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# The Effects of Freeze-Drying on the Strength Characteristics of Naturally Aged Book Papers

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THE EFFECTS OF FREEZE-DRYING ON THE STRENGTH CHARACTERISTICS OF NATURALLY AGED BOOK PAPERS

by

#### Steven D. Sarokin

A Thesis submitted in partial fulfillment of the course requirements for The Bachelor of Science Degree

Western Michigan University Kalamazoo, Michigan May, 1981

#### ACKNOWLEDGEMENTS

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A special thank you to Mr. Larry Massie for his support and guidance.

#### ABSTRACT

Libraries occasionally have water main breaks, fires and floods. These disasters cause considerable water damage to thousands of books. Books that are left wet on the shelves will grow mold. Books that are quickly frozen and eventually freeze-dried can be restored to the shelf in good condition. Several libraries have used freeze-drying for this purpose and have had great success.

Freeze-drying is a process used to remove frozen liquid from a substance. The moist material is frozen and put in a vacuum. A high vacuum with some heat introduced will cause the frozen liquid to sublime. A dry-porous structure is left behind.

There has been no test data found considering the effects of freeze-drying on the strength of papers. The purpose of this report is to examine this process.

Eleven books up to 173 years old were wetted and freezedried. Some cockling of papers was observed. Strength properties of fold, tensile and zero span tensile essentially remained unchanged. Freeze-drying, in respect to the relatively small number of samples tested, does not have any detrimental effect on the strength of paper. Freeze-drying is recommended for restoration of water damaged books.

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#### INTRODUCTION

Books in libraries occasionally get accidently soaked with water. This could be caused by floods, water from putting out fires, or maybe water pipelines breaking. A very successful method used recently to restore these books back to the shelf is freeze-drying.

This report will investigate the effects of freezedrying on the strength properties of naturally aged book papers. The physical appearance of the books and covers will be discussed.

#### LITERATURE REVIEW

#### LIBRARIES WITH WATER DAMAGED BOOKS

In New York city at the Jewish Theological Seminary Library, a fire raged in April of 1966 (1). Over 70,000 books were burned to ashes. Water used to extinguish the fire damaged another 150,000 books. In Florence of that same year, at least ten times as many books were damaged in a flood. Various methods were tried for drying, demolding, and disinfecting. Paper towels, fans, dehumidifiers, chemicals, and blotting paper inserted between pages were tried in the restoration effort. Several of the methods failed. A few methods worked but were very time consuming. Schmelzer (1) does not mention the physical appearance of the air dried books, but a picture in the article clearly shows the book pages having a cockled finish. A cockle finish is defined as "A ripple-like finish caused by shrinkage during drying under little or no tension" (2). These two disasters stimulated research in the area of disaster preparedness  $(\underline{3})$ .

In August of 1975, Case Western Reserve University in Cleveland Ohio, had four hours of rain that damaged thousands of maps and periodicals located at the university's Sears Library ( $\underline{4}$ ). The materials were taken to McDonnell Douglas Aircraft Company for vacuum drying.

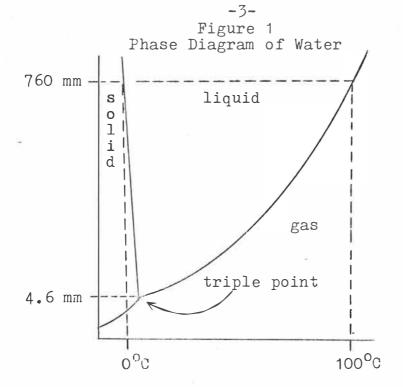
The Upjohn Library at Kalamazoo College in Kalamazoo Michigan had a water main break in May of 1974 ( $\underline{3}$ ). Over 2000 volumes were saturated. The books were frozen in food lockers and cold storage facilities and taken to the Upjohn Company for vacuum drying. The process was successful and the books seemed the same in apparence and in strength as compared to before the disaster.

On November 4, 1978, an eight-inch sprinkler main burst at the J. Henry Meyer Undergraduate Library at Stanford University (<u>5</u>). Over 40,000 volumes were damaged. The water saturated books were frozen at Modern Ice and Cold Storage in San Jose California. The Lockheed Missiles & Space Company in Sunnyvale California freeze-dried the books (<u>6</u>). The process did not produce any apparent adverse effects.

#### FREEZE-DRYING AND SUBLIMATION

Water can be present in three phases: gas, liquid or solid. These three phases can exist in equilibrium with each other at a point called the triple point. The phase diagram below describes the temperatures and pressures involved with this (7, 8).

-2-



Atmospheric pressure is about 760 mm Hg. At this pressure water would be a solid slightly below  $0^{\circ}$ C, liquid above  $0^{\circ}$ C, and a gas above  $100^{\circ}$ C. At 4.6 mm Hg and about  $0^{\circ}$ C, all three phases can exist in equilibrium.

Freeze-drying is a sublimation process. The water in the solid phase goes directly to the gas phase. For this to happen, the pressure must be below 4.6 mm Hg and the temperature (ideally) above  $0^{\circ}$ C. The higher the temperature, the faster the rate of sublimation, assuming the pressure is kept below 4.6 mm Hg.

#### FREEZE-DRYING AND VACUUM DRYING

For freeze-drying the pressure must be kept below 4.6 mm Hg. Vacuum drying would occur at pressures between 5-20 mm Hg (9).

#### THEORETICAL DISCUSSION

Lobben (<u>10</u>) has performed freeze-drying experiments on handsheets. He froze handsheets at different moisture levels to stop web consolidation. Comparisons were made between handsheets frozen and sublimed to handsheets dried normally. Various freeness levels were examined.

Generally, as interpreted by this author, the results showed that freezing of paper at above 70% solids content followed by freeze-drying resulted in papers having nearly identical strength to conventionally-dried sheets.

Fischer (<u>11</u>) conducted experiments comparing various methods of drying flooded books. Visual quality and physical measurements were compared before and after. A freeze-thaw vacuum method rated highest of 17 methods evaluated. This rating method showed the physical properties of the books remained essentially unchanged for the freeze-thaw vacuum method. However, the objective here was not to examine any one process in detail, but to compare processes to each other.

#### FAST FREEZING AND SLOW FREEZING

An example of fast freezing is dipping wet books into liquid nitrogen. This will freeze them instantly. Slow freezing is generally any freezing process that takes several hours.

A library would have large quantities of books damaged by water in any of the previously mentioned disasters. Fast

-4-

freezing is very expensive. Slow freezing, such as putting the books in an ice storage facility, would be the most likely used because of high costs, space limitations and handling time required for fast freezing.

Ice crystals form within structures that are frozen. The size of these ice crystals is determined by how quickly the material is frozen. Small ice crystals are formed in a quick freezing operation. Slow freezing would tend to form large ice crystals.

Mass freezing of books is a slow process. Thus, large crystals should be formed within the paper. It is possible that the forming of these crystals could weaken the fibers by puncturing cell walls (12).

Water expands 9.08% upon freezing if at 1Atm and  $32^{\circ}F(\underline{13})$ . This is expected to move the individual fibers apart which could decrease bonding strength (<u>12</u>).

#### STATEMENT OF PROBLEM

Freeze-drying of water damaged books appears to be an effective method for restoration. Obviously, books that have been freeze-dried after disasters did not have testing performed on them before wetting to facilitate comparison of the strength properties. It is generally agreed that the majority of books are undamaged (3, 5, 6).

Fischers (<u>11</u>) study found a freeze-vacuum method as an effective restoration process. The purpose of this paper is to perform some basic strength tests on a variety of papers before and after freeze-drying and to examine the

- )-

results. Observations will be made discussing appearance changes. Many spinoff research projects can result from this study.

#### EXPERIMENTAL DESIGN

Naturally aged papers will be chosen for testing. Strength tests of fold, tensile and zero span tensile will be performed before and after freeze-drying. Actual flood and freezing conditions will be simulated.

#### EXPERIMENTAL PROCEDURE

#### HANDMADE AGED PAPERS vs. NATURALLY AGED PAPERS

The books in the library that would be freeze-dried without any questions are archival material. These old papers generally are irreplaceable, while newer books that are water damaged can be re-ordered. A library would probably choose to freeze-dry everything only if freezing (and perhaps freeze-drying) facilities are available. However, the irreplaceable items would have first priority if limitations of freezing, freeze-drying and high-costs exists. The archivist can walk down the shelves and readily identify books that cannot be replaced. The librarian might not have this expertise (14).

For the above reasons, old papers were chosen for the testing. Handsheets can be made with specific properties and aged using well known available test data (<u>15</u>, <u>16</u>, <u>17</u>, <u>18</u>). Variables could be changed in the handsheets to check if

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freeze-drying has a direct relationship to these variables.

Trying to simulate actual papers that are in the archives by advanced aging could be done, but no direct correlation would be made between them because each sheet has its own history. Papers can come from an infinite number of backgrounds. None can be considered to be made in an identical way. An endless variety of paper fibers, chemical additives, forming and drying techniques, atmospheric exposure to sulfur and light exist for aged papers. Naturally aged papers were chosen for testing and the controls would be the strengths of each paper before and after freeze-drying.

### SAMPLING AND CONDITIONING

• Eleven books were chosen as representative of samples found in typical libraries (<u>14</u>). All readily available information on these books is listed in Figure<sup>2</sup> in case the readers of this paper would like to perform further research on the materials tested.

Since most aged papers are uncoated, no coated samples were chosen for this study. Further research might investigate coated papers to find if the sheets would stick together due to the binders in the coating dissolving and migrating to the surface. Stanford (5) did experience blocking of coated papers. However, the freeze-drying process released a majority of the stuck pages.

One book was chosen to determine if the books in general had achieved constant weight. It was stored, as the other

-7-

#### Figure 2

Data on the Books Tested

- Book #1. 1809, Edited by Rev. Isaac Milner, "The History of the Church of Christ," Fourth Volume Part 1. Farrand, Mallory and Co., Boston.  $8\frac{1}{4}X5\frac{1}{4}$  sq. inches. 433 total pages.
- Book #2. 1876, Edited by Fish, A.M., Daniel, W., "The Progressive Higher Arithmetric for Schools, Academias, and Mercantile Colleges," Ivison, Blakeman, Taylor & Co., New York. 7<sup>4</sup>/<sub>4</sub>X4.5 sq. inches. 455 total pages.
- Book #3. 1885, Steele, "A Brief History of the United States," Barnes History Series. American Book Company, New York. 8X5<sup>1</sup>/<sub>4</sub> sq. inches. 356 total pages.
- Book #4. 1892, No author found, Ivanhoe, Arlington Edition, No publishers found, 7.5X5 sq. inches. 359 total pages.
- Book #5. 1902, Edited by Morris and written by Master Historians. "The Great Republic" Volume 3, The R.S. Belcher Co., New York.  $8X5\frac{1}{2}$  sq. inches. 368 total pages.
- Book #6. 1910, Alexandre Dumas in Three Volumes. "The Vicomte de Bragelonne or Ten Years Later," Thomas Nelson and Sons, New York. 64X4 sq. inches. 860 total pages.
- Book #7. 1913, Rudyard Kipling. "Departmental Ditties and Ballads and Barrack Room Ballads," Garden City Doubleday, Page & Company. 7X4.5 sq. inches. 214 total pages.
- Book #8. 1922, Donn Byrne. "Messer Marco Polo". The Century Company, New York. 7.5X4.5 sq. inches. 147 total pages.
- Book #9. 1924, Don Quixote by Miguel De Cervantes Saavedra. Translated by Charles Jervas. Printed in England at Oxford University Press by Frederick Hall Humphrey Millford Press, London. Volume 1, 5X3 sq. inches. 551 total pages.
- Book #10. 1932, Burris, Elson and Keck. "Elson Junior Literature," Book 2, Scott Foreman & Company.  $7\frac{1}{2}X5\frac{1}{2}$  sq. inches. 551 total pages.
- Book #11. 1943, Krickenberger and Welchons. "Plane Geometry". Ginn and Company, Boston. 7<sup>1</sup>/<sub>2</sub>X5<sup>1</sup>/<sub>4</sub> sq. inches. 546 total pages.

books were, in a constant temperature and humidity room at 73<sup>°</sup>F and 50% relative humidity. The book was weighed initially and after a random period of three days. Results are below.

> Weight at beginning = 188.0600 grams Weight after three days = 188.5022 grams  $\frac{188.5022 - 188.0600}{188.0600} = 0.2\%$  change

This change was considered insignificant. All of the books were therefore assumed to be at constant weight.

The middle of the books was chosen for testing. The purpose of this was to avoid any sheets that had excessive exposure to light and acid atmospheric conditions. It is expected that the end sheets would be slightly weaker and less consistant in performance as compared to the middle.

For these same reasons, the edges (or margins) of the books were avoided for the strength tests. No effort was made to avoid the printed areas. The printed areas might be compressed slightly more than the areas between lines, however any difference that might exist was considered minute and irrelevant for these tests. No papers with drawings were tested, for these are commonly printed on a type of paper that is different from the rest of the book.

The middle sections of the books were pulled out for testing. By looking at a book, one can see at the binding how the sections are divided (into signatures). The front

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page of the section is from the same sheet as the last page of the section. Front sheets were tested before freezedrying (or wetting) and the corresponding back sheets from these sections were recorded and put back in the book for testing after (wetting and) freeze-drying.

The covers of the books were taken off. The sheets to be tested from the middle section, plus several extra sections that were near the middle, were combined together. The covers of each book  $\frac{\text{were}}{\Lambda}$  positioned back in place. Each book was weighed with the cover and without before any wetting or freeze-drying. These weights will be discussed later in this paper.

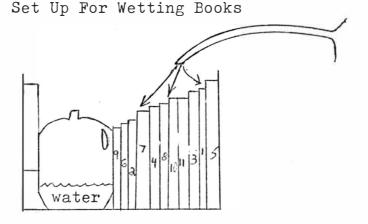
#### SIMULATING ACTUAL CONDITIONS

The procedure to simulate library conditions for wetting and freeze-drying was patterned after articles written by Stanford University Libraries (5) and Lockheed Missiles & Space Company (6). Some steps were changed to permit the generation of more data.

The books were placed in a milk crate in the upright position, as they would be seen on a library shelf. Pressure was simulated on the books by putting an open plastic gallon milk container and one extra book in the milk crate against the books. A sketch below, without the milk crate, shows the set up.

-9-

### Figure 3



Some water was put in the gallon milk carton in hope of obtaining a rate of sublimation during the freeze-drying.

The books were placed in a sink and a tap water supply was turned on to a constant drip over the books to be tested. The supply stream went in three different directions spreading over the width of the books. The rate was set arbitrarily at approximately 3.2 gallons per hour. The books were totally saturated after about three hours. Water was dripped on the books for a total of eight hours. This is considered a typical time which would elapse in a library flood before a water main break would be found and shut off, because this could happen during nights or weekends when no one is around.

It is commonly agreed that the books should be frozen before 24 hours has elapsed and definitly must be frozen before 48 hours has passed in order to prevent and stop any mold growth. These books sat for twelve hours, as suggested by Lockheed and Stanford (5, 6), and were frozen in a food locker at Western Michigan University. It is not known how fast the books froze, but the quantity involved and freezing process was estimated to take one day. These books were frozen slowly and large crystal formation could be expected.

Freeze-dried books in recent years have been frozen to  $-10^{\circ}F$  to  $-20^{\circ}F$ . The chamber used for this test had a temperature of  $8^{\circ}F$  when checked after three days had elapsed. This difference in temperature is believed to be insignificant. As long as the process was slow, the ice crystals would be large. Once the ice is formed it will not change. The colder temperatures make the books more stable for transport to the freeze-dryer.

After five days of freezing, the books (still in the milk crate) were taken to the The Upjohn Company for freezedrying. Upjohn was kind enough to donate the use of their pilot freeze-drier for this experiment. The Upjohn Company is about five miles from the area where the books were frozen. To ensure no melting, the books were packed on the sides with bags of ice during transport. The freeze-drier was manufactured by Usifroid Procedes Rieutord, Model SM JR, with MKS instrumentation.

Although the milk crate fit into the freeze-drier, it was not realized until test time that the materials had to be moved to the back side of freeze-drier for the drying. The area toward the back was too small for the milk crate. It also turned out to be too small for the books to be stood upright. So, the books were removed from the crate and put on their edges. Pressure could not be applied to the books, as was hoped, during the process.

After everything was in place, the process was begun. The freeze-drier was taken to its lowest pressure (within

-11-

minutes) of 0.22 mm Hg. Heat was applied through the shelf the books were on and gradually increased to  $100^{\circ}$ F. This is well below the triple point of water and in the gas region of the phase diagram.

The books were checked after 24 hours. Since no pressure was applied, one of the books (#5) had fallen open. The books were all separated and weighed. Data on the different weighings  $\stackrel{\text{are}}{\wedge}$  presented in Table 1.

Books 5, 6 and 9 were removed because they were at the desired weight or very close to it. These three books were located on the two sides of the books tested. The remaining books were put back in the chamber. This time, masking tape was wrapped once around them to ensure that no others would fall open.

The pressure was again evacuated to 0.22 mm Hg. However, the temperature could not be set at 100°F because it would probably over-dry the books. The objective was to bring the books out at the weight they were before wetting. A book with very little moisture would be brittle and could take months to reach equilibrium condition. Therefore, it was decided to set the temperature at 32°F. This was still in the gas portion of the phase diagram for water, so sublimation would occur.

The freeze-dryer was available for inspection only once every 24 hours. The entire process was stopped after two days. Book numbers 2, 4 and 8 were very close to normal weight. One more day of freeze-drying would over-dry the remaining samples. The others still had excess water.

-12-

The books with excessive moisture were 1, 3, 7, 10, and 11. They were put in an oven at  $100^{\circ}$ F for about eight hours. They were removed when normal weight was achieved. The books were all tested within 48 hours after drying. See Table 1 for the weights of the books.

The books are listed chronologically in all the Tables presented. (Refer to Table 2 for the following discussion). The basis weights were determined using the average of three values and calculated according to the Technical Association of the Pulp and Paper Industry (TAPPI) Standard T40 os79. The pounds per ream  $\stackrel{\text{were}}{\Lambda}$  based on a book size of 25"X38" with 500 sheets.

Any values designated "before" in the results refer to tests done before the samples were wetted or freeze-dried. The "after" values were taken after the freeze-drying and the sheets had reached normal weight.

The caliper values were not taken on identical sheets. It should be noted that book #1 had wide variances in caliper. Caliper was performed to find if an increase in sheet thickness would occur due to the greater volume occupied by ice as compared to water. An automatic hand micrometer was used with numbers read off a dial.

A spot test for groundwood was checked, as recommended by Barrow Research Laboratory  $(\underline{19})$ . This test uses 1 gram of phloroglucinol in 50 ml of HCl.

The pH of the books was tested using TAPPI Standard T509 su 68. This was a cold extraction pH. No printed area was used for this test.

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#### Table 1

	A	В	С	D	E	F
Book #1	200	63	300	236		63
Book #2	146	52	190	154	53	
Book #3	218	94	331	296		94
Book #4	167	75	213	191	76	
Book #5	345	183	378		183	
Book #6	32	32	37		32	
Book #7	52	52	75	64		53
Book #8	132	63	174	152	63	
Book #9	79	31	79	•	31	
Book #1	0 234	110	298	272		112
Book #1	1 251	130	<u>3</u> 70	335		125

#### Weight Measurements of Samples

#### KEY:

- A. The weight of the books with the covers before any wetting or freeze-drying.
- B. The weight of just the papers before any wetting or freeze-drying.
- C. The weight of the books and covers after 24 hours of freeze-drying. Books 5, 6 and 9 were removed at this time.
- D. The weight of the books with covers after 48 hours of freeze-drying. The first 24 hours were at  $100^{\circ}$ F and the last 24 at  $32^{\circ}$ F.
- E. The weight of just the papers after sitting in a constant temperature and humidity room overnight.
- F. The weight of just the papers that were put in a 100°F oven for no shorter than 4 hours and no longer than 12 hours.
- All weights reported in grams.

# Table 2

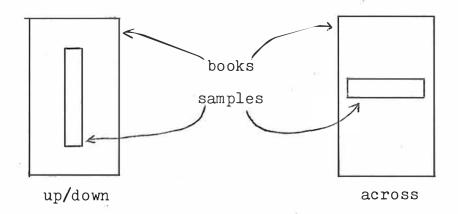
Physical Background Da
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		F H,y S L C	al Dackgr	ounu Data			
		1	milli-inc	hes ""			
	Year	Basis wt. g/m <sup>2</sup> lb/rm	Caliper Before	Caliper After	Spot test groundwood	Нq	Spot Tes Alum
Book #1	1809	52,6 42.3	5.14x10	-3 4.83	No	4.7	???
Book #2	1876	67.8 45.7	4.23	4.26	No	5.5	Yes
Book #3	1885	92.1 62.1	3.81	3.85	No	4.7	No
Book #4 Book #5 Book #6	1892	191.6 61.8	5.19	5.22	Yes	4.0	Yes
Book #5	1902	105.9 71.5	6.09	5.96	No	5.7	Yes
Book #6	1910	31.8 21.9 78.5 53.0	1.40	2.32	No	8.0	Ňo
BOOK #/	1913	18.5 55.0	5.14	3.36	No	5.3	No
Book #8	1922	105.0 70.8	8.15	7.98	No	4.6	Yes
Book #9 Book #10	1924	39.2 26.5 83.4 56.3	1.75	2.07	No	4.5	Yes
Book #11	1932 1943	83.4 56.3 72.4 48.8	3.50 2.96	3.12 3.39	No No	4.1	Yes
	1,54,5	12.4 40.0	2.90	J. J.	NO	4.9	No
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A spot test for alum was performed and procedures outlined by Barrow Research Laboratory were followed (<u>19</u>). It uses 1 gram of aluminon per liter of distilled water.

Test data using the terminology "up/down" and "across" is explained in Figure 4 below. These refer to the direction the test sample was taken.





Fold values were recorded in Table 3. The fold test was performed with two 15 mm wide strips instead of the recommended one (20). A 0.55 Kg dead weight was used. Two strips were used because values tended to be too low with one strip and the possibility of finding a trend would be masked by the strips being too weak to record on the tester. Two strips usually generated numbers high enough so a comparison could be made.

Approximately ten tests were run per direction per book. Books 6 and 9 did not have 10 fold tests run due to the high folds obtained. Some books were not wide enough to test in the across direction. These are noted in the columns by the words "too short".

-14-

Ta	ble	: 3

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FOLD two 15 mm wide strips

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1

		Before Up/Down	After Up/Down	Before Across	After Across	Std. Dev. Bf Up/Dow	Std. Dev.	Std. Dev. Bf Across	Std. Dev Aft Acrss
	Book #1	107	130	214	581	77	95	106	565
	Book #2	83	140	75	109	31	36	18	46
	Book #3	7	16	5	6	2	10	1	2
	Book #4	11	2	1	3		0.5		1
	Book #5	14	28	8	14	3	6	2	2
	Book_#6	3960	5988	Too short		364		loo short	
	Book #7	10	14	13	15	2	2	4	4.1
	Book #8	2	3	Too short	1	1	Too short	Too shor	Too short
	Book #9	1329	1653	Too short	'oo short	544	929	loo short	Too short
	Book #10	2	5	8	11	0.5	2	6	4
	Book #11	373	493	178	261	135	226	59	142
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Standard deviations were calculated. It was assumed that dripping the water on the books for eight hours would wash out any impurities scattered in the sheet and make it more uniform, thus producing a lower standard deviation. An equal number of tests were performed for each sample with corresponding pages tested before and after.

The Instron results were recorded in Table 4. The Instron test was used at a crosshead speed of 2 cm/min and a chart speed of 10 cm/min. The full scale load adjustment was alternated between 5 and 10 Kg. Test data was accurately recorded and repeated identically for each test sample direction. Ten tests were run per direction per sample.

The zero span tensile results were recorded in Table 5. The Zero Span Pulmac Tester was used to get an approximate value of individual fiber strength before and after freezedrying. A constant load rate was used for each test. Ten tests were run per sample direction.

#### RESULTS

The caliper values followed no trend. They increased for some and decreased for others.

The only book showing positive results for groundwood was book #4.

The only book with alkaline characteristics was book #6 with a pH of 8.0. The other books were between 4.0 and 5.7 pH.

The color spot test for alum showed books 2, 4, 5, 8, 9 and 10 to possess alum. Book #1 could not be confirmed

-15-

Table 4 INSTRON

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	elongatio	n_elngatn	counts	counts	kg•cm	kg•cm	kg•cm/cm <sup>2</sup>	kg·cm/cm <sup>2</sup>
	(cm) Bf Up/Down	(cm) Aft	Integ.Bf Un/Down	Integ. Af Up/Down	t Area Bf Un/Down	Area Aft Up/Dn	TEA Bf Up/Dn	TEA Aft Un/D
Book #1	47	50	203	260	.81	1.04	.0320	.0409
Book #2	47	45	135	162	.27	• 32	.0106	.0128
Book #3	31	30	55	78	.22	• 31	.00866	.0123
Book #4	41	60	42	108	.08	.22	.00330	.0085
Book #5	66	63	168	245	• 336	.49	.0132	.0193
Book #6	33	30	81	87	. 324	• 348	.0128	.0137
Book #7	95	88	159	150	.318	. 300	.0125	.0118
Book #8	80	81	90	106	.180	.212	.0071	.0087
Book <u>#9</u>		. 34	35	55	. 140	•220	.0055	.0087
Book #10	32	43	28	42	.112	• 168	.0044	.0066
Book #11	35	61	47	110	<u>• 188</u>	•440	.0074	.0173
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			<u>v</u> .					

## Table 5

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Zero Span Pulmac Tester, all zero gaps

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	Before Up/Down	After Up/Down	Before Across	After Across	Std. Dev. Bf Up/Dn	Std. Dev. Aft Up/Dr	Std. Dev. Bf Across	Std. Des Af Acres
Book <u>#1</u>	32	28	24	23	1	2	3	3
Book #2	26	26	30	34	2	1	2	3
Book #3	20	21	27	25	1	1	1	2
Book #4	15	21	19	23	2	3	1	6
Book #5	21	23	24	29	2	1	1	2
Book #6	17	19	10	11	1	1	4	5
Book #7	25	26	16	18	1	2	1	1
Book #8	22	26	20	28	4	1	5	2
Book #9	22	23	14	14	2	1	1	1
Book #10	26	26	16	18	1	1	1	4
Book #11	35	38	22	20	2	1	1	6
				•				
		•		•				
				-				

one way or the other. The remaining books tested negatively for alum.

Fold increased by an average of about 1.7 times. The reason for these results is not understood. Possible mechanisms are discussed later in Possible Mechanisms for Further Study.

Standard deviations were not lower after freezedrying. Books 1, 2 and 11 significantly increased in standard deviation while the others remained about the same.

The elongation readings show little difference before and after, with the exception of book #11 showing a marked increase. The integrator readings, the area, and the tensile energy absorbtion (TEA) show no significant change.

It was expected that the zero span tensile values would decrease due to crystal structures puncturing cell walls. Instead, there was no adverse effect from freezedrying. The numbers show the sheet to be undamaged by the process.

#### CONCLUSIONS AND RECOMMENDATIONS

Freeze-drying does not adversely affect the strength properties of fold, tensile and zero span. Fold, tensile and zero span strength were maintained. The increase in fold could possible by attributed to hysterisis (21).

Freezing the books under pressure and maintaining the applied pressure during freeze-drying should decrease cockling. It should be noted that no pressure was applied in the Kalamazoo College vacuum drying case and the books resulted in good shape. Their books were stacked on top

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of each other. The correlation between the simulated conditions used in this experiment and real-life flood disasters should be re-examined.

Packing the books for freezing with sizes as closely matched as possible will ensure uniform pressure and help minimize cockling.

The books should be frozen as fast possible to help prevent mold growth (that would occur in the high humidity conditions present after the books have been saturated).

The books should be watched closely during freezedrying. Books on the edges will dry first. The direction of stacking does not affect the sheets but might affect the binding. If any pages are frozen together, the book still needs more drying. If all the pages are free of each other and do not feel damp, remove the book from the dryer.

Use the lowest pressure (highest vacuum) available in the dryer and set the temperature no higher than  $100^{\circ}$ F.

#### FURTHER SUGGESTIONS

An attempt was made to do fiber analysis using the Herzberg and Selleger stain (22). This failed when all the stains turned identical colors. Further testing in this area should include the C stain and fiber microscopy to establish a better background knowledge of the sheet.

If possible, electron photomicrographs should be taken of the individual fibers and compared to the corresponding pages after freeze-drying. This may establish if cell wall damage occurs.

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Caliper should be taken with the most sensitive instruments available. A comparison on identical sheets will establish if ice formation pushes the fibers apart. However, over 70% of the sheet is void space, and the fibers might just be shifting into already empty spaces, making the "pushing" effect unnoticeable using caliper measurements (21).

The freezing of the books should be watched and an estimate of the time elapsed when completely frozen would aid further research. It should be kept in mind that not every molecule of water will be entirely frozen through at levels of temperatures used here. The sheet will still appear frozen even though every bit of water in the sheet might not be frozen.

It would be best to design or obtain a fold tester that uses a  $90^{\circ}$  angle instead of the conventional fold of  $270^{\circ}$  (23). This will be more sensitive to the aged papers.

Perform the tear test and stiffness test.

#### POSSIBLE MECHANISMS FOR FURTHER STUDY

The water used to drip on the books was not checked for hardness, but was expected to have a high mineral content. If minerals are deposited on the sheet, they could interfere with fiber bonding. Do the minute mineral particles instead fill gaps in the sheet and create a bridge for the fibers to bond at more sites than were possible before?

Does the washing produce a more neutral sheet? If it does, than the sheet would slow its aging process, but

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would it change its strength immediately?

Does the washing give fibrils mobility to latch on to each other? Are more hydrogen bonds created?

An article about freezing tools at -310<sup>o</sup>F claims the tools last up to four times longer as a result (24). Particles of austenite are changed to smaller, more stable particles of mortensite which decrease wear resistance. Does freezing change the chemical structure of cellulose, making it stronger?

While pulling a vacuum on the paper, is air pulled out of the individual fibers? If so, this could collapse the fiber and increase its bonding area.

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## -26-APPENDIX 1

## DISCUSSION OF PHYSICAL APPEARANCE

The freeze-dried samples and the original papers are available in the first ten copies of this report. The remaining samples and the book covers are on deposit at the archives at Western Michigan University, Waldo Library. It should be noted that the evaluation to follow is subjective.

A water stain is evident on books 1, 3 and 7. These were some of the books that were removed from the freezedrier before they were dry. The stain is symmetric around the center of the page. This suggests a uniform drying rate from the book edges.

Book 6 had a red stain on some of the pages. This color leached out from one of the book covers.

The book covers suffered considerable warpage. It should be kept in mind that the covers in this experiment were removed from the binding. The binding could provide uniform pressure on the covers and thus possibly minimize warpage.

The books were rated on the scale below. The drawings to the right are approximations of the A through E ratings. A cockle finish is most noticeable with a complete signature from a book rather than from the single pages provided here.

Rating	Description	Representation
A B C D E	very slight cockle- slight cockle- very noticeable cock severe cockle- very severe cockle	7

The book ratings appear below.

look	Rating
1 2	C B
3	D
4 5 6	Α
5	С
6	D
7	Α
8	A
9	A
10	В
11	D
8 9	A A B

The print on all the samples remained unchanged. The books are not as aesthetic when viewed from the sides. APPENDIX 2

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SAMPLES

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showing under what strong delusions thos e are permitted to lie, who love not the truth, but have pleasure in unrighteousness. The judicious L'Enfant, who is rarely liberal in his censures, breaks out on occasion of the last mentioned sentiment of Gerson, in the following terms: "I own, I don't understand Gerson's logic on this occasion. He draws a very blunt and rash inference; especially as it was the most improper thing in the world he could say to induce the hussites of Bohemia to come to Constance, whither they were summoned."

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BOOK #1 FREEZE-DRIED

The five nations, (for the Spaniards were now added to the French, the Germans, the English, and the Italians,) proceeded to elect a pope; and the choice fell upon Othc de Colonna, who took the name of Martin V. This happened in the latter end of the year 1417. All these nations, on the day after the pope's coronation, concurred in a resolution to demand of the new pope the reformation of the church which he had promised to make after he should be elected. He gave them good words, but did nothing effectual. The Germans were uneasy at his delays, and so were the French; though these, by joining with the Italians and the Spaniards, had caused the deferring of the reformation till after the election of a pope. The answer, which Sigismund gave to the French, was severe, but just. "When I urged you that the church might be reformed before the pope was elected, you would not consent. You would have a pope before the reformation. Go to him yourselves. I have not the same power which I had while the see was vacant."\* It is the office of history to do justice to all characters; on which account it behooves us to declare, that Sigismund, grossly perfidious as he had shown himself in regard to Huss, appears to have been sincerely desirous of a partial reformation in the church. He had neither the knowledge nor the zeal, sufficient to lead him to any thing like an evangelical reformation; but, with many other popish princes, he

\* L'Enfant, vol. ii. p. 207.

INS ANT BUILT

GOVERNMENT STANDARDS

BOOK #2 FREEZE-DRIED

#### **GOVERNMENT STANDARDS**

## OF MEASURES AND WEIGHTS.

**306.** In early times, almost every province and chief city had its own measures and weights; but these were neither definite nor uniform. This variety in the weights and measures of different countries has always proved a serious embarrassment to commerce; hence the many attempts that have been made in modern times to establish uniformity.

The English, American, and French Governments, in establishing their standards of measures and weights, founded them upon unalterable principles or laws of nature, as will be seen by examining the several standards.

#### UNITED STATES STANDARDS.

**307.** In the year 1834 the U. S. Government adopted a uniform standard of weights and measures, for the use of the custom houses, and the other branches of business connected with the General Government. Most of the States which have adopted any standards have taken those of the General Government.

**308.** The invariable standard unit from which the standard units of measure and weight are derived is the day.

Astronomers have proved that the diurnal revolution of the earth is *entirely uniform*, always performing equal parts of a revolution on its axis in equal periods of duration.

Having decided upon the invariable standard unit, a measure of this unit was sought that should in some manner be connected with extension as well as with this unit. A clock pendulum whose rod is of any given length, is found always to vibrate the same number of times in the same period of duration. Having now the day and the pendulum, the different standards hereafter given have been determined and adopted.

#### STANDARD OF EXTENSION.

**309.** The U. S. standard unit of measures of extension, whether linear, superficial, or solid, is the yard of 3 feet, or 36 inches,

## BOOK #3 FREEZE-DRIED

#### DEVELOPMENT OF ENGLISH COLONIES. 95

and deacons took their seats in front of the preacher's desk, facing the congregation. The old men, the young men, and the young women each had their separate placo. The boys were perched on the pulpit-stair or in the galleries, and were kept in order by a constable. The light came straggling through the little diamond-shaped window-panes, weirdly gilding the wolf-heads which hung upon the walls-trophies of the year's conquests. The services began with the long prayer, and was followed by reading and expounding of the Scriptures, a psalm-lined by one of the ruling elders-and the sermon. Instrumental music was absolutely proscribed, as condemned by Amos v. 23. The sermon was often three or four hours long, and at the end of each hour the sexton turned the hour-glass which stood upon the desk. Wee to the youngster whose eyelids drooped in slumber! The ever-vigilant constables, with their wands tipped on one extremity with the foot, and on the other with the tail of a hare, brought the heavier end down on the nodding head. The care-worn matron who was betrayed into a like offense, was gently reminded of her duty by a touch on the forehead with the softer end of the same stick. After the sermon, came the weekly contribution; the congregation, marching to the front, and depositing their offerings in the money-box held by one of the elders. After dismissal, the people returned home in as orderly a way as they came.

The Middle Colonies. -- The manners of the New York people were essentially Dutch. Many customs then inaugurated still remain in vogue. Among these is that of New Year's Day visiting, of which General Washington said, "New York will in process of years gradually change its ancient customs and manners, but whatever changes take place, never forget the cordial observance of New Year's Day." To the Dutch we owe our Christmas visit of Santa Claus, colored eggs at Easter, doughnuts, crullers, and New Year's cookies.

The Dutch mansion was built, usually, of brick. Its gable-end, receding in regular steps from the base of the roof to the summit, faced the street. The front-door was decorated with a huge brass knocker, burnished daily. While the Connecticut mistress spun, wove, and stored her household linens in crowded chests, the Dutch matron scrubbed and scoured her polished floor and wood-work. Every family had a cow that fed in a common pasture at the end of the town, and their tinkling bells, as they came and went, of their own accord, at night and morning, proclaimed the milking-hour. The happy burghers breakfasted at dawn, dined at eleven, and retired at sunset. On dark evenings, as a protection for belated wanderers, lighted candles were placed in the front windows.

Along the Hudson, the great patroons, supported by their immense estates and crowds of tenants, kept up the customs of the best European society of the day.  $\checkmark$ 

Philadelphia was not only the largest city in the United States, but it was famous for its flagged side-walks—then a rare luxury in any city, the regularity of its streets, and the elegance of its brick and stone residences. The trees bordering the carriage-ways and the gardens and orchards about the houses made it just such a "fair greene country town" as Penn wished it to be.

The Southern Colonists differed widely from the Northern in habits and style of living. In place of thickly-settled towns and villages, they had large plantations, and were surrounded by a numerous household of servants. The negro quarters formed a hamlet apart, with its gardens and poultry yards. An estate in those days was a little empire. The planter had among his slaves men of every trade, and they made most of the articles needed for common use upon the plantation. IVANHOE.

Book#4

presse dried

Saxons who robbed the chapel at St. Bees of cup, candlestick, and chalice, were they not?"

They were godless men," answered Cedric.

"Ay, and they drank out all the good wine and ale that lay in store for many a secret carousal, when ye pretend ye are but busied with vigils and primes!—Priest, thou art bound to revenge

such sacrilege." "I am indeed bound to vengeance," murmured Cedric; "Saint Withold knows my heart." Front-de-Bœuf, in the meanwhile, led the way to a postern,

where, passing the moat on a single plank, they reached a small barbican or exterior defense, which communicated with the open field by a well-fortified sally-port. "Begone, then; and if thou wilt do mine errand, and if thou

return hither when it is done, thou shalt see Saxon flesh cheap as ever was hog's in the shambles of Sheffield. And, hark thee, thou seemest to be a jolly confessor, come hither after the on-slaught, and thou shalt have as much Malvoisie as would drench thy whole convent."

thy whole convent." "Assuredly we shall meet again," answered Cedric. "Something in hand the whilst," continued the Norman; and, as they parted at the postern-door, he thrust into Cedric's reluc-tant hand a gold byzant, adding, "Remember, I will flay off both cowl and skin, if thou failest in thy purpose!" "And full leave will I give thee to do both." answered Cedric, leaving the postern, and striking forth over the free field with a iovful step. "if when we meet next I deserve not better at thing

joyful step, "if when we meet next, I deserve not better at thine hand." Turning then back toward the castle, he threw the piece of gold toward the donor, exclaiming at the same time, "False Norman, thy money perish with thee!"

Front-de-Bœuf heard these words imperfectly, but the action was suspicious. "Archers!" he called to the warders on the outer battlements, "send me an arrow through yon monk's frock! Yet stay!" he said, as his retainers were bending their bows, "it avails not; we must thus far trust him, since we have no better shift. I think he dares not betray me. At the worst, I can but treat with these Saxon dogs, whom I have safe in kennel. Ho! Giles jailer, let them bring Cedric of Rotherwood before me, and the other churl, his companion-him, I mean, of Coningsburgh—Athelstane there, or what they call him. Their very names are an encumbrance to a Norman knight's mouth, and have, as it were, a flavor of bacon. Give me a stoup of wine, as jolly Prince John said, that I may wash away the relish.

Place it in the armory, and thither lead the prison away in terms His commands were obeyed; and, upon entering the Gothic apartment, bung by many spoils won by his own valor and that of his father, be found a flagon of wine on the massive oak table, and the two Saxon captives under the guard of four of his de-pendents. Front-de-Bœuf took a long draught of wine, and then addressed the prisoners; for the manner in which Wamba drew the cap over his face, the change of dress, the gloomy and broken light, and the baron's imperfect acquaintance with the features of Cedric (who avoided his Norman neighbors, and seldom stirred

Boolf#5 freeze-dived

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#### PARKMAN] CHAMPLAIN AND THE IROQUOIS.

aided them in their wars, and roused the undying enmity of powerful foes. A description of the settlement of Quebec, of Champlain's first excursion with the Indians, of the discovery of the lake which bears his name, and of his first encounter with the Iroquois, may be taken from Parkman's "Pioneers of France in the New World."]

AND now, peace being established with the Basques, and the wounded Pontgravé busied, as far as might be, in transferring to the hold of his ship the rich lading of the Indian canoes, Champlain spread his sails, and once more held his course up the St. Lawrence. . . .

Above the point of the Island of Orleans, a constriction of the vast channel narrows it to a mile: on one hand the green heights of Point Levi; on the other, the cliffs of Quebec. Here a small stream, the St. Charles, enters the St. Lawrence, and in the angle between them rises the promontory, on two sides a natural fortress. Land among the walnut-trees that formed a belt between the cliffs and the St. Lawrence. Climb the steep height, now bearing aloft its ponderous load of churches, convents, dwellings, ramparts, and batteries,- there was an accessible point, a rough passage, gullied downward where Prescott Gate now opens on the Lower Town. . . . Two centuries and a half have quickened the solitude with swarming life, covered the deep bosom of the river with barge and steamer and gliding sail, and reared cities and villages on the site of forests; but nothing can destroy the surpassing grandeur of the scene. . . .

A few weeks passed, and a pile of wooden buildings rose on the brink of the St. Lawrence, on or near the site of the market-place of the Lower Town of Quebec. The pencil of Champlain, always regardless of proportion and perspective, has preserved its semblance. A strong wooden wall, surmounted by a gallery loop-holed for musketry, enclosed three buildings, containing quarters

# Booli # 6 frieze divid

THE VICOMTE DE BRAGELONNE. 387 by his eminence in my service is his own wealth and not

mine." "But, sire, does your majesty reflect," said Anne of Austria, "that you have not ten thousand crowns in your coffers ?

" Madame, I have just performed my first royal action,

and I hope it will worthily inaugurate my reign." "Ah! sire. you are right!" cried Mazarin; " that is truly great—that is truly generous which you have just done." And he looked, one after the other, at the pieces of the act spread over his bed, to assure himself that it was the original and not a copy that had been torn. At length his eyes fell upon the fragment which bore his signature, and recognizing it, he sunk back on his bolster in a swoon. Anne of Austria, without strength to conceal her regret, raised her hands and eyes toward heaven. "Oh! sire," cried Mazarin, " may you be blessed! My God ! May you be beloved by all my family. Per Baccho ! If ever any of those belonging to me should cause your displeasure, sire, only frown, and I will rise from my tomb ! "

This *pantalonnade* did not produce all the effect Mazarin had counted upon. Louis had already passed to considerations of a higher nature, and as to Anne of Austria, unable to bear, without abandoning herself to the anger she felt burning within her, the magnanimity of her son and the hypocrisy of the cardinal, she arose and left the chamber, heedless of thus betraying the extent of her grief. Mazarin saw all this, and fearing that Louis XIV. might repent his decision, in order to draw attention another way he began to crv out, as, at a later period, Scapin was to cry out, in that sublime piece of pleasantry with which the morose and grumbling Boileau dared to reproach Molière. His cries, however, by degrees, became fainter; and when Anne of Austria left the apartment, they ceased altogether.

" Monsieur le cardinal," said the king, " have you any recommendations to make to me?"

"Sire," replied Mazarin, "you are already wisdom

Book#7 Breezedviel

## THE BALLAD OF THE "BOLIVAR"

Seven men from all the world, back to Docks again, Rolling down the Ratcliffe Road drunk and raising Cain:

Give the girls another drink 'fore we sign away — We that took the "Bolivar" out across the Bay!

- We put out from Sunderland loaded down with rails;
  - We put back to Sunderland 'cause our cargo shifted;
- We put out from Sunderland met the winter gales
  - Seven days and seven nights to the Start we drifted.
    - Racketing her rivets loose, smoke-stack white as snow,

All the coals adrift a deck, half the rails below

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Book #3 breeze-dued

## VIII

ND so they set forth with their great train of red, snarling camels and little patient donkeys and slender, nervous horses toward the rising sun. Behind them the green hills of Palestine died out as a rainbow dies out, and now there was sand before them and now bleak mountains, and by day the wind was swift and hot and by night it was black and cold. And moons were born and died . . .

And they passed through the land of the King of Armenia, and they passed Ararat, the mountain where Noë brought his ark to anchor, and where it still is, and where it can be seen still, but cannot be reached, so cold and high and terrible is that mountain.

And they passed ruined Babel, that was built of Nimrod, the first king of the 71

Book # 9 freeze-dried

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### PART I, CHAPTER XXIX

he offered them. The barber, who all this time had stood silent and in suspense, paid also his compliment, and, with no less goodwill than the priest, made them an offer of whatever was in his power for their service. He told them also, briefly, the cause that brought them thither, with the strange madness of Don Quixote, and that they were then waiting for his squire, who was gone to seek him. Cardenio hereupon remembered, as if it had been a dream, the quarrel he had with Don Quixote, which he related to the company, but could not recollect whence it arose.

And this instant they heard a voice, and, knowing it to be Sancho Panza's, who, not finding them where he had left them, was calling as loud as he could to them, they went forward to meet him; and asking him after Don Quixote, he told them, that he had found him, naked to his shirt, feeble, wan, and half dead with hunger, and sighing for his lady Dulcinea; and though he had told him, that she laid her commands on him to come out from that place, and repair to Toboso, where she expected him, his answer was, that he was determined not to appear before her beauty, until he had performed exploits that might render him worthy of her favour: and, if his master persisted in that humour, he would run a risk of hever becoming an emperor, as he was in honour bound to be, nor even an archbishop, which was the least he could be: therefore they should consider what was to be done to get him from that place. The licentiate bid him be in no pain about that matter; for they would get him away, whether he would or no.

He then recounted to Cardenio and Dorothea what they had contrived for Don Quixote's cure, or at least for decoying him to his own house. Upon which Dorothea said, she would undertake to act the distressed damsel better than the barber, especially since she had a woman's apparel, with which she could do it to the life; and they might leave it to her to perform what was necessary for carrying on their

Booh #10 Breeze-dried

## AS YOU LIKE IT

you will seem more bright and virtuous when she is gone; therefore open not your lips in her favor, for the doom which I have passed upon her is irrevocable."

When Celia found she could not prevail upon her father to let Rosalind remain with her, she generously resolved to accompany her; and leaving her father's palace that night, she went along with her friend to seek Rosalind's father, the banished duke, in the forest of Arden.

Before they set out, Celia considered that it would be unsafe for two young ladies to travel in the rich clothes they then wore; she therefore proposed that they should disguise their rank by dressing themselves like country maids. Rosalind said it would be a still greater protection if one of them dressed like a man; and so it was quickly agreed between them, that, as Rosalind was the taller, she would wear the dress of a young countryman, and Celia should be habited like a country lass, and that they should say they were brother and sister. Rosalind said she would be called Ganymede, and Celia chose the name of Aliena.

In this disguise, and taking their money and jewels to defray their expenses, these fair princesses set out on their long travel; for the forest of Arden was a long way off, beyond the boundaries of the duke's dominions.

The lady Rosalind (or Ganymede as she must now be called) with her manly garb seemed to have put on a manly courage. The faithful friendship Celia had shown in accompanying Rosalind so many weary miles made the new brother, in recompense for this true love, exert a cheerful spirit, as if he were indeed Ganymede, the rustic and stout-hearted brother of the gentle village maiden, Aliena.

When at last they came to the forest of Arden, they no longer found the convenient inns and good accommodations they had met with on the road; and being in want of food and rest, Ganymede, who had so merrily cheered his sister with pleasant speeches and happy remarks all the way, now owned to Aliena that he was so weary he could find it in his heart to disgrace his man's apparel, and cry like a woman. And Aliena declared she AREAS OF POLYGONS

book # 11 freeze-dries

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## EXERCISES (GROUP A). AREAS OF RECTANGLES

Find the area of a rectangular lot 50 feet wide and 135 feet long.
Find the cost of sodding a football field 300 feet long and 160 feet wide at 18 cents a square yard.

(5) Find the cost of a field 50 rods wide and 80 rods long at \$80 an acre.

(4) What is the cost of placing clay over a tennis court 36 feet wide and 78 feet long at 20 cents a square yard?

(5) The area of a rectangle is 499.7913 and the base is 26.43. Find the altitude.

6) Find the altitude of a rectangle whose base is 16.5 inches and whose area is 1300.3 square inches.

(7) Find the area of a rectangle whose base is 2x - 1 and whose altitude is x + 4.

Example. Find the dimensions of a rectangle if the width is  $\frac{2}{3}$  of its length 1 and its area is 1574.64.

Solution. The method of finding the square root is as follows: x = the length. Let 8.6 Then  $\frac{2}{3}x =$ the width. 23 61.96  $\frac{2}{3}x^2 = 1574.64$ 4116  $2 x^2 = 4723.92.$ 88 7 61  $2 \times 4 = 8.$  $x^2 = 2361.96.$ 7 04 x = 48.6, length.  $2 \times 48 = 96.$ 966 57 96 57 96  $\frac{2}{3}x = 32.4$ , width.

(8) The altitude of a rectangle is  $\frac{3}{5}$  of its length. Find its dimensions if its area is 2535.

9 The ratio of the width to the length of a rectangular city lot is 3:8. Find the dimensions of the lot if its area is 9600 square feet.

10. Find the area of this cross section of an I-beam if the width of each of the upper and lower rectangles is  $1\frac{3}{8}$  inches.

11. If you double the altitude of a rectangle and leave the base the same, how is the area changed?

12. If both the altitude and base of a rectangle are doubled, how is the area changed?