ACCELERATED AGING TEST FOR PAPER

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ABSTRACT

A series of Government bond and ledger papers was studied with reference to certain properties that appear to be related to permanence. These papers, purchased on specification, were assumed to be representative of the different commercial grades.

A thorough study was made of the physical and chemical properties of the papers as well as of their behavior when exposed to various accelerated aging tests.

The results indicate the feasibility of classifying papers by means of suitable chemical, strength, and accelerated aging tests. The chemical tests include tests for fiber purity and noncellulosic impurities, such as acid and rosin, used in sizing. The fiber purity, which is indicated by the alpha cellulose content and copper number, was found to bear an important relation to the stability of the papers. The accelerated aging test, which consists in exposing the papers to a temperature of 100°C. for 72 hours, and then determining the effect produced on the chemical and physical properties, appears to arrange the papers in about the same order of stability that would be expected on natural aging. A comparison of the results of the above-mentioned aging treatment with results obtained by using more nearly normal conditions (60°C. for 860 hours, and sunlight exposures) shows that they all classified the papers in the same order of stability. These two findings are evidence that the more drastic treatment, which is desirable in respect to convenience and rapidity, gives results in line with more normal aging effects.

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I. INTRODUCTION

In previous work, which involved the comparative study of highly purified wood fiber papers with other common commercial types of papers, it was indicated that the source of the fibers composing a paper did not necessarily determine certain qualities which have an important bearing on its serviceability, and that accurate evaluations of the quality of paper could be made only on the basis of pertinent performance and purity tests.[2]

While the samples employed in the previous tests were probably typical high-grade products of the manufacturers from whom they were obtained, there was some question as to how well this series of papers represented the different commercial grades. It was decided, therefore, that the testing of papers purchased on definite quality specifications would be of value. A series of samples was obtained for study, consisting of 11 bond and ledger papers representative of deliveries on contracts to the Government Printing Office.

The Government, which is a large user of paper of all kinds, purchases paper on a competitive-bid basis, and awards contracts to the lowest bidders. Papers furnished by contractors must conform to certain minimum requirements which are stated in the proposal. These requirements include specifications of source of fiber, strength, and, in the case of the higher grade papers, purity requirements, such as content of rosin and acid; because it is now generally recognized that even the highest-grade paper-making fibers may be ruined at various steps in the paper-making process. While there has been an increasing use of paper-testing methods to aid in the selection of papers for various purposes, few attempts have been made to grade papers on the basis of fiber quality or purity. The following data, and those previously obtained, show, however, that such a basis of classification is feasible.

II. DESCRIPTION OF SAMPLES STUDIED

The samples studied were 11 in number. The bond papers consisted of four grades, classified as 100 per cent sulphite fiber, 30 per cent rag, 50 per cent rag, and 100 per cent rag. The ledger papers, in addition to these grades, included a 75 per cent rag paper. Three different 100 per cent rag ledger papers were tested. All of the papers, with the exception of the 100 per cent sulphite fiber papers, which were beater sized only, were surface sized with starch or glue.

III. TEST METHODS EMPLOYED

1. OFFICIAL METHODS OF THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY[3]

All the strength tests, as well as tests for sizing quality, ash, resin, glue, and starch, were made according to the official T. A. P. P. I. methods.

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[2] Rasch, R. H., A Study of Purified Wood Fibers as a Paper-Making Material, B. S. Jour. Research 3 (RP107), p. 469; September 6, 1929. Price 15 cents. Superintendent of Documents, Washington, D. C.

[3] Copies of the Official Paper-Testing Methods of the Technical Association of the Pulp and Paper Industry may be obtained from the secretary at 18 East Forty-first Street, New York, N. Y.

2. TENTATIVE CHEMICAL TESTING METHODS

(a) ACIDITY

(1) *Kohler-Hall method.*[4]—Two methods for the determination of acidity were employed—the Kohler-Hall method, and a method first proposed by Dr. J. E. Minor, of the Rag Content Manufacturers Association, in which a fiber suspension is swept free of CO_2 and titrated directly. Each method appears to have certain inherent advantages. In the method described by Kohler and Hall 5 g of the ground paper is extracted with 250 ml portions of hot water. The authors express the result as the "total acidity number," which is defined as the number of milliliters of 0.01 normal sodium hydroxide required to make the first three extracts of 10 g of oven-dry fiber neutral to phenolphthalein indicator. For convenience and uniformity, however, results in this paper are expressed as per cent SO_3. In the case of surface-sized papers we have the acidity of the basic paper (internal acidity), and the acidity due to the alum present in the glue or starch surface sizing (external acidity). As stated by Kohler and Hall, the former is harmful from the standpoint of permanence, and the latter is relatively without effect.[5] The important feature of the Kohler-Hall method is that it includes a method for estimating the internal acidity. This is obtained indirectly by determining the external acidity and subtracting it from the total acidity. Kohler and Hall show that a single short extraction of pieces of the unground paper will bring practically all the acid contained in the surface sizing into solution without dissolving any significant amount of the internal acid. The procedure for the determination of the external acidity consists in boiling 10 g of paper, in the form of one-half-inch squares, for two minutes in 250 ml of water. The extract is decanted, cooled, and titrated with 0.01 normal sodium hydroxide solution.

(2) *Acidity of fiber suspension.*—The method for determining the acidity of a fiber suspension appears to offer an advantage over the Kohler-Hall method in that it determines the total titratable acidity present in the fiber, whereas the latter determines what is apparently a variable fraction of the total acidity. The procedure used follows: Weigh out about 1 g of air-dry, ground paper in a small beaker. Heat to boiling 150 ml of distilled water, and moisten the fibers with a little of the hot water. With the aid of a stirring rod and the remainder of the water, transfer the fibers to an Erlenmeyer flask. Heat to boiling and boil for one minute, then set aside until cooled to room temperature. After cooling, bubble a stream of carbon dioxide-free air through the suspension for 15 minutes in order to remove any dissolved carbon dioxide, then titrate with 0.01 normal sodium hydroxide solution, using phenolphthalein indicator. Continue to bubble a slow stream of air through the suspension during the titration. The end point is taken as a pink color which pervades the liquid for one minute. In making the titration the tip of the burette containing the sodium hydroxide solution is inserted through the stopper of the test flask. In order to aid in the distribution of air bubbles, a glass rod reaching to the bottom of the flask is secured to the stopper.

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[4] S. Kohler, and G. Hall, Acidity in Paper, Paper Ind., 7, pp. 1–5; October, 1925.
[5] See footnote 4.

Total acidity, as per cent SO_3, may be calculated by the following formula.

$$\text{Acidity} = \frac{4 \times (ml\ \text{NaOH} - ml\ \text{NaOH for blank}) \times N}{W}$$

where N is the normality of the sodium hydroxide solution, and W is the oven-dry weight in grams of the sample.

(b) COPPER NUMBER

The method employed was essentially that of Schwalbe-Braidy, as described by Clibbens and Geake.[6] The procedure as given by them was followed with a few slight modifications to make it more adaptable for the testing of papers. The changes applied to the original Braidy modification were: (1) Reduction of the sample to a finely ground condition, (2) the use of a 1.5 g sample instead of a 2.5 g sample, (3) the use of the phosphomolybdic acid solution instead of ferric ammonium sulphate in the determination of cuprous oxide. The modified procedure as used at the bureau follows.

(1) *Preparation of solutions.*—Copper sulphate solution: Dissolve 100 g $CuSO_4.5H_2O$ in distilled water, and make up to 1 liter.

Sodium carbonate—sodium bicarbonate solution: Dissolve 50 g $NaHCO_3$ and 350 g $Na_2CO_3.10H_2O$ in distilled water, and make up to 1 liter.

Phosphomolybdic acid solution: Dissolve 100 g of sodium molybdate ($Na_2MoO_4.2H_2O$) and 75 ml of phosphoric acid (83 per cent) in a solution containing 275 ml of concentrated sulphuric acid and 1,750 ml of water.

Sodium carbonate solution: Make up a solution containing approximately 5 per cent Na_2CO_3.

Potassium permanganate solution: Make up a solution having a normality of 0.05.

(2) *Procedure.*—Immediately before use add 5.0 ml of the copper sulphate solution to 95 ml of the sodium carbonate-sodium bicarbonate solution. Bring the mixture to a boil, and pour over 1.5 g of the finely ground sample in a 125-ml Erlenmeyer flask. Stir well with a glass rod in order to distribute the fibers and to remove air bubbles. Fit the flask with a Bunsen valve and submerge completely in a steam bath at atmospheric pressure. Occasionally fibers tend to float to the surface, therefore the flask should be shaken from time to time in order to redistribute them. Remove the flask from the steam bath at the end of three hours. Filter on a filter paper in a Büchner funnel, using suction. Wash with 100 ml of 5 per cent sodium carbonate solution, and then with 250 ml of hot water. Transfer fiber and filter paper to a small beaker, add 25 ml of the molybdate solution, and macerate well with a flattened glass rod. Transfer to a Büchner funnel again and wash thoroughly with cold water until the blue molybdenum color is removed from the fibers. Titrate the filtrate with 0.05 normal potassium permanganate to a faint pink.

The copper number is defined as the number of grams of copper in the cuprous oxide reduced from the cupric hydroxide solution by 100 g of the sample. It may be calculated from the following formula.

$$\text{Copper number} = \frac{6.357 \times ml\ KMnO_4 \times N}{W}$$

[6] Text. Inst., Trans., *15*, pp. 27-38; 1924.

TABLE 1.—*Data o*

Sample No.	Lot No.	Fiber composition		Physical tests [1]						
				ding endurance at 50 er cent relative umidity.			Tensile strength			
		Rag	Chemical wood	a-ne ec-n	Across machine direction	Average	Machine direction	Across machine direction	Average	M ch di ti
		Per cent	*Per cent*	*ble ds*	*Double folds*	*Double folds*	*kg*	*kg*	*kg*	*Per*
A-O	102	0	10	212	201	207	7.0	3.9	5.5	-
A-H72							5.7	3.3	4.5	2
B-O	110	30	7	588	381	485	7.5	3.9	5.7	-
B-H72							7.3	3.6	5.5	1
C-O	121	50	5	585	405	495	7.5	4.1	5.8	-
C-H72							7.4	4.1	5.8	7
D-O	128	100		859	2,375	2,617	11.3	7.3	9.3	-
D-H72							10.3	6.7	8.5	7
E-O	151	0	10	693	123	408	10.7	5.1	7.9	-
E-H72							8.7	4.7	6.7	6
F-O	154	40	6	487	333	410	9.6	5.7	7.7	-
F-H72							9.1	5.2	7.2	8
G-O	161	55	4	635	311	473	13.3	6.8	10.1	-
G-H72							12.3	6.4	9.4	2
H-O	169	85	1	179	862	2,021	13.1	7.4	10.3	-
H-H72							12.8	7.0	9.9	7
I-O	176	100		055	1,439	1,747	13.6	8.3	11.0	-
I-H72							13.0	7.9	10.5	5
J-O	178	100		123	1,302	2,213	14.2	8.8	11.5	-
J-H72							13.7	8.3	11.0	6
K-O	177	100		035	3,375	3,705	14.5	7.6	11.1	-
K-H72							14.6	7.6	11.1	8

[1] Physical tests made at 6 al acidity equals total acidity minus external acidi

where N is the normality of the potassium permanganate solution and W is the oven-dry weight of the sample.

The amount of copper in the cuprous oxide reduced from the reagent alone under the conditions of the method should be determined and applied as a correction to the copper number.

(c) ALPHA-CELLULOSE CONTENT

Alpha cellulose determinations were made according to the procedure outlined in a previous publication.[7]

3. ACCELERATED AGING TESTS

(a) OVEN TESTS

In order to obtain information on relative stability, all the paper samples included in this study were subjected to a heat treatment at 100° C. for 72 hours in a special air-circulating oven, described in a previous publication.[7] The stability is estimated by a consideration of the effect of this treatment on the strength properties of the paper, as well as on the alpha cellulose content and copper number.

A criticism commonly directed at the above-mentioned method of estimating the permanence of papers is that it subjects papers to exaggerated conditions which never obtain under natural aging. Therefore a few supplementary experiments were made in which papers were subjected to a temperature of 60° C. for various lengths of time, in order to determine whether a much less drastic treatment of this type would classify papers in the same order of stability as the oven treatment at higher temperature. Other experiments were made in which papers were subjected to temperatures of 90° and 120° C., respectively.

(b) SUNLIGHT EXPOSURES

In order to compare the effects of sunlight and oven heat treatments, a few of the papers were exposed, side by side, to the direct rays of the sun for 100 sun-hours on each surface.

IV. RESULTS

1. STRENGTH TESTS

Results of strength tests are given in Table 1. Except in one case all these tests were made at 65 per cent relative humidity, the folding endurance being determined at 50 per cent relative humidity as well. In general, no great differences in original strength are noted among papers ranging from 0 to 50 per cent in rag fiber content. The 100 per cent rag bonds and ledgers are, however, markedly higher in strength than the other papers, reflecting, undoubtedly, the superior quality of fibers in these papers, relative to that of the other papers included in this study.

2. PERMANENCE TESTS

(a) CHEMICAL PURITY TESTS

(1) *Copper number.*—The copper number appears to be the most convenient and best single method for estimating the purity of the

[7] See footnote 2, p. 466.

Table 1.—*Data on bond and ledger papers*

Sample No.	Lot No.	Fiber composition: Rag	Fiber composition: Chemical wood	Kind of sizing used	Aging test: Temperature	Aging test: Duration	Physical tests [1]: Weight 500 sheets 25 by 40 inches	Thickness	Bursting strength	Ratio bursting strength to weight	Folding endurance: Machine direction	Folding endurance: Across machine direction	Folding endurance: Average
		Per cent	Per cent		°C.	Hours	Pounds	Inch	Points [6]	Per cent	Double folds	Double folds	Double folds
A-O	102	0	100	Engine sizing	(7)	-------	53.1	0.0040	29.2	55	484	191	338
A-H72					100	72	52.9	.0039	19.7	37	3.4	7.3	5.4
B-O	110	30	70	Engine and tub sizing.	(7)	-------	42.6	.0030	29.6	69	630	239	435
B-H72					100	72	44.1	-------	26.6	60	149	104	127
C-O	121	50	50	do.	(7)	-------	43.1	.0031	30.9	72	726	321	524
C-H72					100	72	43.9	-------	30.4	69	346	154	250
D-O	128	100	0	do.	(7)	-------	58.1	.0048	63.0	108	4,039	2,870	3,455
D-H72					100	72	58.0	-------	58.4	101	2,154	1,462	1,808
E-O	151	0	100	Engine sizing	(7)	-------	92.7	.0064	43.1	47	1,529	220	925
E-H72					100	72	92.4	.0064	27.8	30	1.0	1.6	1.3
F-O	154	40	60	Engine and tub sizing.	(7)	-------	78.3	.0055	45.3	58	867	272	570
F-H72					100	72	77.8	.0056	30.5	51	177	84	131
G-O	161	55	45	do.	(7)	-------	85.6	.0055	57.6	67	951	361	656
G-H72					100	72	85.1	-------	50.9	60	126	94	110
H-O	169	85	15	do.	(7)	-------	88.4	.0061	61.9	70	4,576	705	2,641
H-H72					100	72	88.3	-------	51.6	70	1,809	532	1,171
I-O	176	100	0	do.	(7)	-------	68.5	.0047	77.9	114	3,344	2,663	3,004
I-H72					100	72	68.5	-------	71.4	104	2,692	2,199	2,446
J-O	178	100	0	do.	(7)	-------	88.1	.0064	75.5	86	3,876	2,112	2,994
J-H72					100	72	88.3	-------	73.0	83	2,506	993	1,750
K-O	177	100	0	do.	(7)	-------	78.3	.0052	71.1	91	6,426	4,384	5,405
K-H72					100	72	77.8	-------	69.0	89	5,269	3,329	4,299

Sample No.	Folding endurance in per cent of initial: Machine direction	Across machine direction	Average	Folding endurance at 50 per cent relative humidity: Machine direction	Across machine direction	Average	Tensile strength: Machine direction	Across machine direction	Average	Elongation: Machine direction	Across machine direction	Average	Elongation in per cent of initial: Machine direction	Across machine direction	Average
	Per cent	Per cent	Per cent	Double folds	Double folds	Double folds	kg	kg	kg	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
A-O	-------	-------	-------	212	201	207	7.0	3.9	6.5	2.8	6.8	4.8	-------	-------	-------
A-H72	1	4	3	-------	-------	-------	5.7	3.3	4.5	1.4	3.0	2.7	50	57	54
B-O	-------	-------	-------	588	381	485	7.5	3.9	5.7	2.6	6.7	4.7	-------	-------	-------
B-H72	24	44	34	-------	-------	-------	7.3	3.6	5.5	2.4	5.5	4.0	92	82	87
C-O	-------	-------	-------	585	405	495	7.6	4.1	6.8	2.7	7.0	4.9	-------	-------	-------
C-H72	48	48	48	-------	-------	-------	7.4	4.1	5.8	2.6	5.4	4.0	93	77	85
D-O	-------	-------	-------	2,859	2,375	2,617	11.3	7.3	9.3	5.2	7.6	0.4	-------	-------	-------
D-H72	53	51	52	-------	-------	-------	10.3	6.7	8.5	4.6	7.0	5.8	88	02	90
E-O	-------	-------	-------	693	123	408	10.7	5.1	7.9	2.6	5.0	3.8	-------	-------	-------
E-H72	0	1	1	-------	-------	-------	8.7	4.7	6.7	1.4	3.0	2.2	66	60	58
F-O	-------	-------	-------	487	333	410	9.6	5.7	7.7	3.4	5.4	4.9	-------	-------	-------
F-H72	20	31	26	-------	-------	-------	9.1	5.2	7.2	3.0	4.9	4.0	88	77	83
G-O	-------	-------	-------	635	311	473	13.3	6.8	10.1	3.4	7.4	5.4	-------	-------	-------
G-H72	13	26	20	-------	-------	-------	12.3	6.4	9.4	2.9	6.0	4.5	85	81	83
H-O	-------	-------	-------	3,179	862	2,021	13.1	7.4	10.3	4.3	7.4	5.9	-------	-------	-------
H-H72	40	76	58	-------	-------	-------	12.8	7.0	9.9	3.9	7.2	5.6	91	97	94
I-O	-------	-------	-------	2,055	1,439	1,747	13.6	8.3	11.0	4.8	8.4	6.6	-------	-------	-------
I-H72	80	83	82	-------	-------	-------	13.0	7.9	10.5	4.1	7.8	0.0	85	93	89
J-O	-------	-------	-------	3,123	1,302	2,213	14.2	8.8	11.5	4.2	8.5	6.4	-------	-------	-------
J-H72	65	47	56	-------	-------	-------	13.7	8.3	11.0	4.1	7.9	6.0	98	96	97
K-O	-------	-------	-------	4,035	3,375	3,705	14.6	7.6	11.1	3.9	8.7	6.3	-------	-------	-------
K-H72	82	76	79	-------	-------	-------	14.6	7.6	11.1	4.0	7.9	6.0	103	91	97

Sample No.	Tearing strength: Machine direction	Across machine direction	Average	Tearing strength in per cent of initial: Machine direction	Across machine direction	Average	Degree of sizing: Indicator (21° C.)	Curl (21° C.)	Chemical tests: Sizing materials present: Resin	Glue	Starch	Ash	Acidity [2], Kohler-Hall method: Total	External	Internal [3]	Acidity of fiber suspension	Alpha cellulose content [4]	Decrease in alpha cellulose in per cent of initial	Alpha-cellulose content corrected for sizing materials present [5]	Copper number	Increase in copper number
	g	g	g	Per cent	Per cent	Per cent	Seconds	Seconds	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent		
A-O	57.0	51.0	54.0	-------	-------	-------	20.6	23.4	0.9	0	Trace.	0.77	0.126	-------	-------	0.185	78.3	-------	70.0	3.51	-------
A-H72	23.4	20.8	22.1	41	41	41											72.1	7.0	-------	4.43	0.92
B-O	39.8	39.8	39.8	-------	-------	-------	9.6	15.4	1.0	0	5.42	1.00	.104	0.019	0.085	.223	77.5	-------	83.0	2.50	-------
B-H72	35.5	34.0	34.8	89	85	87											75.3	2.9	-------	3.01	.51
C-O	45.0	45.0	45.0	-------	-------	-------	10.1	15.0	1.7	2.56	-------	1.15	.110	-------	-------	.208	77.0	-------	81.0	2.50	-------
C-H72	38.8	38.8	38.8	86	86	86											74.7	3.8	-------	2.87	.37
D-O	104.0	96.0	100.0	-------	-------	-------	14.3	23.3	1.3	7.34	Trace.	.69	.188	.087	.101	.204	85.4	-------	93.5	0.99	-------
D-H72	90.0	86.0	88.0	87	90	89											84.1	1.5	-------	1.46	.47
E-O	144.0	130.8	137.4	-------	-------	-------	33.9	51.1	1.0	0	0	.60	.144	-------	-------	.202	79.0	-------	80.0	3.83	-------
E-H72	54.2	49.4	51.8	38	38	38											72.4	8.3	-------	4.49	.66
F-O	112.4	99.6	106.0	-------	-------	-------	24.1	34.1	2.1	0	2.49	.59	.098	.012	.086	.185	78.7	-------	82.5	2.47	-------
F-H72	86.0	76.8	81.4	77	77	77											74.5	5.4	-------	3.15	.68
G-O	123.6	111.6	117.6	-------	-------	-------	28.7	41.4	1.7	2.05	Trace.	.68	.108	.032	.076	.188	76.9	-------	80.0	2.35	-------
G-H72	100.4	86.4	93.4	81	78	80											73.7	4.2	-------	2.87	.52
H-O	160.8	147.0	154.2	-------	-------	-------	24.2	37.9	1.8	1.94	1.23	.68	.066	.010	.056	.103	83.5	-------	88.0	1.61	-------
H-H72	140.8	124.8	132.8	88	85	87											80.7	3.4	-------	1.78	.17
I-O	128.8	112.4	120.6	-------	-------	-------	16.0	24.0	1.4	7.05	Trace.	.75	.142	.038	.104	.147	82.8	-------	90.5	1.80	-------
I-H72	106.4	108.4	107.4	83	96	90											81.9	1.0	-------	2.05	.25
J-O	170.2	169.2	174.2	-------	-------	-------	20.1	28.4	1.6	4.05	2.51	.52	.140	.058	.082	.171	87.8	-------	95.5	0.96	-------
J-H72	169.2	140.0	154.6	94	83	89											86.8	1.1	-------	1.32	.36
K-O	178.4	164.0	171.2	-------	-------	-------	18.1	31.2	0.8	3.62	Trace.	.96	.152	.031	.121	.163	90.1	-------	94.5	1.01	-------
K-H72	163.0	146.0	154.8	92	89	91											89.3	0.9	-------	1.09	.08

[1] Physical tests made at 65 per cent relative humidity except as noted.
[2] Acidity expressed as per cent SO_3.
[3] Internal acidity equals total acidity minus external acidity.
[4] Not corrected for sizing materials present.
[5] Values to nearest 0.5 per cent.
[6] Approximately pounds per square inch.
[7] Control sample.

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basic fibers in papers. The relation between the different grades of papers and their copper numbers is quite straightforward in this series. Thus, the two all-sulphite fiber papers, the lowest grade included in this series, have the highest copper numbers, while the highest grade papers, the 100 per cent rag papers, have the lowest copper numbers. Intermediate grades correspond with copper numbers of intermediate value.

(2) *Alpha-cellulose content.*—The alpha-cellulose contents of the papers when corrected for sizing materials present classify the samples in approximately the same order of chemical purity as the copper numbers do. As will be seen in Figure 1, there is a distinct tendency for high copper numbers to be associated with low alpha cellulose contents, and vice versa. It is important that corrections for sizing

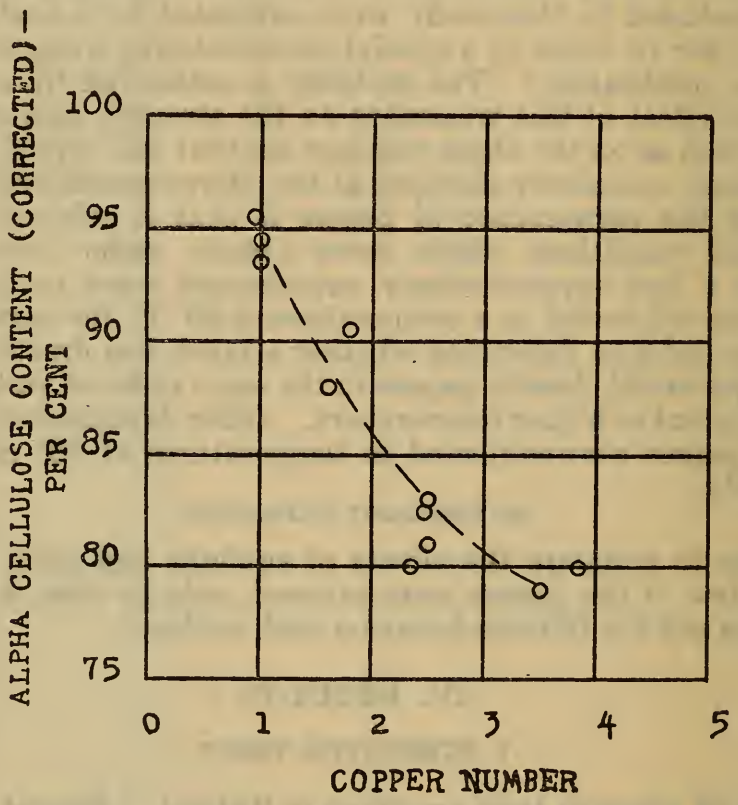

FIGURE 1.—*Relation between alpha-cellulose content and copper number*

materials present in the paper be applied to the alpha cellulose values before conclusions are drawn regarding the purity of the fibers. In this paper corrected alpha cellulose values are reported only to the nearest one-half per cent, to allow for cumulative errors, which are unavoidably introduced when applying corrections for sizing materials.

(3) *Acidity.*—The relation between the acidity of a fiber suspension, and the "total acidity" by the Kohler-Hall extraction method is apparently not a constant one for different papers. The ratio of the results by the former method to results by the latter method varies with different papers anywhere from 1 to 2. Acidities of the all-sulphite fiber papers, as determined by the Kohler-Hall method, are noticeably higher than the internal acidities of the rag content, surfaced-sized papers, as is to be expected.

(4) *Rosin.*—It is noteworthy that the rosin contents of the papers, particularly the two 100 per cent sulphite fiber papers, are relatively low, revealing a laudable tendency among paper manufacturers to reduce the amount of rosin used to just that required for adequate sizing.

3. ACCELERATED AGING TESTS

(a) OVEN TREATMENT AT 100° C. FOR 72 HOURS

In Table 1 the effects of the heat treatment at 100° C. on the strength and chemical properties of the papers are shown. If we

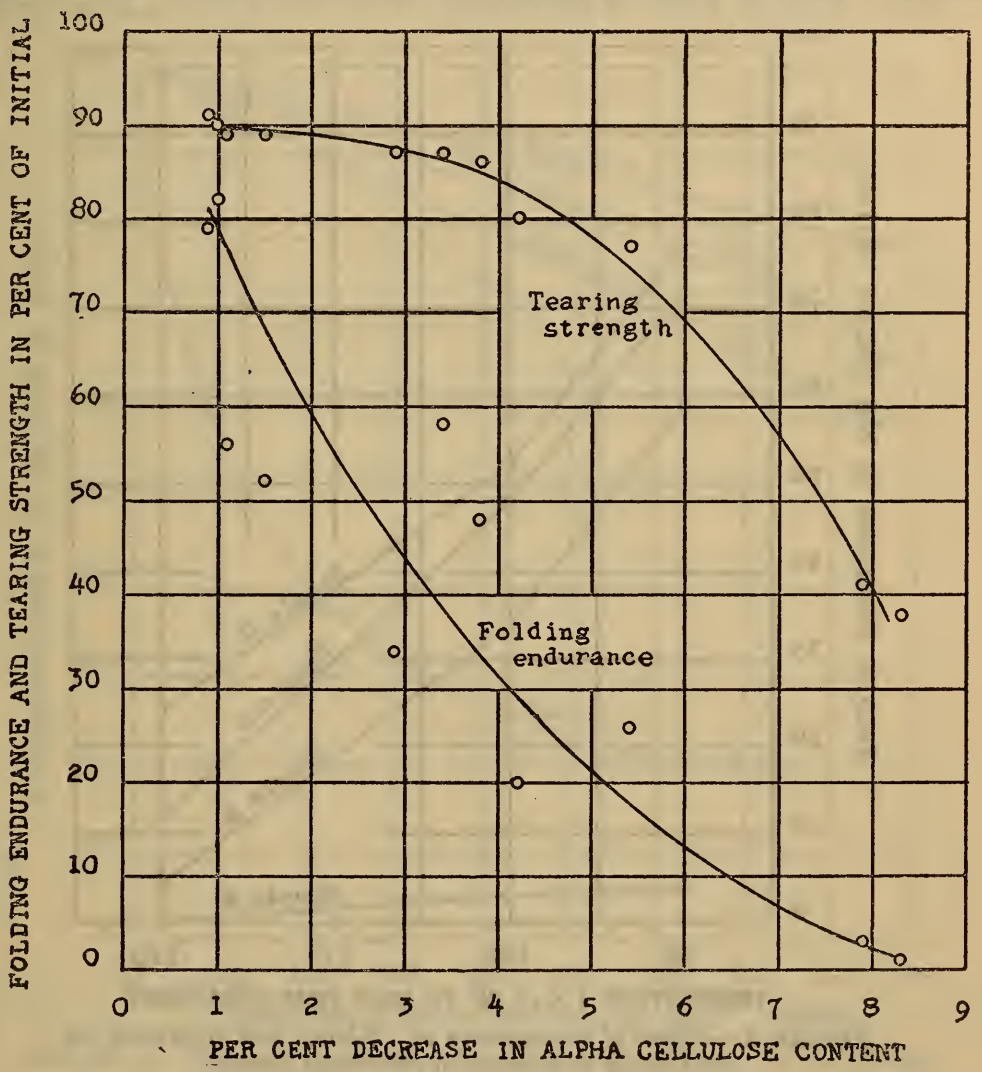

FIGURE 2.—*Relation between effects produced by heating on the strength and alpha-cellulose content*

assume that the papers represented in the highest grade are the most permanent, that those in the lowest grade are the least permanent, and that the intermediate grades occupy an intermediate position in regard to permanency, it is evident from the test data that the heat treatment rates the papers of this series in the same order as normal aging would. The assumptions made above are in accord with the purposes for which the Government paper specifications were designed, and with the general consensus of present opinion regarding factors which influence permanence. Thus, it is well

known that paper carefully prepared from new white rags has lasted for centuries. Specifications for Government papers require that those intended for permanent uses shall be made of "100 per cent new rags, white, cream, or unbleached." This and the other requirements for strength, acidity, and sizing make it probable that this grade of paper is, for all practical purposes, permanent. The lowest grade of Government bond and ledger paper is made from chemical wood pulp prepared by the usual sulphite process. While numerous examples are available of sulphite papers which have resisted deterioration a score or more of years, and while their lasting qualities are

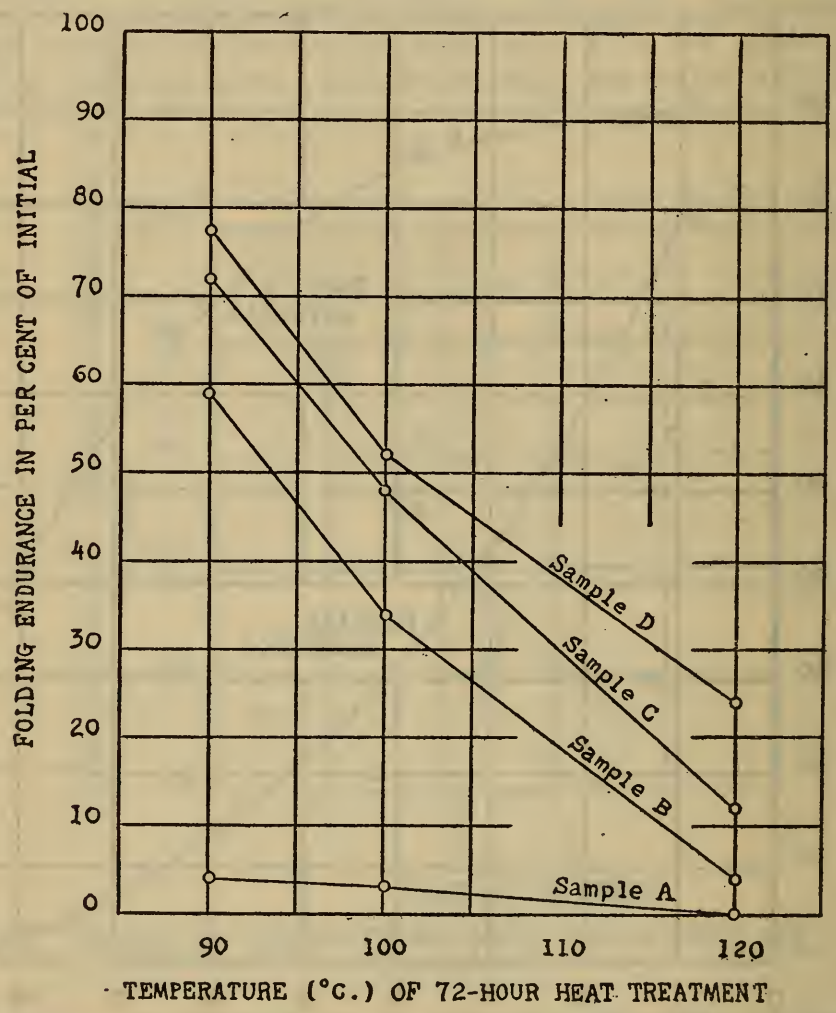

FIGURE 3.—*Effect of temperature of 72-hour heat treatment on the folding endurance of four bond papers*

frequently underestimated, it is safe to say that the ordinary types of such papers stand lower on a relative scale of permanency than those made from purer grades of paper-making fibers.

The test results show that the stability of the papers toward the heat test is related to their chemical purity. Samples A and E were found to be composed of fibers having the highest copper numbers, and the lowest alpha cellulose content, pointing to a lower degree of purity relative to the other papers in the series. Samples D, H, I, J, and K, on the other hand, had the lowest copper numbers and the highest alpha cellulose values. On comparing the effects produced on the papers by heating them for 72 hours at 100° C., it will be noted

that A and E retain but 3 and 1 per cent of their initial folding endurances, respectively, while D, H, I, J, and K retain 52, 58, 82, 56, and 79 per cent, respectively. In the case of the tearing strengths the retentions of these papers are, respectively, 41 and 38, on the one hand, and 89, 87, 90, 89, and 91, on the other. The decreases in alpha cellulose content for the same papers are, in order, 7.9 and 8.3, as against 1.5, 3.4, 1.0, 1.1, and 0.9. The data show that the heat treatment produces a definite alteration in the chemical as well as the physical properties of the cellulose fibers. Figure 2 shows the relation between the decrease in alpha cellulose content and the corresponding decrease in folding endurance and tearing strength.

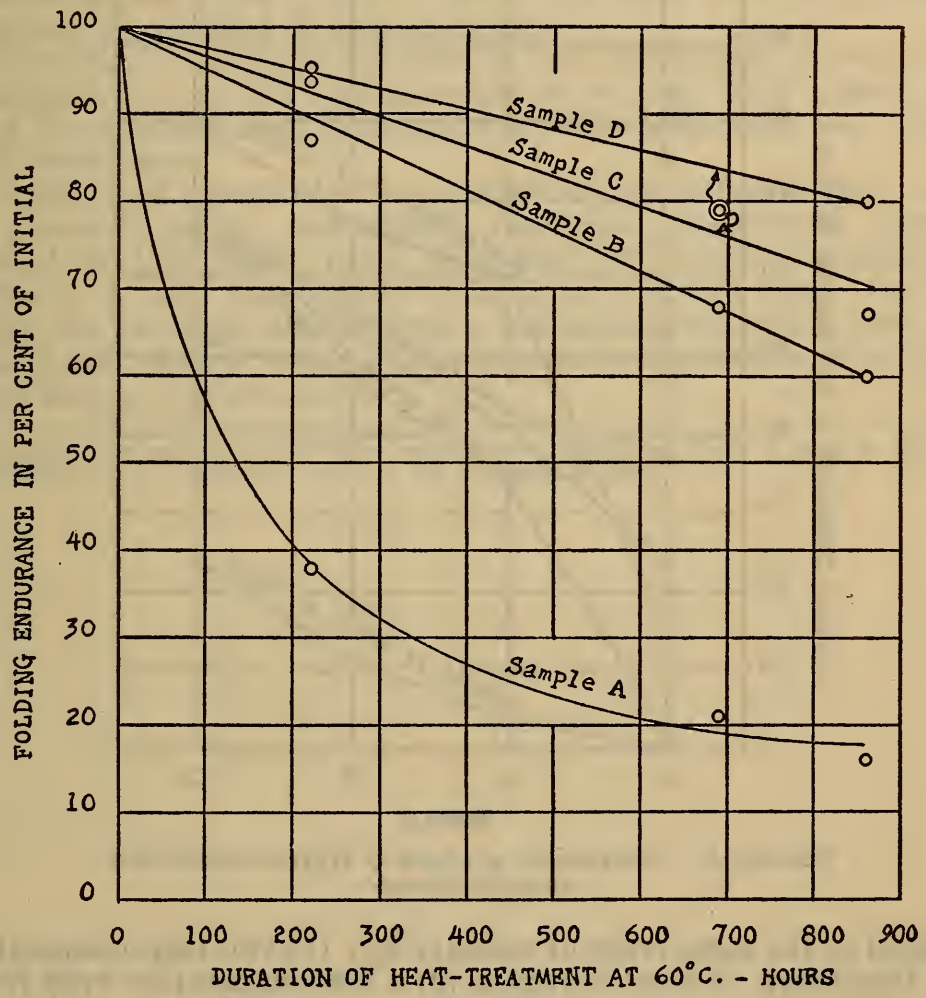

FIGURE 4.—*Effect of duration of heat treatment at 60° C. on the folding endurance of four bond papers*

(b) SPECIAL OVEN TREATMENTS INVOLVING VARIATIONS OF TEMPERATURE AND DURATIONS OF TREATMENTS

Treatments for 72 hours at 90° C. and 120° C. as well as at 100°C. were made on the four bond papers. The effect of the treatments on the folding endurance is shown graphically in Figure 3.

Next, treatments of the same papers at the comparatively low temperature of 60° C. were carried out for 240, 692, and 860 hours, respectively. Exposures for such long durations were necessary in order to obtain noticeable effects. Treatments at this temperature are much less drastic than at 100° C., and yet the papers rate in the same order of stability. The results are shown graphically in Figure 4.

(c) SUNLIGHT EXPOSURES

Another stability test tried consisted in the exposure of these papers to the action of the sun's rays. Here, too, the effects on folding endurance rate the papers in the same order of stability as the heat tests do.

In Figure 5 a comparison of the effects of the various treatments on the folding endurances of the four papers is shown graphically. The fact of significance shown in Figure 5 is that the four papers, probably covering a fairly wide range of permanence qualities, are

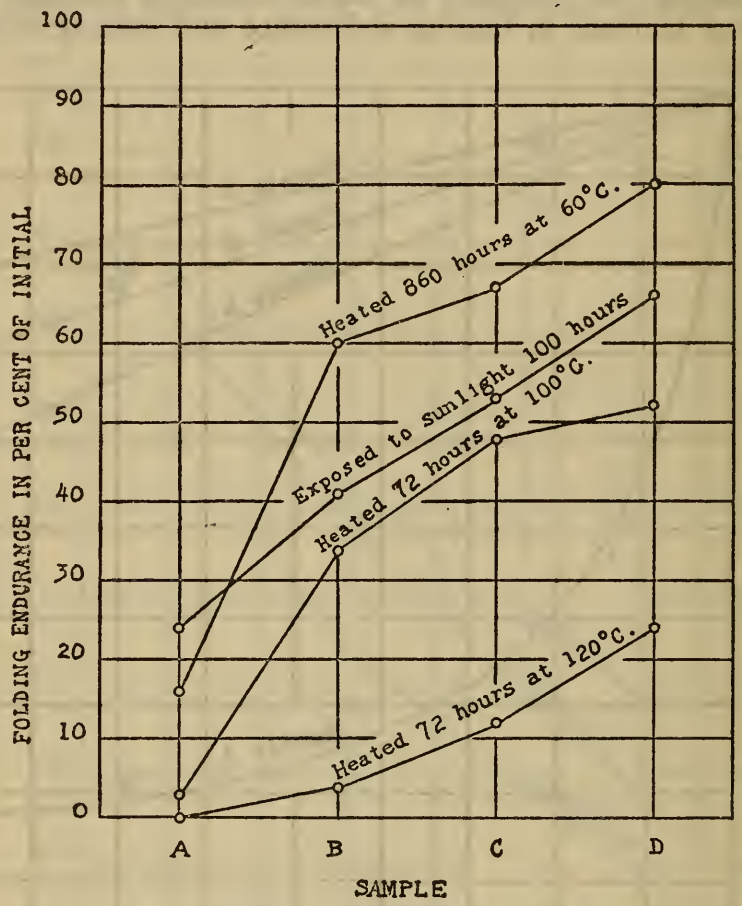

FIGURE 5.—*Comparison of effects of different accelerated aging treatments*

arranged in the same order of stability by: (1) two high-temperature oven treatments of short duration, (2) a low-temperature oven treatment of long duration, and (3) an exposure to direct sunlight.

4. CLASSIFICATION OF PAPERS

From a consideration of these results, and the others previously reported, then, it appears that the various chemical and physical tests mentioned agree in their order of classification of the various papers with respect to their deterioration under the test conditions, when the folding endurance, tearing strength and alpha cellulose tests are used as an index of this deterioration. Furthermore, this order is substantially the same as that of the life expectancy of the papers in so far as this can be inferred from the information available. On this assumption two of the attributes of a permanent paper appear

to be low copper number and high alpha cellulose content. The heat test seems to be a promising means of estimating the probable relative life of a paper. Recently a system of classification of printing and writing papers has been suggested, in which performance tests and purity tests alone are the determining factors in grouping papers according to their aging properties.[8]

V. SUMMARY

1. An accelerated aging test, which determines the effect of a 72-hour heat treatment at 100° C. on various physical and chemical properties arranged the papers in about the same order of stability that would be expected on natural aging.

2. The effect of sunlight exposure, or heat treatments of relatively low temperature, on the folding endurance of four bond papers arranged the papers in the same order of stability as the more convenient 100° C. treatment.

3. Samples of papers have been examined for strength, fiber content, chemical purity, and general condition. Samples of papers described in this report have been filed for future examination. Eventually it may be possible to build up evidence upon which a decision can be based as to whether the accelerated aging test described herein does or does not rate papers in accordance with their relative degree of permanence.

[8] B. W. Scribner, Permanence Standards for Printing and Writing Papers, Trans. A. S. M. E., **52**, No. 19, PI-52-5; May-August, 1930; also Paper Mill., **53**, p. 13; June 21, 1930.