undercooked (165°C + 15%)

			<u>"P"</u>	<u>Corrected</u>
East Red Cedar	$\frac{(57.7 - 18.0)^2}{(.98)(1.4398)}$	= 56.27	(1.064)	= 59.874
Red Spruce	$\frac{(57.7 - 18.2)^2}{(.98)(1.5553)}$	= 51.83	(1.064)	= 55.1478
North Pine	$\frac{(57.7 - 13.8)^2}{(.98)(1.4761)}$	= 60.695	(1.083)	= 65.7326
Balsam Fir	$\frac{(57.7 - 15.6)^2}{(.98)(1.5300)}$	= 56.1557	(1.074)	= 60.3113

APPENDIX IV  
(Cont.)

KAPPA NUMBERS - Blank 57.7 ml

Softwoods - Overcooked (175°C + 25%)

			<u>"P"</u>	<u>Corrected</u>
East Red Cedar	$\frac{(57.7 - 18.3)^2}{(.98)(1.0871)}$	= 73.9657	(1.064)	= 78.699
Red Spruce	$\frac{(57.7 - 21.0)^2}{(.98)(1.1210)}$	= 66.813	(1.050)	= 70.154
North Pine	$\frac{(57.7 - 32.4)^2}{(.98)(1.5210)}$	= 33.946	(1.002)	= 34.0144
Balsam Fir	$\frac{(57.7 - 22.5)^2}{(.98)(1.4865)}$	= 48.326	(1.004)	= 48.519



APPENDIX V

	g/l	Average Absorbance	Absorbance	l/g.cm
North Pine				
raw	.410	.14	1.8, .10	.34
over	.206	1.12	1.20, 1.04	5.436
Balsam Fir				
raw	.228	2.005	2.000, 2.01	9.758
over	.288	.985	1.101, .867	3.42
Red Spruce				
raw	.364	3.205	3.10, 3.31	8.804
over	.236	1.51	1.74, 1.28	6.398
East Red Cedar				
raw	.272	1.515	1.23, 1.80	5.569
over	.384	1.49	1.73, 1.25	3.880
Weeping Willow				
raw	.208	.76	.69, .83	3.840
over	.178	.685	.73, .64	3.653
Paper Birch				
raw	.196	.185	.201, .169	.943
over	.196	.23	.282, .178	1.173
Rock Elm				
raw	.224	.265	.261, .269	1.183
over	.230	.485	.500, .470	2.108
Burr Oak				
raw	.202	.545	.693, .397	2.698
over	.198	.55	.531, .569	2.777

Sample Calculations - Northern Pine (raw)

$$A = lc$$

$$.14/(1\text{cm})(.410 \text{ g/l}) = .34 \text{ l/g-cm}$$

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