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# PAPER TESTING METHODS

Microscopical, Chemical, and Physical Processes  
Described, with the Apparatus  
Employed

*By*

**Committee on Paper Testing**

Technical Association of the Pulp and  
Paper Industry



Price \$3.00

Published by the

**Technical Association of the Pulp and Paper Industry**

18 East 41st Street, New York City

1922

(Reprinted 1924)

The following list of books and pamphlets consists of works which should be on the desk or in the library of every paper mill executive. It includes the best works dealing with American practice, with a few of European origin that will be of general interest.

**HISTORY OF PAPER MANUFACTURING IN THE UNITED STATES**, by Lyman Horace Weeks (1916). An account of the origin and development of paper manufacture in the United States from 1690 to 1916. (Lockwood Trade Journal Co., 10 East 39th St., New York, \$6.)

**PULPWOOD AND WOOD PULP**, by Royal S. Kellogg (1923). A short treatise on the basic raw material for paper with statistical data on production and consumption in North America. (McGraw-Hill Book Co., 370 Seventh Ave., New York, \$4.)

**MANUFACTURE OF PULP AND PAPER**, in five volumes, edited by J. N. Stephenson. Vols. I and II deal with the sciences in their relation to the manufacture of pulp and paper (1921). Vol. III, Preparation and Treatment of Wood Pulp (1922). Vol. IV, Preparation of rags and waste papers; beating, sizing and coloring; paper machines. Vol. V, in preparation (to be issued in 1924). Manufacture of paper and general mill equipment. (Technical Association of the Pulp and Paper Industry, 18 East 41st St., New York, \$5 per vol. McGraw-Hill Book Co., publishers.)

**MODERN PULP AND PAPER MAKING**, by G. S. Witham, Sr. (1920). A practical American treatise which gives particulars of every phase of papermaking from the saw mill to the finishing room. (Chemical catalog Co., 170 Metropolitan Tower, New York, \$6.)

**A TEXT BOOK OF PAPERMAKING**, by C. F. Cross and E. J. Bevan. Fifth edition, containing additional matter, and in part rewritten with collaboration of J. F. Briggs. (Spon & Chamberlain, 120 Liberty St., New York, \$9.)

**THE ACTION OF THE BEATER**, by Dr. Sigurd Smith (1923). An exhaustive treatment of the subject. Published by British Technical Section. (Distributed in U. S. by Technical Association of the Pulp and Paper Industry, 18 East 41st St., New York, \$3.60.)

**THE RECOVERY AND MANUFACTURE OF WASTE PAPER**, by James Strachan (1918). This is a most useful and suggestive work which can be recommended to all practical paper makers. (Spon & Chamberlain, 120 Liberty St., New York, \$4.50.)

**THE TREATMENT OF PAPER FOR SPECIAL PURPOSES**, by Louis Edgar Andes (1907). The processes of manufacture for a variety of paper products are outlined in this book of 239 pages. It is not a

working manual, though it contains a collection of formulas and furnishes for parchment and greaseproof papers, as well as for a whole series of novelty papers. (D. Van Nostrand Co., 8 Warren St., New York, \$3.)

**CHEMISTRY OF PULP AND PAPER MAKING**, by Edwin Sutermeister (1920). A practical book for paper mill chemists, superintendents and students. It is based on notes and experiences of the author during a long term of service as chemist in the industry, as well as study of the literature relating to the subject. It assumes a moderate knowledge of chemistry on the part of the reader. (John Wiley & Sons, New York, \$6.)

**CHEMISTRY OF PAPERMAKING**, by Griffin and Little (1894). This book has been out of print for some time. A photographic reproduction of it was issued from Switzerland some time ago when a copy was procured from the Baker Company of Cleveland, Ohio. (G. E. Stechert & Co., 31 East 10th St., New York, \$8.)

**TECHNICAL METHODS OF ANALYSIS**, by Roger C. Griffin (1921). A good work for analytical chemists. (McGraw-Hill Book Co., 370 Seventh Ave., New York, \$6.)

**THE PAPER MILL CHEMIST**, by Henry P. Stevens (1907). (D. Van Nostrand Co., 8 Warren St., New York, \$4.)

**PAPER TESTING METHODS**, by the Committee on Paper Testing, TAPPI (revised 1922). A practical treatise on the analysis of paper. (Technical Association of the Pulp and Paper Industry, 18 East 41st St., New York, \$3.)

**FROM PAPER MILL TO PRESSROOM**, by William Bond Wheelwright (1920). (George Banta Publishing Co., Menasha, Wis., \$2.)

**PHILLIPS' PAPER TRADE DIRECTORY OF THE WORLD**, by S. Charles Phillips. Issued yearly. (S. Charles Phillips & Co., 47 Cannon St., E.C., London.)

**LOCKWOOD'S DIRECTORY OF THE PAPER AND ALLIED TRADES**. Issued annually, in September. Contains a directory of paper and pulp mills in United States and Canada, and classified lists of paper and mill products, besides addresses of paper and board merchants, twine manufacturers and wall paper printers. There is also a useful reference list of watermarks and brand names. (Lockwood Trade Journal Co., 10 East 39th St., New York, \$7.)

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New York

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Technical Association of the Pulp and Paper Industry

New York



TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

18 East 42nd Street, New York, City

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## Annual Report of the Committee on Paper Testing Presented at the Annual Meeting of the Technical Association of the Pulp and Paper Industry, April 11, 1922

Since the last publication of the methods proposed by this committee, there have been a number of methods and devices developed for testing paper. There seems to be a growing realization that there are not a sufficient number of good methods or apparatus for testing paper. The two notable instances of improvements will be found in the interest in a sizing test and in the tearing strength quality.

In view of the above facts, it was thought desirable to revise the Paper Testing Methods as a whole. Certain of the data have been rearranged to make it more convenient and there have been added a contents, list of illustrations, bibliography and an index. It is believed that these additions will make these methods of considerably greater value because of their increased convenience.

Minor changes have been made in some of the methods or formulæ and an attempt has been made to make the methods and illustrations as up-to-date as possible. In addition, a number of methods have been added which will widen the field of use of the Paper Testing Methods. The additional methods are:

1. Sutermeister's calcium chloride stain for microscopic work.
2. Lofton-Merritt stain for unbleached sulphate.
3. Relation between relative humidity and certain physical qualities of paper.
4. Methods for determining the machine and cross direction and also the wire and felt side.
5. Factors for conversion to and from the metric system and also for computing roll lengths.
6. Wet tensile test.
7. Stress-strain test for heavy bag paper, indicating stretch under load.
8. Additional methods for absorption.
9. Tearing test; a brief discussion of five methods or apparatus.
10. Conductivity or electrolytic method for measuring the sizing quality of paper.
11. Volumetric composition of paper.
12. Method for measuring the number of conducting particles in thin paper.
13. Resistance to water penetration.
14. Sulphur in paper.
15. Tarnishing test.

Throughout the report, wherever the author, inventor or originator of a method or apparatus is known, reference is made and credit is given. Wherever data or information are taken directly from a publication, reference is made to the corresponding number of the bibliography.

In reference to the investigation of microscopic examination of fibers and the bursting strength of paper, as proposed by the committee last year, it was not possible to include the results in this report. Data have been received from about half of the co-operating laboratories, but it is planned to complete the study and publish the results later. The indications are, however, that the various laboratories do not get as close check results as would be expected.

It is believed that the Paper Testing Committee has arrived at a place where a change of policy is desirable. In the past, this committee has suggested and proposed various methods of testing

which, in its judgment, were of value in determining the quality of paper. It is thought desirable that TAPPI authorize this committee to investigate the following subjects with the view to putting paper testing on a more scientific basis. With the active co-operation of various laboratories equipped for paper testing, it is believed that data can quickly be collected to permit of more definite official methods of testing.

1. A survey be made of the various laboratories with a view to determining the extent of paper testing equipment available.
2. The development of standard official methods of the Association to be used by its members.
3. A study of the relation of the various physical tests of paper and an attempt made to reach positive conclusions.
4. A study of the proper meaning or interpretation of the various tests and an attempt to reduce the tests (chiefly physical) to some fundamental units.
5. The determination of the proper test for a particular use.

In conclusion, it is thought that paper testing should be put on a firmer basis and that this Association is responsible to the paper industry for the development of methods of measuring the quality of the paper and it is hoped that this committee will be permitted to follow the program suggested above.

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# PAPER TESTING METHODS

## Microscopical, Physical and Chemical Processes

### I. PAPER TESTING

#### 1. Purpose

The testing of paper is performed for three reasons and it is possible that methods suitable for one purpose may not be suitable for another. These purposes are: (a) to study the manufacture in order to improve the quality, (b) to maintain a predetermined quality, and (c) to determine whether the quality is equal to a predetermined standard or specification. The manufacturer is interested chiefly in (a) and (b), while the user or buyer is interested in (c), when paper is bought on specification. It is obvious that various methods may be developed for use in mills that are entirely satisfactory for the development of quality and for maintaining that quality. It is thought, however, that the methods used by testing laboratories in connection with the purchase of paper on specifications, should be so defined and standardized that comparable results will be obtained by different laboratories. The methods herewith given are in some cases merely tentative suggestions which can not be accepted as standard without further investigation. It must be understood, however, that in determining what tests to make, that the purpose for which the paper is to be used is of primary importance, and that that test should be used which will indicate the quality that is specifically desired.

#### 2. Development

Paper testing has developed rather slowly in this country and many of the methods are of foreign extraction, as are some of the instruments and apparatus. However, a considerable amount of development has taken place, and there are a greater number of methods now available. This development has not, however, been in any systematic manner and has been spread over the whole field of testing, to meet special conditions. A systematic study should be made and standard methods developed and used.

#### 3. Groups of Methods

For convenience, the various methods of testing are grouped into three classes:—microscopical, physical and chemical. In most cases, some of the methods from each class are necessary. The accompanying chart (Fig. 1) indicates some of the tests given and shows the relation between them. It is obvious that all the tests indicated are not necessary in any one particular case but such tests should be used that will indicate the quality of paper necessary for a particular purpose.

#### 4. Record Cards

Complete laboratory records should be kept of all tests (especially original data) and in such a manner as to be always available. The accompanying 5 by 8 in. record with both sides reproduced is offered as a suggestion, (Fig. 2) though individual requirements may necessitate certain alterations.

#### 5. Sampling

The proper sampling of paper for test or the interpretation of the test data in connection with sampling has been neglected. It is pointed out, however, that no test data is more accurate than the sampling. This applies with especial force in connection with the testing of a shipment of paper to determine whether it conforms to a definite specification. It is obvious that cases, bundles,

frames, rolls, etc., must be sampled differently but as much care should be exercised in this connection as in the sampling of wood pulp for moisture.

#### 6. Tolerances

The value of the test data is accurate only when a large number of tests are made or when proper tolerance is allowed for. This tolerance is necessary, owing to the errors which are inherent in the whole process of paper testing. The errors are introduced (a) by improper or incomplete sampling, (b) by the natural lack of uniformity in paper, due to its structure, and (c) by the error of the apparatus or method of testing which may either be inherent in the apparatus or due to improper manipulation.

#### 7. Test Sample

The original sample, obtained by proper sampling, should be sufficiently large and of enough sheets to enable all the proposed tests to be made without recourse to an additional sample. The various tests should be made on the several sheets of the sample in order to obtain a reasonably fair average.

### II. MICROSCOPICAL EXAMINATION

#### 1. Estimation of Fiber Content

*a. Preparation of Slide.*—Secure a representative sample by clipping a piece of about the area of a cent from the corners of several of the sheets to be tested. Place the samples in a dish, small beaker, or test tube, cover with a 0.5 per cent caustic soda solution and bring to a boil to remove sizing or other binding material. The pieces are next drained, washed several times in tap water, rolled into a small pill or ball between the thumb and first finger for about 1 min., then placed in a test tube, about half filled with water and shaken vigorously, so as to defiber thoroughly the particles of paper. A small part of this defibered mass is removed from the test tube by the aid of a microscopic needle (Note 1) thoroughly dried on absorbent paper (Note 2) that is free from lint, placed on a microscopic slide and covered with several drops of Herzberg's stain. The fibers are carefully pulled apart, by the aid of microscopic needles, so that they will not lie too much in a bunch and are then covered with the cover glass. (Note 3.) The slide is now ready for an estimation by the aid of the microscope.

It is suggested that after the small sample of paper has been boiled with 0.5 per cent caustic soda, that the sample be next washed with 0.5 per cent hydrochloric acid and finally with water. It is difficult to wash all the caustic from the fibers and an addition of hydrochloric acid seems advisable.

Note 1.—There are two methods in use for removing a representative sample of fibers from the test tube.

First method. Use a test tube of about  $\frac{5}{8}$ -in. diameter and about 6 in. long, keep the fibers diluted with water, so that they will mix readily when shaken vigorously. The fibers mix very easily if the test tube is about two-thirds full of water and fibers. The microscopic needle referred to is a pointed steel needle imbedded in a small wood or metal handle. Shake test tube, and then quickly incline it at a sharp angle. Insert the point of the microscopic needle and remove a small bundle of fibers for use in making up the microscopic slide. The foregoing method of procedure is best where the fibers are long, such as in a rag bond, ledger, or writing paper, also for long-fibered wood papers made of new sulphite or sulphate pulps. For groundwood papers, or where the fibers are very short and contain a large quantity of fine broken particles such as cooked old paper stock, the use of the needle to secure a representative sample will result in securing more long fibers than short fibers, with a result of inaccuracy. For papers containing much short fine fiber it is best to use the second method.

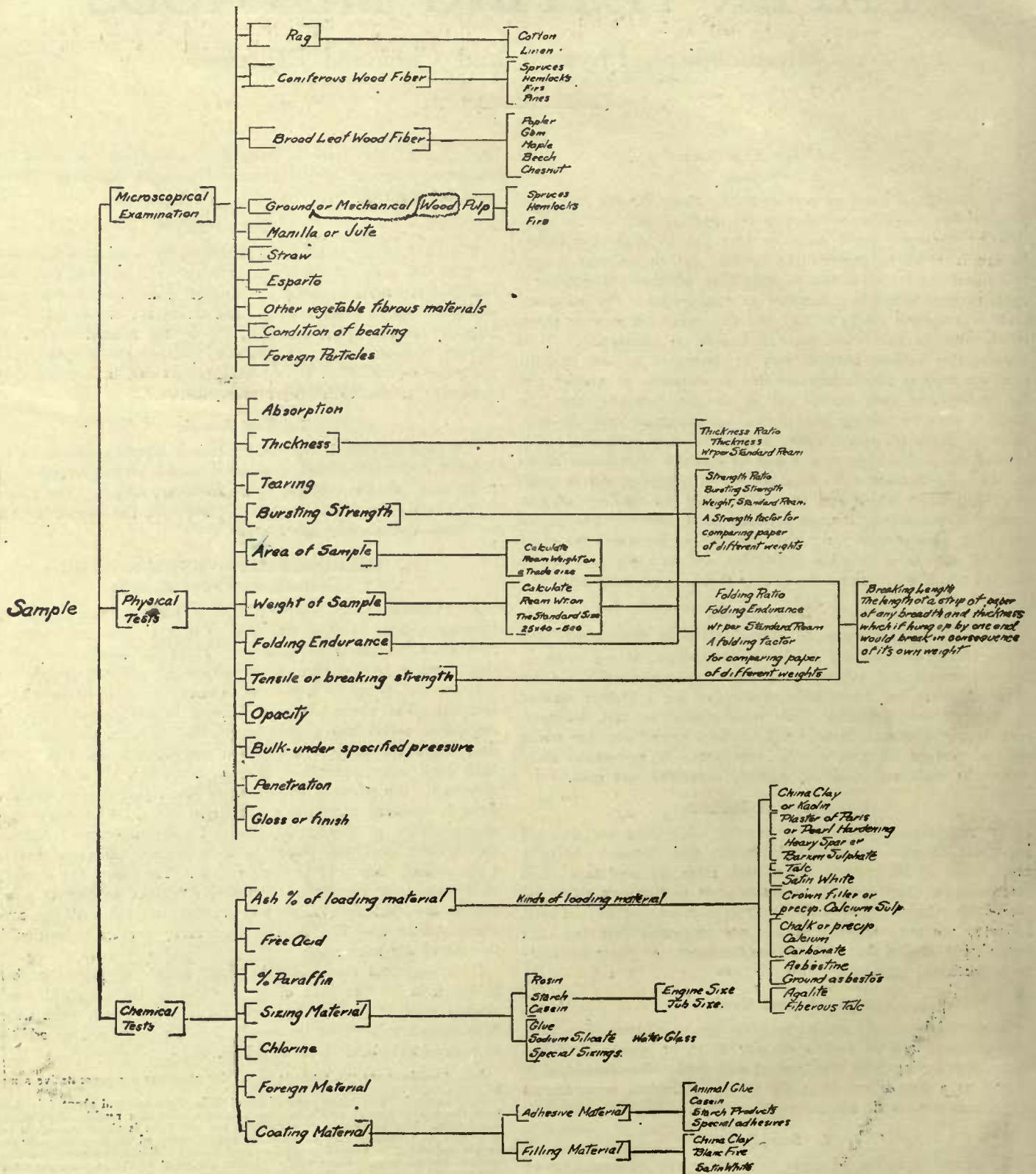


FIG. 1. CHART OF PAPER TESTS

Serial No. 6946

FROM

Received 3-15-22

Folder No. 33633

The Paper Company of America

Reported 3-22-22

MARKED Roll 62 File #342

Unsensitized Blue Print Paper

Weight (25 x 40, 500) = 65 lbs.  
 Weight (17 x 22 - 500) = 24.3 lbs.  
 Bursting strength = 41 points  
 Thickness = inches  
 Ratio bursting strength to wt. (25 x 40, 500) = 63.1 %

FIBER COM-POSITION	Rag	50 %
	Chemical pulp	%
	Chemical pulp, bleached	%
	Chemical pulp, unbleached	%
	Coniferous fiber	50 %
	Broad-leaf fiber	%
	Ground-wood pulp	%
Manila and jute	%	

CHEMICAL TESTS	Ash	15 %
	Total resins	%
	Animal size	Present %
	Starch	Present %

SPECIAL PHYSICAL TESTS	Machine Direction	Cross Direction	Unit
Wet Breaking Strength			grams
Absorption in 10 minutes (Klemm)	752	384	mm
Double folds (Schopper)	65	63	Numb.
Tearing strength	41		gram.
Breaking strength Dry	69	63	Kg
Elongation under load of.....lbs.			%
Breaking length	5500	5020	yds.
Weight data	64.25 = 65		
	1098 x 10 x 9		24.3

REMARKS:  
 Wet Breaking Strength  
 Correction Formula  $y = [(x-1.6)139]$

No.	BURSTING STRENGTH (Points)	Breaking Strength Wet Test		THICKNESS (Inches)	BREAKING STRENGTH		TEARING STRENGTH		FOLDING ENDURANCE (No. of Double Folds)	
		Mach	Cross		Dry Test		4-Ply		(Strip 15 mm wide)	
					Machine Direction	Cross Direction	Machine Direction	Cross Direction	Machine Direction	Cross Direction
1	40	79	38		77	38	104		42	65
2	41	81	43		60	41	105		43	51
3	40	72	39		71	41	106		50	61
4	42	80	44		69	42	105		76	90
5	40	73	46		68	44	106		75	52
6	38	62	46		65	39	106		51	51
7	42	76	46		64	40	106		66	58
8	39	65	44		76	42	106		60	67
9	39	60	47		72	38			53	62
10	40	53	43		64	42	844		126	69
Mean	41.1	7.01	4.36		6.87	4.08	41.1		64.8	634

NO.	ABSORPTION IN 10 MINUTES Klemm Method		NOTE			
	Machine Direction	Cross Direction	Machine Direction		Cross Direction	
1			Bursting strength			
2			Wet Breaking			
3			Schopper 50 Kg #516B		Factor $y = [(x-1.6)139]$	
4			Schopper 30 Kg #516B		Kg	
5			Elmendorf 4 Ply		Grams	
Mean			Schopper		Double Folds	

FIG. 2. TEST RECORD CARD

Second method\*(37). In using this second method it is advisable to have the fibers slightly more dilute in the test tube than is the case in the first method, also the test tubes should be about three-quarters of an inch in diameter by about 8 in. long. In place of the microscope needle a 10-in. glass tube of about seven thirty-seconds of an inch in diameter is employed. This glass tube has one end rounded so as not to have sharp edges and the other end is provided with a small rubber bulb. This serves as a dropper. Spence and Krauss describe the *modus operandi* as follows: "When ready to prepare the slides, the test tube is well shaken, the dropper inserted, with as little delay as possible, two inches below the surface, two bubbles of air expelled and a little less than half an inch of the mixture drawn into the tube. This is transferred to slides, completely emptying the dropping tube, which will make four drops. The slides are placed in an air bath to expel moisture, cooled, and each drop stained with Herzberg stain, just before it is to be examined. The excess stain is then removed—after the colors have developed to a maximum point, about 3 min. required—by tilting the slide and the cover glass placed over the spot." After the cover glass is in place, it should be pressed down gently to expel excess stain and any excess stain removed by absorbing it with a piece of blotting or filter paper.

Note 2—The absorbent paper used should have a hard, smooth surface so that no lint will adhere to the sample of fibers. As soon as the sample is dry it may be removed to the microscopic slide and is then ready for the drop of stain. A second method of drying the sample is to put it on the microscope slide and then touch it with the corner of a piece of folded filter paper of ordinary quality. For this purpose a cheap grade of filter paper may be cut into pieces about 1¼ by 4 inches. This makes a handy size for use in drying the sample and also in removing the excess stain from around the edges of the

eter. The round or square cover glasses are necessary for high magnification and have one disadvantage in that they are very fragile. The third type of cover glass is the same size as the microscope slide, and if thin microscope slides are used then a second microscope slide may be used as the cover for the first. The chief advantage of the large cover glass is that it permits three or four fields to be made up one each slide.

b. *Discussion of Manipulation.*—The following suggestions are offered to those just beginning these tests:

It is absolutely essential to have a satisfactory stain or else the

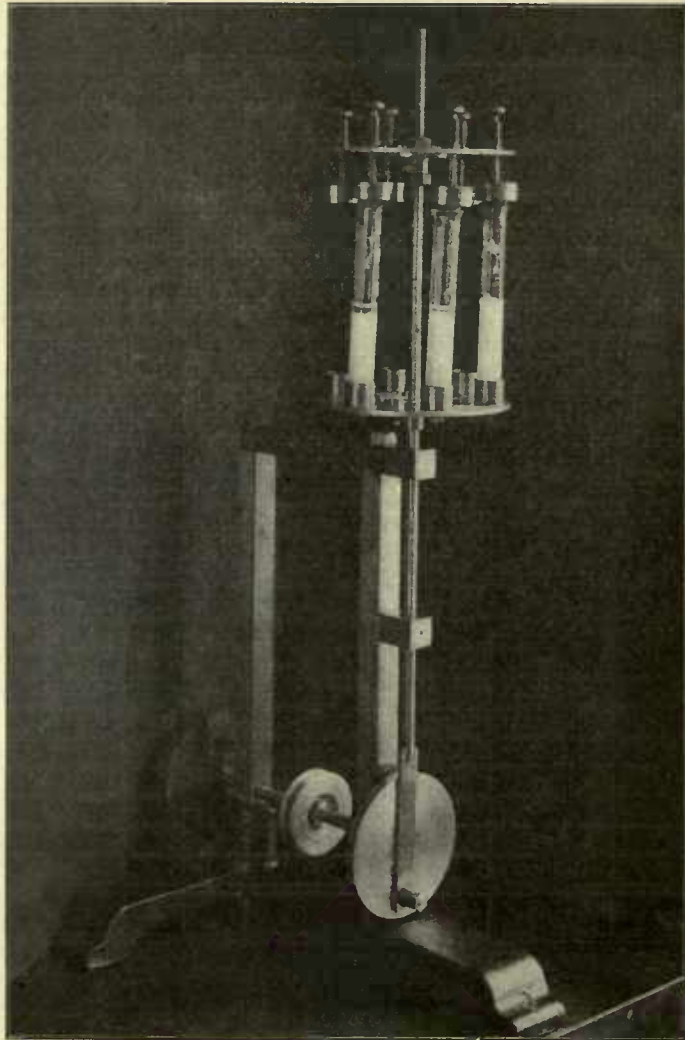


FIG. 3. TEST TUBE SHAKER

By M. B. SHAW, BUREAU OF STANDARDS

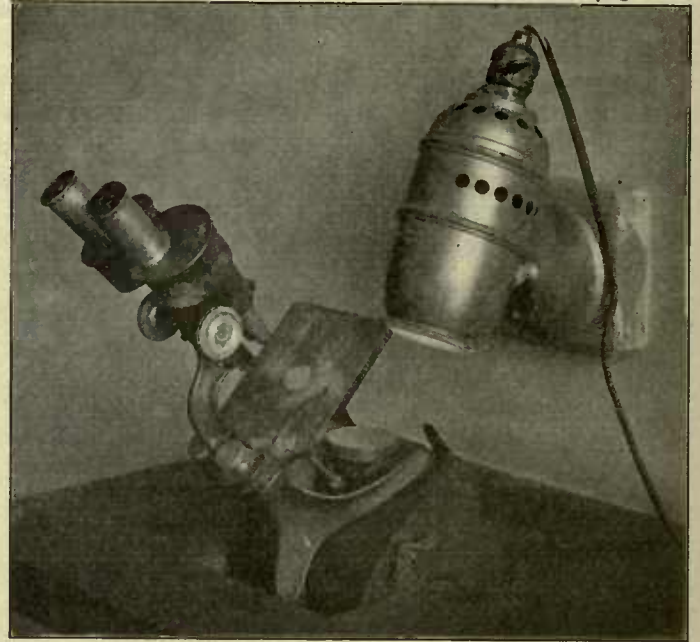


FIG. 4. BINOCULAR MICROSCOPE

results will be worthless. To test out a stain make up a mixture of about equal parts of bleached soda pulp, bleached sulphite pulp and rag filter paper. Prepare a microscope slide from this mixture and stain with the stain to be tested. If the stain is correct, then the soda pulp should show a *dark blue* color, due to the thicker and more opaque fiber walls, the sulphite pulp should show a *light blue*, due to the thin fiber walls and the rag fibers will show a *red or wine-red* color. If the blue color is more of a violet, then too much iodine is present and more water or zinc chloride should be added. Zinc chloride produces the blue color, iodine produces the red and the yellow colors and the addition of water serves to weaken the color that predominates.

In some cases where it is necessary to examine all grades of paper, it is advisable to keep several stains on hand. A stain that gives the best color on groundwood and bleached sulphite seldom gives a correct color on mixtures of rag, bleached sulphite and soda pulps. In such a case, make up one stain so that it will give a *bright lemon yellow* on a known sample of groundwood pulp and a *slightly greenish blue* on unbleached sulphite. For the mixture of rag, bleached sulphite and soda pulp, so adjust a second stain that the rag shows as a *clear wine-red*, the sulphite as a *blue* and the soda fibers as a *dark blue*. In testing out a stain always have on hand authentic samples of pulp so these mixtures may be made up.

To check estimates of fiber analysis, slides of fibers in known proportions are made. Pure stock is beaten in a small beater and made into hand sheets. Sheets of the various pure fibers are kept under the same atmospheric conditions. To make up a field of known composition take weights of the pure fiber sheets and make up a total of at least 5 g. in proportions to give the percentage desired. Disintegrate and mix thoroughly by shaking with shot in a bottle or by the action of a small disintegrator.

cover glass. Care must always be exercised to prevent the sample of fibers to be tested from becoming contaminated with fibers from the absorbent paper or filter paper. A third method of drying the sample of fibers is to place them on a microscope slide and evaporate the moisture in a current of heated air, in an oven or by some other suitable arrangement.

In regard to drying the small sample of the fibers on the microscope slide, attention is called to the fact that the sample must be dried so that the stain will not be diluted and yet must not be dried too hard because then it is difficult to separate the fibers and the staining does not take place uniformly.

Note 3—There are three kinds of cover glasses. The first two are very thin pieces of glass either round or square of approximately half an inch in diam-

Sample and make up the slide as for any disintegrated paper sample.

The estimation of the fiber content is based on the relative proportion of the kinds of fibers contained therein, expressed on the percentage basis, considering the total fiber content as 100 per cent. In making a fiber estimation no account is taken of the per cent of clay, alum, size, etc., that may be contained in the paper. It is always advisable to make up at least two separate samples of fiber taken from the test tube and the final result should be the means of all observations on these two separate slides. In special cases it may be necessary to make up four separate fields.

There are two methods of making the determination for fiber content. One is the *count method*, the other is the *estimation method*. Both methods have their advocates and both give good results. This committee, however, recommends the *estimation method*, believing it to have the following advantages:

(1) It is more accurate under certain conditions, namely, in making groundwood determinations, and of equal accuracy under all other conditions; (2) it is much quicker; (3) it is easier to teach an individual to estimate correctly than to count correctly; (4) it is possible to make up standard mixtures for ready comparison.

The *estimation method* involves training the eye by the comparison of unknown samples with standard mixtures of known composition. The result of each observation on each part of a field examined, should be written down and the mean of all the observations is the result to be reported as final. Accuracy in the *estimation method* involves practice and continual reference to known standards. Unstained slides of these standard mixtures should be kept handy to be made up in case there is any doubt about the sample being tested.

There is a third method for fiber determination that has been proposed by Spence and Krauss \*(37) which is worthy of description here and recommendation to the Technical Association. This is known as the *fiber-weight-length method*. The procedure is as follows: Samples are made up as described under Note 1, second method. The slide is placed under a microscope of 160 diameters and the lengths of the various fibers are measured in terms of the diameter of the field seen through the microscope. An adjustable stage is also essential as otherwise it would not be possible to move systematically over the entire sample to be examined. After four samples have been estimated as above described, the figures are added together to get the total length of each kind of fiber present. The total length of each kind of fiber present multiplied each by its own weight factor gives a set of results that are directly comparable and may be converted into the per cent of each kind of fiber present. The weight factors as determined by Spence and Krauss are as follows: Rag, 1.000; hemlock pulp, 0.870; poplar pulp, 0.454; birch pulp, 0.652; beech pulp, 0.525; maple, 0.365. This method, which is undoubtedly a step in the right direction, is recommended as a method to be used in cases of dispute between two different analyses. It is a very slow method and cannot therefore be used where many routine samples must be examined each day. The Spence-Krauss method is undoubtedly the only method that will enable a determination of the proportion of the various kinds of wood present, such as a mixture of hemlock, beech, poplar, birch, maple, etc.

In any method of testing it is always advisable to make use

No definite recommendation is given in regard to the microscope magnification. This must be left to the individual preference. Satisfactory estimations may be made with a magnification as low as 45 diameters and equally satisfactory work is being done with magnifications as high as 120. The lower magnification has the advantage of giving larger fields, whereas the higher magnification gives more of the detail of the markings of a fiber. The monocular and binocular microscopes both have their advocates, and like the magnifying power, it is very largely a matter of getting used to a certain procedure. Where only one microscope can be purchased it is better to use a monocular, as it can be fitted with more attachments to suit special needs. It may be well to add that a low power of about 25 diameters for examining specks and surfaces, also a high power of 350 or 400 for details of fiber markings will often be found to be of use. Spence and Krauss\*(37) recommend a magnification of 160 diameters.

of all possible apparatus that may be of assistance in carrying out the method described. There are no holders for microscope slides on the market, therefore a brief description will be given and it may be constructed at almost no expense for labor or material.

The holder for the microscope slides is made as follows: Take two pieces of brass  $\frac{3}{8}$  in. thick by  $\frac{1}{2}$  in. wide by 3 in. long (oak or maple may be used if the brass is not obtainable), then cut a groove  $\frac{1}{8}$  in. wide by  $\frac{1}{4}$  in. deep along one of the longitudinal edges of the brass strip. This groove then serves as a rest for the glass slides.

The pins (see Fig. 5) serve to prevent the glass slide from slipping out of the grooves while the bundles of fibers are being teased apart. Also note the parts on the sketch marked "Painted black" and "White surface." These serve as a background. The glass slide is placed over the black background when the unstained fibers are first put on the glass slide, as the light (almost white) colored fibers show up best with a black background. After the Herzberg stain is added, the glass slide is pushed to the other end of the brass holder, which brings it over the white background and causes the dark-stained fibers to show up more distinctly and enables even the smallest bundles to be separated.

For best results for microscopic work, a clear north light is desirable, and is to be preferred. However, where there is a large amount of routine testing that must be done, it is more advisable to have a more constant source of light. There are various types of lamps available but good results can be obtained with a Mazda nitrogen-filled lamp of 150 watts. It is necessary, however, to use a blue "daylight" filter in that case. It is to be noted that the color of the stained fibers on the slide will be somewhat different for the two kinds of illumination.

### c. Common Stains.

HERZBERG'S. \*(10).—The Herzberg stain is made according to the following formula:

Solution A—20 g. zinc chloride.  
10 cc. of water (preferably distilled).  
Solution B—2.1 g. potassium iodide.  
0.1 g. iodine crystals.  
5.0 cc. of water (preferably distilled).

Dissolve solutions A and B separately, then mix and allow to stand several hours, or until all sediment has settled out. The clear liquid is next decanted and is ready to be used in staining the fibers. All iodine solutions must be kept in the dark, as otherwise they deteriorate rapidly. The Herzberg stain is a selective stain, that is, it has selective staining properties. Ground or mechanical wood pulp, jute, flax tow, uncooked manila hemp and in fact most every vegetable fibrous material containing large quantities of lignocellulose, is colored *yellow or lemon yellow*. The removal of their lignocellulose content changes the staining effect from a *yellow* to a *blue* or *wine-red* color, though jute and a few other fibers remain unchanged in color. Thoroughly cooked and bleached soda and sulphite pulps, cooked and bleached straw pulp and esparto are colored *blue* or *navy blue*. Cotton and linen rags, thoroughly cooked and bleached manila hemp, and certain of the Japanese fibers are colored a *wine red*.

In connection with the Herzberg stain, the following alternative formula is suggested: 25 cc. zinc chloride solution (saturated) at 70° F.; 5.25 g. potassium iodide; 0.25 g. of iodine, and 12.5 cc. water. Owing to the difficulty of obtaining zinc chloride of uniform moisture content, it has been found more satisfactory to use a saturated zinc chloride solution. By mixing the ingredients as stated above, the proper stain can be obtained at once.

JENK'S.—The stain known as "Jenk's Stain" is of value when it is desired to ascertain definitely small amounts of rag fiber with only a poor Herzberg stain available: To 50 cc. of saturated magnesium chloride solution add  $2\frac{1}{2}$  cc. of iodine potassium iodide

solution made up as follows: Potassium iodide, 2 g.; iodine, 1:15 g., and water, 20 cc. Use exact quantities and keep solutions from the light; the stain is kept best in a small brown bottle with a pipette. Rag fiber is stained *brown*, straw is stained *blue-violet*, groundwood is stained *yellow*, and chemical wood either *no color* or *deep red*.

SUTERMEISTER'S. \*(3).—A stain which is considered by some to be better than the Herzberg stain is made up as follows:

Solution A.—1.3 g. iodine and 1.8 g. potassium iodide in 100 cc. of water.  
Solution B.—A clear, practically saturated solution of calcium chloride.

In using this stain apply a drop or two of *Solution A* to the moist fibers on the microscope slide. After a minute or so remove the stain by means of a blotter and immediately put on a drop or two of *Solution B*. Pull the fibers apart and distribute them by means of needles as before and drop on a cover glass or thin microscope slide. Any excess of *Solution B* should be removed by absorbing it with moist blotting paper. This stain is also selective in its action, the colors produced being as follows:

*Red or brownish red*: cotton, linen, hemp, ramie.

*Dark blue*: bleached soda pulps from deciduous woods.

*Bluish or reddish violet*: bleached sulphite fibers and the thoroughly cooked part of the unbleached sulphite.

*Greenish*: jute, manila and the more lignified fibers in unbleached sulphite.

*Yellow*: groundwood.

As with the Herzberg stain this one should be adjusted by trial on known mixtures of fiber until it shows satisfactory difference in color. The two solutions should be protected against evaporation

kept in tightly stoppered separate bottles, and mixed only when wanted for use:

A—Malachite green .....	2 g.
Distilled water .....	100 cc.
B—Basic fuchsine .....	1 g.
Distilled water .....	100 cc.

Since there is considerable variation in the quality of dyes from various sources, it is not to be expected that any given combination of dyes or method of procedure will best fit all cases; it is, indeed, more than probable that the compound stain will have to be modified somewhat as to its two components, depending on the source of the dyes.

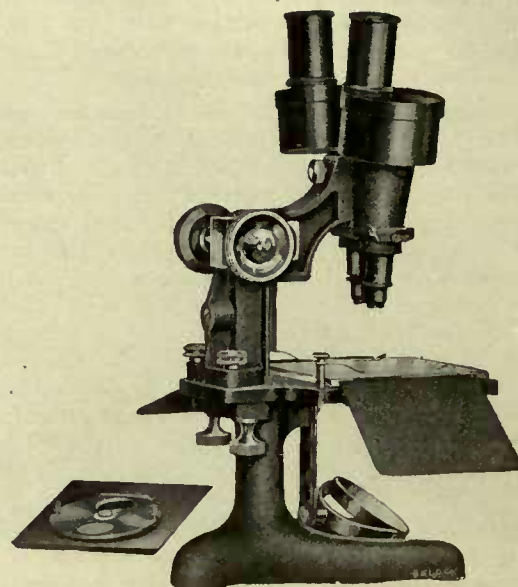


FIG. 6. BINOCULAR MICROSCOPE

After this stain, therefore, has been made up according to formula, it will be necessary to test it out on samples of sulphite and sulphate fibers. To do this, samples of unbleached sulphite and sulphate pulps should be prepared and a few fibers of each placed on a slide, care being taken not to get the two samples mixed. The fibers are then dried and stained, as directed below, and then examined under the microscope. All the sulphate fibers should have a *blue* or *blue-green* color, and all the sulphite fibers should have a *purple* or *lavender* color. If any purple fibers appear in the sulphate pulp this indicates that too much fuchsine is present in the combination, and a little more malachite green solution must be added to counteract this effect. If, on the other hand, some of the sulphite fibers show green or blue, there is too much malachite green in the combination, and more fuchsine solution must be added. Of course the analyst must be sure that he is using authentic samples of the two pulps for this test. When tested out in this manner and the proper combination found, the stain is ready to be used on unknown combinations of fibers containing either unbleached sulphite or sulphate, or both.

A mixture of one-half sulphite and one-half sulphate may also be used to test out the stain, the proper combination for the stain being indicated when one-half of the fibers are colored blue, and the other half purple.

The stain should not be used for more than a few hours after being compounded and should be made up anew at least each day.

PHLOROGLUCINOL.—Dissolve 5 g. of phloroglucinol in a mixture of 125 cc. of distilled water and 125 cc. of concentrated hydrochloric acid. The solution should be kept in the dark as much as possible as it is prone to lose its staining property on exposure to light. This solution produces a *magenta* or *wine-red* color on

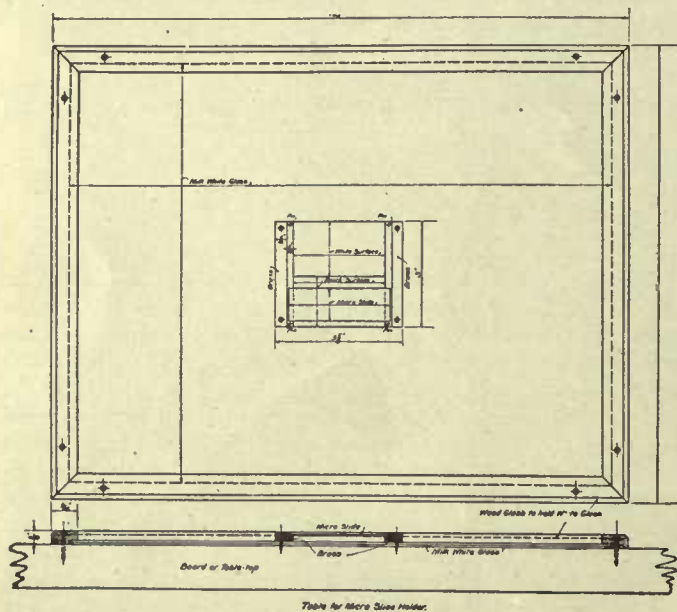


FIG. 5. HOLDER FOR MICROSCOPE SLIDE.

and dust but light does not change their staining properties to any extent.

#### d. Special Stains.

There are many stains in use for special purposes and a description of them is therefore advisable.

LOFTON-MERRITT SULPHATE STAIN. \*(99).—The stain which was found to be most satisfactory in differentiating between unbleached sulphite and sulphate pulps or fibers was a mixture of one part of a 2 per cent aqueous solution of malachite green and two parts of a 1 per cent aqueous solution of basic fuchsine, or magenta. The solutions were made up according to the following formulas,





## CLASSIFICATION AND CHARACTERISTICS OF THE MORE COMMON VEGETABLE FIBERS USED IN THE INDUSTRY

Fiber	A. Seed fibers or Seed hairs		B. Stem or Bast fibers (Dicotyledonous plants)						
	Cotton ( <i>Gossypium</i> )	Flax ( <i>Linum usitatissimum</i> )	Hemp ( <i>Cannabis sativa</i> )	Jute ( <i>Corchorus capsularis</i> )	Abacá ( <i>Musa textilis</i> )	Straw (Wheat, barley, oat)	Esparto ( <i>Stipa tenacissima</i> )		
1. Shape	Flat twisted ribbon	Cylindrical	Blunt ends often with lateral branches	Rounded ends	Uniform diameter, sharp narrow ends		Similar to straw cells, but smaller		
2. Cell wall	Thick	Very thick	Thick	About as wide as lumen	Thin				
3. Lumen	Broader than wall thickness	Very narrow	Broad exceeding wall thickness	Often wider than cell wall. Irregular	Broad and distinct				
4. Markings	Granular between thick edges	Fine cross lines like joints or nodes	Folds, swellings and cross fissures	Longitudinal striations no ridges or cross marks	None		Cross markings on Bast cells		
5. Parenchyma cells	None	None	None	None	None	Thin walled. Shaped like coffee bean	Small pear-shaped cells		
6. Epidermal cells	None	None	None	None	None	Thick walled serrated characteristic of straw and esparto			
7. Bast cells	None	None	None	None	None	Long thin fibers of regular form. Small canal. Taper to fine point			
8. Other features	Diam. uniform $\frac{3}{4}$ of length. Ends cylindrical and pointed	Cross lines sometimes intersect like an X	Longitudinal striations	Lumen broadens at end of fiber	None				
9. Length	$\frac{1}{2}$ " to $\frac{3}{4}$ " average 1"	$\frac{1}{2}$ " to $2\frac{3}{8}$ " average $1\frac{1}{8}$ "	$1/20"$ to $1/4"$	$1/20"$ to $1/5"$	$1/10"$ to $1\frac{1}{2}"$ average $3/4"$	$1/250"$ to $1/20"$ average $1/35"$	$1/50"$ to $1/10"$		
10. Breadth	.0074" to .0070" average .0062"	.0030" to .0062" average .0040"	.0040" to .0080" average .0050"	.0025" to .0080" average .0045"	.0040" to .0080" average .0060"		.0017" to .0045" average .0030"		
11. Color reactions with chlorfide of zinc	Wine red	Purple to yellow	Purple to yellow	Yellow	Uncocked — yellow cooked — red	Blue to greenish yellow	Same as straw		
12. With phloroglucine	Not colored	Not colored or slightly red	Slightly red	Deep red		Colorless to red	Same as straw		
13. With aniline sulphate	Not colored	Not colored or slightly yellow	Yellowish green	Slightly yellow		Colorless to yellow	Same as straw		

Fiber	B. Stem or Bast fibers			C. Leaf fibers (See B for Manilla hemp) (From leaves of many monocotyledonous plants)			E. Wood fibers, from the trunk or stems of various coniferous and hard wood trees.		
	Ramie ( <i>Urticaceae</i> ) a nettle fiber ( <i>Baccharis tenacissima</i> )	New Zealand hemp ( <i>Phormium tenax</i> )	Sisal ( <i>Agave sisalana</i> )	Pineapple leaf fiber or silk grass ( <i>Ananas sativa</i> )	Coniferous wood fibers	Broad leaf hard wood fibers			
1. Shape	Jointed and with transverse fissures	Regular and uniform	Stiff. Broad at middle	Extremely fine and durable	Flat ribbons with spots and pores	Short and cylindrical. Often cross marked. Ends pointed.			
2. Cell wall	Thick at ends	Thicker than lumen	Often very thick in cells						
3. Lumen	Thin as a line at ends. Irregular at center	Very uniform in width	Broad lumen	Narrow like a line					
4. Markings		Fragments of parenchyma and epidermis often on fiber	Peculiar spiral cells present. Surface pores show on short cells	Distinguished by extreme fineness	Shape and position of pores indicate kind of tree				
5. Parenchyma cells			Present in peculiar forms		None	None			
6. Epidermal cells									
7. Bast cells	Long and broad. Irregular diameter. Rounded ends								
8. Other features	Irregular base	Sharp ends not divided. Smooth fibers	Blunt thick ends, seldom forked	Markings less distinct in well boiled pulps. Occasionally twisted.		Always accompanied by short tubular vessels of large diameter with numerous pits			
9. Length	1/4" to 2.0", average 1.0"	1/10" to 3/8", average 1/3"	1/10" to 1/4"	1/20" to 2"					
10. Breadth	.016" to .020", average .0125"	.0020" to .0070", average .0040"	.0050" to .0085"	.0010" to .0020"					
11. Color reactions with chloriodide of zinc	Dark red to green yellow	Deep yellow or brownish	Yellow	Blue to greenish yellow	Blue	Blue			
12. With phloroglucine	Colorless	Reddish	Red	Colorless to light red	Colorless	Colorless			
13. With aniline sulphate	Colorless	Yellow green	Yellow	Colorless to yellow	Colorless	Colorless			

FIG. 8—CHARACTERISTICS OF FIBERS\* (143)  
A table which gives information in regard to the shape and structure of various fibers used for paper making and also gives the color reaction with several of the more common stains.

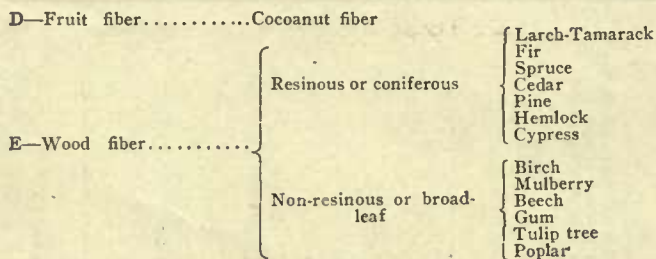


FIG. 7

### 3. Degree of Beating

By a careful examination of the fibers under the microscope, it is often possible to determine something of the amount of beating to which the stock has been subjected. The length of the fibers, the amount that the ends have been frayed and the degree of the breaking down of the cell-walls all give information in regard to the beating treatment. It is necessary, however, to have considerable experience before the results are reliable. The use of photomicrographs assists in this study and the accompanying plates indicate some of the characteristic differences of fibers and in beating treatment.

### 4. Specks or Dirt in Paper

The appearance of a sheet may show imperfections caused by foreign materials or malformation on the wire. These are the most common causes of poor-looking paper.

Generally, specks need microscopic examination. A Bausch and Lomb binocular microscope shown in Fig. 6 and a set of dissecting needles are useful. For chemical tests on small particles small test tubes made by sealing one end of small glass tubing are convenient if the reaction is to be watched under the microscope.

**Rubber.** This is very objectionable. It finds its way into the stock along with rag stock, sometimes as rubber paste in tire fabrics and the like, and sometimes in paper stock as rubber bands from office waste.

Under the magnifying glass rubber specks can be stretched by pinning down one end with a dissecting needle and pulling out the speck with another needle point.

Rubber specks will give a characteristic rubber odor if burned by sticking into a flame on the end of a needle. They are soluble in carbon tetrachloride.

**Rosin specks.** These are translucent amber-colored specks so resembling rosin that they are easily recognized. Proof of their identity can be had by dissolving the separated speck in ether in a small tube so that the action can be watched under the microscope. Qualitative rosin tests can be applied to the speck as given under qualitative tests for rosin.

Other specks resembling small bark particles may come from size which was made from impure rosin without proper filtration. Although not as translucent as the ordinary rosin speck they usually carry enough rosin to respond to the qualitative test.

**Wood specks.** Chips or wood fibers which might result from the accidental grinding off of a beater paddle or similar cause can be quickly identified by applying phloroglucinol; they give a characteristic red coloration as in the groundwood test.

**Iron specks.** Washer or beater bars, jordans, scaly pipes, corroded overhead ironwork, and iron buttons from rags contribute iron in metallic or oxidized form at times. The metallic particles will be attracted by a magnet after being freed from the sheet. The scale or oxidized iron can be dissolved in concentrated hydrochloric acid and a drop of potassium sulphocyanate added. Iron gives a characteristic wine-red color. This test can be applied to the separated particle in a small tube, or the sheet suspected to contain iron may be placed on a glass plate, wetted with concentrated hydrochloric acid for five minutes, and then with 10 per cent

potassium sulphocyanate solution. Each iron speck shows red when the sheet is held up to the light. The glass plate forms a convenient holder for the sheet. The red color fades in a few minutes and count should be taken immediately.

Another method is to immerse the paper in 2 per cent potassium ferrocyanide, then in 2 per cent acetic acid, then wash well in water. Hang the sheets vertically until dry. There will be a blue coloration wherever there was an iron speck in the sheet. This method makes a more permanent record than the sulphocyanate treatment.

**Oil spots.** Oil spots are translucent and can be spread or thinned with ether or chloroform. Extraction with either of these solvents removes the oil, unless it is of a peculiar pasty formation caused by use of oily rags in the stock. Mineral oil in rags is prone to form a dirty congealed mass in the washers, which specks the halfstuff with black specks in which mineral oil is the binder. Such specks in the finished sheet are not entirely removed by ether or chloroform. They are slightly translucent, and unaffected by solution in concentrated sulphuric acid.

**Color spots.** Poorly ground colors such as poor ultramarine give a fine specky appearance usually identified by color only.

**Alum spots.** These are usually pulverized by the pressure of the calender rolls. They are soluble in water and give a slight acid reaction with indicators. This reaction is best watched by dissolving the speck in a very small test tube and adding the indicator while the tube is under the microscope and against a white background.

**Coal particles.** Coal dust is insoluble and gives no color reactions with any reagent. In appearance iron scale can be mistaken for it, and in doubtful cases an iron test should be made on the sheet and the unaffected black particles examined for coal.

Under the microscope it can be seen that coal particles in a calendered sheet have been so pulverized by the pressure of the rolls that they shatter very easily when picked with a dissecting needle. Large particles give a characteristic black smear when crushed and rubbed across the sheet.

**Button specks.** Bone buttons ground by beaters or jordans into small pieces come through into the finished sheet as a light colored powdered spot due to crushing in the calenders. A hole is often made at a button speck due to the crushed button piercing the sheet and then partly crumbling out after calendering. Such specks can be differentiated from alum as they are insoluble in water and give no acid reaction with the indicators.

**Paper specks.** In stock made from old papers small undefibered pieces may slide through the screens and form a speck on the sheet. Such specks are fibrous and when lifted out of the sheet they can be defibered under the microscope with dissecting needles, showing their identity by this characteristic.

**Foam spots.** Because of the depression left after each foam bubble there is a circular spot more translucent than the rest of the sheet formed wherever foam bursts on the partly formed sheet. The result is characteristic, circular, and translucent as a small round watermark would look.

**Drag spots.** Stock adhering to the slices on the wire forms small uneven lumps when it drags off upon the sheet. These spots are not very common but can be recognized as an irregular formation having no foreign material present.

**Knots.** Fabrics in rag stock with knotted threads very often show the knots in the finished sheet. The knotted thread is easily recognized under the microscope.

### 5. Starch

In addition to chemical tests for the determination of starch in paper, it is possible to determine the kind of starch used. The various untreated starches have characteristic shapes and markings which may easily be identified under the microscope. This is also possible in some cases with treated starches, used in the size-tub.

### III. PHYSICAL TESTING

#### 1. Effect of Relative Humidity\*

A superficial examination of the published data will indicate that the physical qualities of paper are affected to a considerable degree

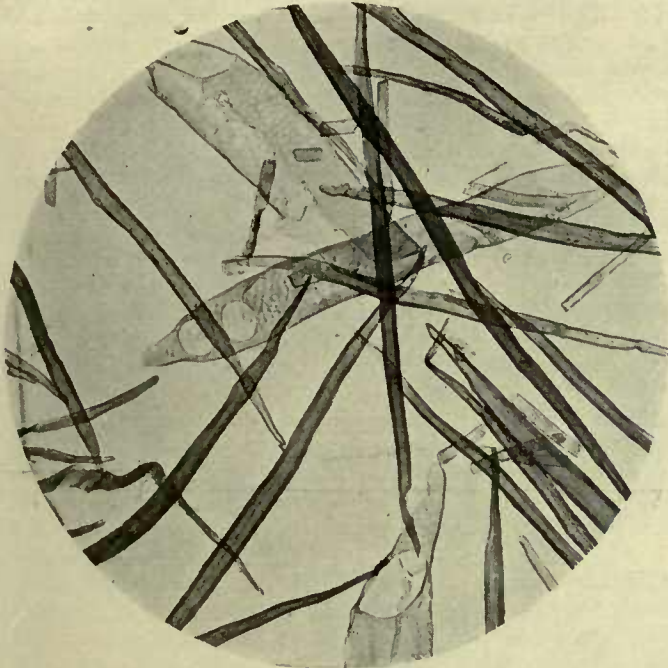


FIG. 9

Aspen (*Populus tremuloides*)—x100—(Bureau of Standards).

by changes of the moisture content of the test sample. Different kinds of paper as well as different qualities are affected to a different degree, but certain tendencies are obvious and the importance of the consideration of the condition of the test sample at the time of

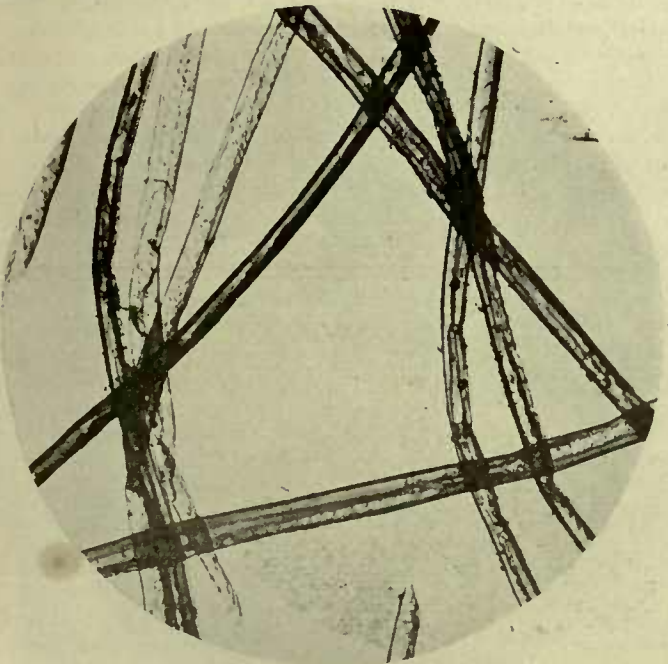


FIG. 10

Balsam Fir (*Abies balsamea*)—x100—(Bureau of Standards).

test should not be underestimated. The suggestions contained herein are not conclusive nor complete but the conclusions have been determined after a careful study of existing data.

\* The reader is referred to an exhaustive treatment of this subject in Technical Association Papers, Series VI (1923).

(a) *Relative Humidity*—The moisture content of the test sample is affected by changes in humidity, either absolute or relative. Absolute humidity is defined as the number of grains of moisture per cubic foot of air at the temperature in question. Relative humidity is defined as the percentage of moisture present, at any particular



FIG. 11

Chestnut (*Castanea durata*)—x100—(Bureau of Standards).

temperature, to the amount of moisture present if the air were saturated at that temperature. The available data seem to indicate that in most cases, the variation of quality of paper bears a relation to relative humidity, rather than to absolute humidity.

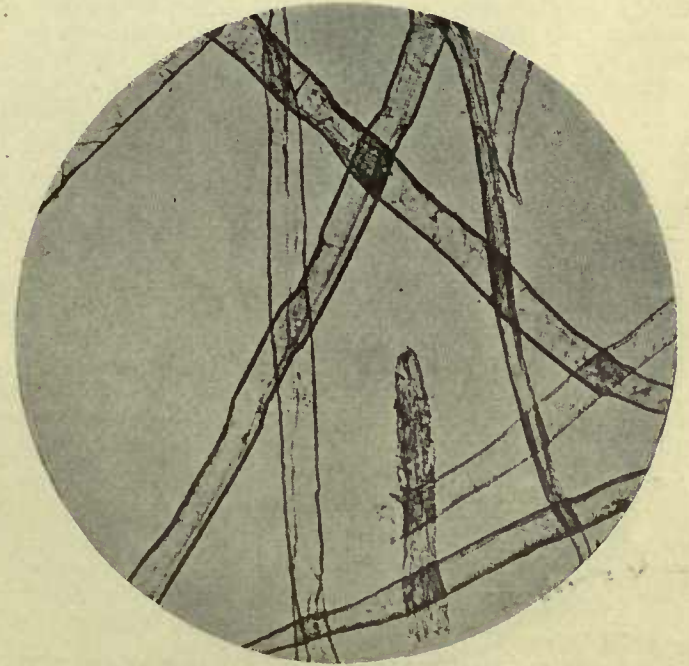


FIG. 12

Hemlock (*Tsuga canadensis*)—x100—(Bureau of Standards).

In most testing laboratories that attempt to control their atmospheric conditions, a temperature of 70° F. and a relative humidity of 65 per cent is maintained. These conditions have been

adopted because of work done in the past in Germany and because of the increased cost to maintain a lower relative humidity during the warm weather, when the moisture must be taken out of the air by some method of refrigeration. It is not uncommon, however, in steam heated rooms, during the winter, to obtain a relative humidity as low as 15 per cent.

and, at 85 per cent relative humidity, the range is from 9 to 14 per cent, with 20 per cent as a possible saturation point at 100 per cent relative humidity. The accompanying curve (Fig. 17) indicates in a general way the tendencies of change of moisture content with relative humidity.

(c) *Weight*—In general, it may be said that the variation of



FIG. 13

Fibers from Rag Pulp— $\times 100$ —(Bureau of Standards).

(b) *Moisture*—The moisture content of paper increases with increase of relative humidity and in general seems to be independent of the furnish, kind of paper, or the method of test. A composite average with the moisture content, when plotted, as ordinate and

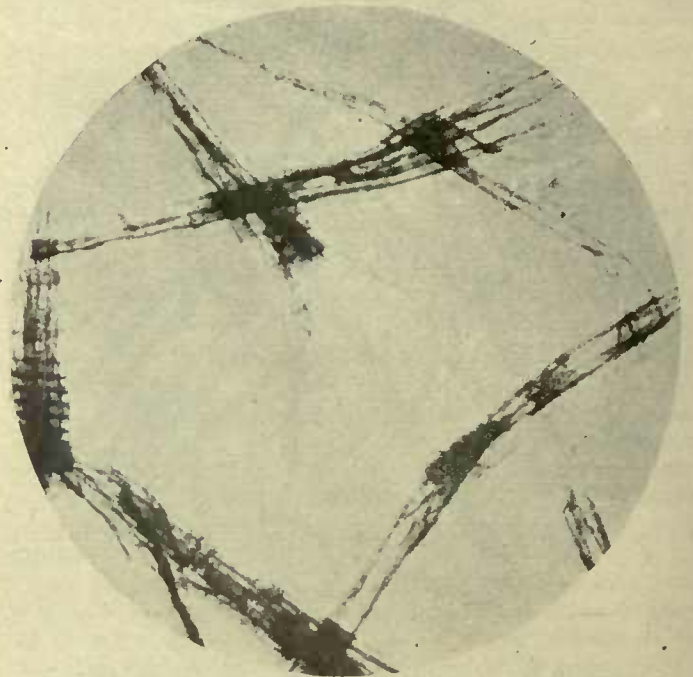


FIG. 15

Spruce Mechanical Pulp (*Picea canadensis*)— $\times 100$ —(Bureau of Standards). weight due to changes of relative humidity is similar and proportional to the variation of moisture content of the paper. The variation in weight from 15 per cent relative humidity to 85 per cent relative humidity seems to be about 6 per cent.

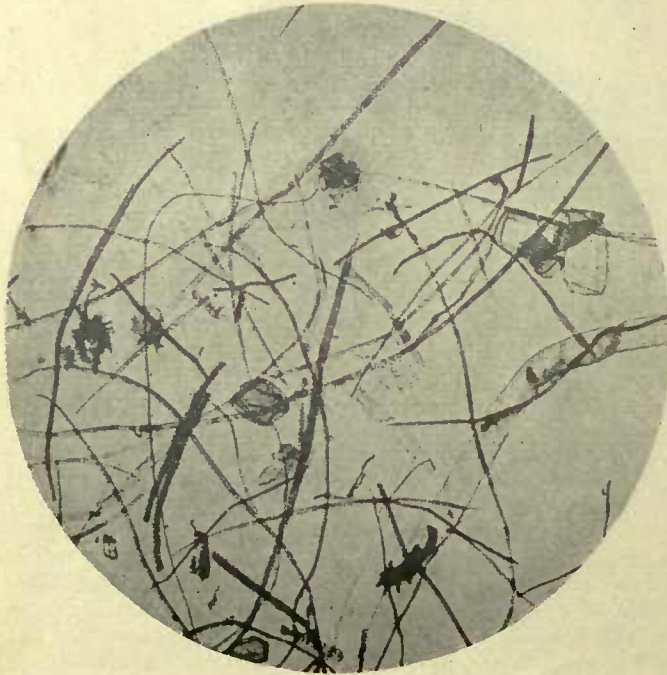


FIG. 14

Rice Straw— $\times 100$ —(Bureau of Standards).

with relative humidity as the abscissa produces a regular curve, slightly concave. At 15 per cent relative humidity, the moisture content varies from 3 to 7 per cent for different kinds of paper

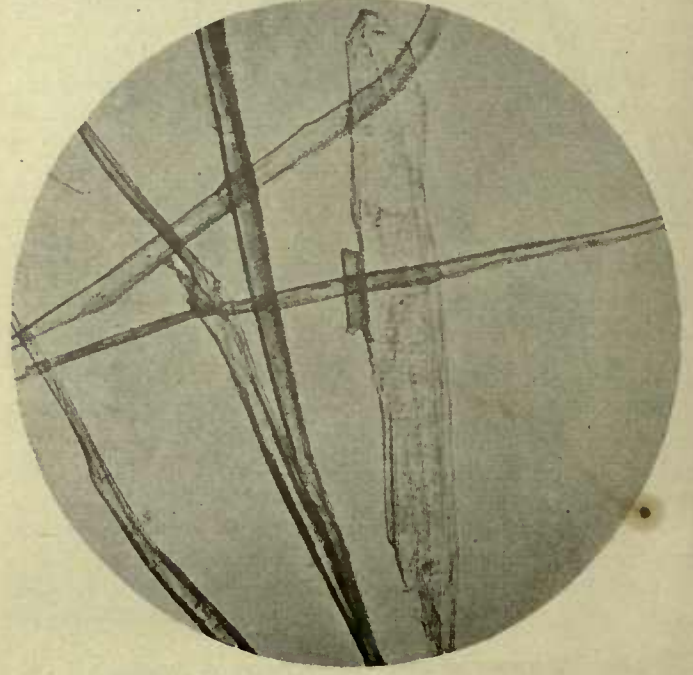


FIG. 16

Tulip Tree (*Liriodendron tulipifera*)— $\times 100$ —(Bureau of Standards).

(d) *Bursting Strength*—Data available at this time in regard to the effect of relative humidity upon the bursting strength seem to indicate that bursting strength increases with relative humidity,

up to about 35 per cent relative humidity and that from that point decreases equally rapidly with increasing relative humidity. This conclusion seems to be assured from the considerable amount of data available but it is not believed that any conversion factor may yet be developed. The amount of variation is widely different for different papers and it seems to be evident that long-fibered papers are affected to a greater extent than short-fibered papers. In any case, this variation is quite evident and should be taken into consideration when careful and accurate tests are to be made.

(e) *Tearing Strength*—Very little data are available in regard to the relation between tearing strength and relative humidity but such work that has been done indicates that this test is markedly affected by changes of relative humidity. Tearing strength increases to a considerable extent with increase of relative humidity and the amount of this variation seems to be comparable with that in the case of the folding and tensile test.

(f) *Folding Endurance*—The effect of relative humidity upon this test seems to be somewhat erratic with different papers but in any case the variation is very marked. In general, the folding endurance increases rapidly with increase of relative humidity, the machine direction more rapidly than the cross direction. With certain kinds of paper, there seems to be a peak in the curve at 80 to 90 per cent relative humidity with a rapid decline, while with other papers, this peak does not appear. Data seem to indicate that this test is affected by relative humidity to a greater extent than any other.

(g) *Breaking or Tensile Strength*—The variation in this test seems to be very similar to that in the case of the bursting strength, but to a greater degree. Strength increases with relative humidity up to a point of about 35 per cent and then decreases at a similar rate. This variation is similar in both the machine and cross direction and in either case seems to be over twice as much as in the case of the bursting strength.

## 2. Characteristics of Paper

(a) *Machine Direction*: \*(3) Several methods are available for determining the machine direction in a sample of paper. It may sometimes be ascertained by mere inspection of the sheet, as the formation noted on looking through it is often conclusive to the trained observer.

The usual machine wire imparts to the sheet of paper a "wire mark" consisting of a series of diamond-shaped marks, the long diagonal of which points in the machine direction. If the wire mark is sufficiently prominent so that its direction can be determined this will establish the machine direction.

If the paper is well sized and a circular piece is cut out and moistened on one side by floating on water, it will tend to roll up into a cylinder whose axis is in the machine direction of the sheet. If the paper is unsized it will become entirely soaked through on floating on water and will not curl up. This may be avoided by sizing the paper with an alcoholic solution of rosin or with a solution of gelatine in water, drying and then making the test.

Another method of determining the machine direction is to cut two narrow strips of the paper one from either direction, place these one over the other and hold them upright in the fingers. They will droop over of their own weight and if they cling close together the under strip is in the machine direction while if the under slip falls away from the upper the latter is in the machine direction.

The form of the break made by the Mullen tester shows the machine direction, as the longest, or chief, line of rupture is always across the sheet.

(b) *Wire or Felt Side*. \*(3) In many cases this may be determined very easily by a simple inspection but in some papers the wire marks do not stand out at all plainly. Sometimes they may be made more prominent by plunging the sample for a moment into water and draining or blotting off the excess. The moisture causes the fibers to expand, thus undoing the work of the calenders

and resorting the texture of the sheet as it left the machine wire. Inspection of a sheet thus dampened will often show that the wire marks stand out plainly, where before they were indistinguishable. This method very often proves satisfactory even for coated papers.

## 3. Area of Sample

For convenience use a straight edge graduated into inches and tenths and read to hundredths of an inch. Calculate area in square inches.

## 4. Weight of Sample

The sheet-weighing device that indicates the equivalent weight

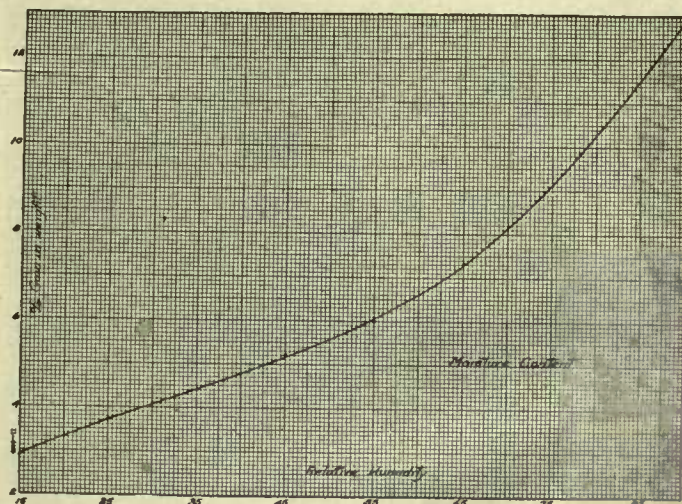


FIG. 17. MOISTURE CONTENT OF PAPER

A curve which represents the relation between the percentage of relative humidity and the moisture content of paper. This curve is a composite of data obtained from several sources and under different conditions.

in pounds in terms of a 500-sheet ream, is most suitable for laboratory or mill use.

In weighing very small samples, it is not desirable to use a weighing device graduated in terms of a 500-sheet ream. For such cases a chemical balance should be used and the weight in grams multiplied by 1.102, will give the equivalent weight of 500 sheets of the size weighed.

Formula for sample weight on sheet paper scales:

$$\frac{(\text{wt. in lb.}) \times (1,000)}{\text{Area of sheet in sq. in.}} = \text{weight } 25 \times 40,500.$$

$$\frac{(\text{wt. in lb.}) \times (\text{Area of trade size desired})}{\text{Area of sheet in sq. in.}} = \text{wt. of trade size desired.}$$

It is obvious that the samples being weighed must be accurately measured to determine their size, and this is done by means of an accurate rule, graduated in tenths of an inch. The following formula is of assistance, where *a* is scale reading, *b* is one dimension of the sample, *c* is the dimension at right angles to *b*, and *d* is the number of sheets of paper in the sample:

$$\frac{a \times 1,000}{b \times c \times d} = \text{weight in lb. per ream } 25 \times 40, 500.$$

For samples of paper weighing less than 20 lb. on the quadrant scale a chemical balance is used. For convenience, the following formula is used:

$$\frac{(\text{Weight in grams}) \times (1,102) \times (1,000)}{(\text{Area of samples in sq. in.}) \times (\text{number of sheets})} = \text{weight in lbs. per ream } 25 \times 40, 500.$$

To convert the weight of the standard ream to the weight of a ream of the desired trade size, it is only necessary to multiply the weight of the former by the area of the latter and divide by 1,000, provided, of course, that the latter ream contain 500 sheets.

a. *Balances and Scales.* There are a number of available as illustrated. (Figs. 18, 19, 20, 21.) They may be calibrated by placing small accurate weights in the pan and taking readings on the scale. An average of several readings at uniform distances apart on the scale should be obtained.

b. *Conversion Factors.* The weight of a ream folio size, 17 x 22, 500, can be stated as the substance number.

A method for determining the substance number on small samples by the analytical balance is as follows: A flat piece of thin metal cut exactly 2 x 2-1/16 in. is held upon the sample and a sharp instrument run around the edge of the metal. The sample cut exactly 2 x 2-1/16 in. has a substance number equal to its weight in centigrams.

$$\frac{\text{Weight in centigrams} \times 500 \text{ sheets} \times .374 \text{ sq. in. per sheet}}{45,360 \text{ centigrams per pound} \times 4.125 \text{ sq. in. in sample}} = \text{substance.}$$

$$\frac{\text{Weight in centigrams} \times 178,000}{187,110} = \text{substance number.}$$

TYPICAL EQUIVALENT WEIGHTS IN STANDARD AND TRADE SIZES\*

Weight of ream, 25 X 40, 500 Lb.	Trade size ream, 500 sheets In.	Area of sheet In.	Weight of ream, trade size Lb.
52.6	25 X 38	950.0	50
64.2	17 X 22	374.0	24
100.0	20 X 25	500.0	50
156.0	22.5 X 28.5	641.3	100

Conversion between ream basis weight and grams per square meter.  
 Weight in grams per square meter =  

$$\frac{(\text{Weight in lb. of any ream, 500 sheets}) \times 1406.13}{\text{Area of sheet in sq. in.}}$$

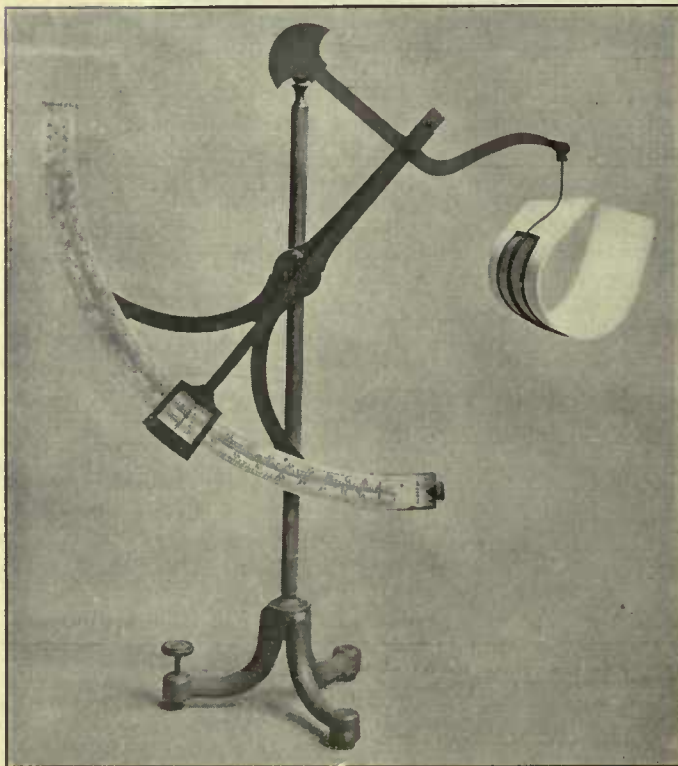


FIG. 18. QUADRANT SCALE

A typical scale for weighing paper. (Foreign Paper Mills, Inc., New York City).

$$\text{Wt. in lb. per ream of 500 sheets} = \frac{1406.13}{(\text{Wt. in grams per square meter}) \times (\text{Area of sheet in sq. in.})}$$

To convert to lb.	Ream size	To convert to grams
0.267	17 x 22 500	3.75
0.714	25 x 40 500	1.40
0.429	20 x 30 ...	2.33
0.591	24 x 36 480	1.69
0.675	25 x 38 500	1.48
0.618	24 x 36 500	1.62

Length of paper in a roll.

Length in ft. =	Roll weight	Ream area (sq. in.)
	$\frac{\text{Ream weight} \times \text{roll width}}{12}$	
Ream size	Factor	
17 x 22 500.....	15583	
25 x 38 500.....	38583	
25 x 40 500.....	41667	
20 x 26 500.....	21667	
20 x 30 480.....	24000	
24 x 36 480.....	34500	

5. Bursting Strength

a. *Description*—There are two types of testers available for de-

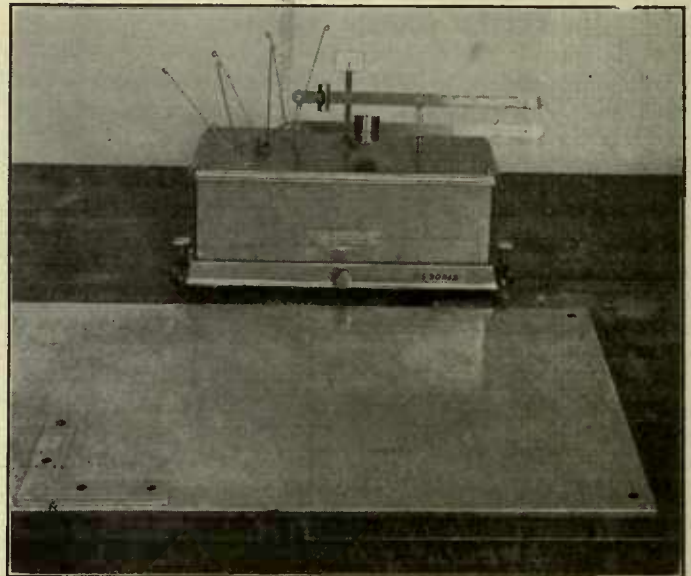


FIG. 19. TORSION BALANCE

A balance designed on the torsion principle, and with scale calibrated in pounds per ream. (Torsion Balance Co., New York City).

termining the bursting strength of paper and board. One is of the hydraulic type in which the paper is clamped against a rubber diaphragm, through which the pressure is applied to a circular area of the paper measuring approximately 1 sq. in. The pressure is indicated on a special Bourdon tube gage. The second type is of the spring operated metal plunger design in which the paper is clamped between annular rings, through which a spring operated plunger is forced.

b. *Comparison*—Although a large amount of data have been collected by individual laboratories with the instruments shown in the accompanying photographs (Figs. 22, 23, 24) very little information has been published. Certain conclusions, however, may be assumed and certain recommendations made. There is very little, if any, relation between the data obtained with these three testers. With averages of equal number of tests, the variation seems to be inversely proportional to the diameter of contact. There is no standard method of manipulation to assist various laboratories in duplicating results. There is no fundamental unit to which the testers may be referred for calibration. In addition to this, little attention is given to the care of these testers and in the case of the Mullen tester, the deterioration of the diaphragm is often neglected, as well as calibration of the gage. It is recommended that a definite method of making these tests be determined upon and precaution given for the care of the testers. In any case, the bursting strength of a sample as reported should be the average of not less than ten individual tests.

c. *Ratio*—The bursting strength test to be of greatest use must be expressed in terms of the weight of the sample. This ratio of



strength to weight may then be directly compared with the strength ratio of any other paper.

The strength ratio may be expressed as a percentage.

$$\text{Strength ratio} = \frac{\text{Bursting strength} \times 100}{\text{Wt. in lb. (on a size 25 x 40, 500)}}$$

### 6. Thickness

*a. Description*—The thickness of a paper may best be determined by the use of a spring micrometer having a hand that travels around a circular dial. This dial is graduated into thousandths of an inch. This direct reading type of thickness gage should not be read closer than half of a thousandth, as they are not accurate beyond that point. The following is a list of manufacturers of this type of thickness gage: B. C. Ames Co., Waltham, Mass.; B. F. Perkins & Son, Inc., Holyoke, Mass.; Storrs & Bement Co., 140 Federal Street, Boston, Mass.; The Ashcroft Mfg. Co., 85 Liberty

compactness of several papers, the following formula is suggested:

$$\frac{\text{Thickness in thousandths of an inch}}{(\text{Weight } 25 \times 40, 500)} \times 10,000 = \text{Relative compactness.}$$

The factor 10,000 serves to give a resultant figure more rapidly remembered. A very highly compressed paper may show a relative compactness of 0.600, while a very spongy or fluffy sheet may bulk to 1.300. This last sheet is more than twice as bulky as the former mentioned.

*b. Variations*—A superficial inspection of the different types of

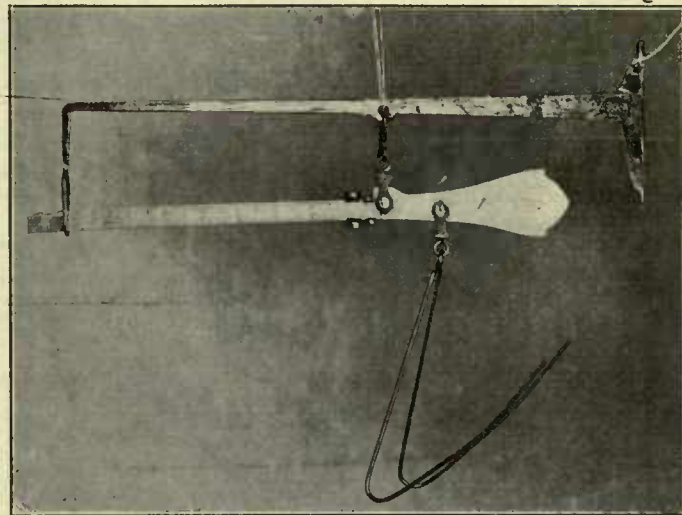


FIG. 21. PEA AND BEAM SCALE

A typical scale for rough work for use in the machine room. (Fairbanks & Co., New York City).

micrometer gages available for determining the thickness of paper, indicates that there is little or no uniformity in their construction. It is found that there are considerable differences in spring pressure, in size and shape of the contact areas and in the size of the dial divisions. It would seem that these differences would affect the accuracy of the test when different gages are used and it is, therefore, emphasized that the test is not accurate closer than one-half of a thousandth of an inch.

### 7. Bulk

The bulk of paper is the thickness of a certain number of pages and applies more particularly to book papers where the printer desires a book of a certain number of pages to bulk one inch. The bulk of a paper is measured by cutting out short strips of paper, piling them up to the required number and measuring the combined height of the pack. This measurement may be made by the use of a Perkins bulk tester (Fig. 26). This instrument measures the bulk in inches, also the pressure of clamping, and takes the place of the ordinary graduated sliding clamp which is in common use. In specifying the bulk of a paper, where the hand clamp is used, it is necessary to specify whether heavy, medium or light pressure is used. In using the Perkins bulk tester, the pressure is specified in pounds per square inch, as indicated on the dial.

### 8. Folding Endurance

*a. Description*\* (53). The folding endurance of a paper is measured on a machine in which a strip of paper of definite width and length is clamped. The clamps are held apart under definite tension and the paper is caused to bend back and forth upon itself, until the fibers wear through at the line of folding. The number of double folds is recorded automatically.

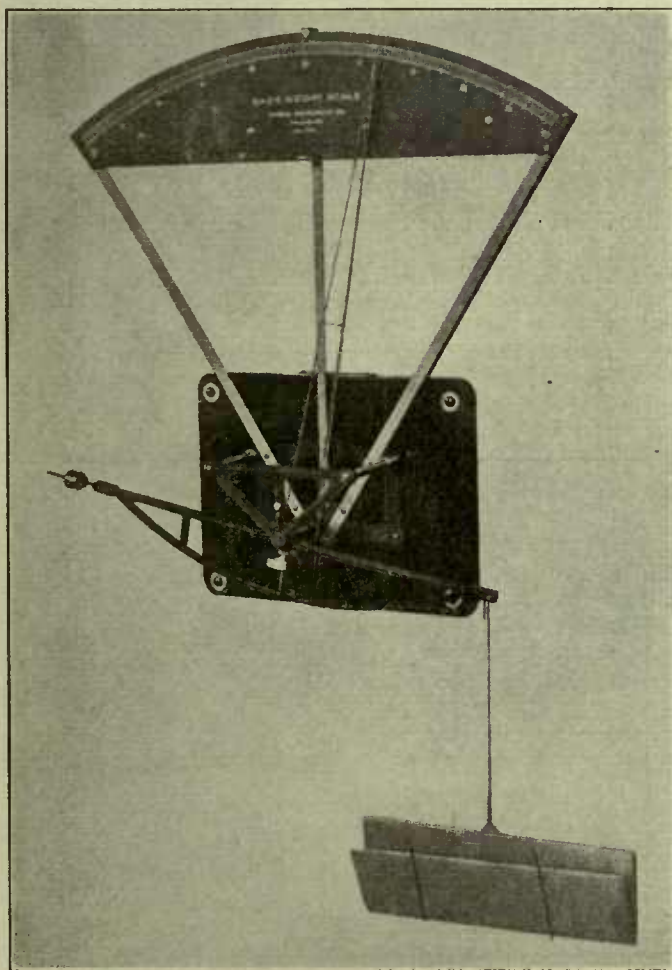


FIG. 20. SHEET WEIGHING DEVICE

An accurately made delicate balance for weighing paper. (Thwing Instrument Co., Philadelphia, Pa.)

Street, New York; Cornelius Kahlen, 349 Broadway, New York; E. J. Cady & Co., Chicago, Ill.

It is advisable to have all thickness gages calibrated before use. This may best be done by securing a set of standard sheet metal leaf gages, which range from 0.001 to 0.015 inch. This range of leaf gages covers the ordinary range needed in testing most papers, and should be used periodically to see that the instrument for measuring thickness remains accurate.

For the purpose of obtaining a quick comparison of the relative

The folding strength of paper is dependent not only upon the strength and durability of the paper, but also is very largely influenced by the relative humidity. To perform this test in the most accurate manner it is therefore necessary to keep the relative humidity constant for all tests. This can only be done by making the test in a room where the humidity is under control. Where such a room is not available then note must be made of the per cent relative humidity of the air at the time of the test. No tests should be attempted when the humidity is either very high or very low. A relative humidity between 65 and 70 per cent is more easily attained throughout the year and is the standard humidity recommended by the paper testing committee.

The folding factor is determined by the following formula:

$$\frac{\text{Folding endurance}}{(\text{weight } 25 \times 40, 500)} = \text{Folding factor.}$$

The folding factor will vary between about 0.1 and 200.

*b. Calibration.* The machine illustrated (Fig. 28) was designed at the Bureau of Standards for the purpose of calibrating the springs acting on the clamping jaws. It consists of a stand on one end of which the tester can be screwed firmly. On the other end is provided a support, with leveling screws, on which may be mounted a wheel provided with knife-edge bearings. Around the rim of the wheel is a groove of about  $\frac{1}{4}$  in. deep. This wheel should be made of three plies of wood, glued to prevent warping, and should be so balanced that when mounted on the supports with the back of the knife edge horizontal, it will remain in any position within 45 degrees either side of this without tending to rotate. The table on which the folding tester is mounted should be made large enough so that the machine can be set up with either jaw facing the large grooved pulley. The relation between the height of this table and that of the bearings for the knife edge supporting the grooved pulley should be such that a fine piece of thread resting on the base of the groove in the pulley and attached to the center of one of the clamping jaws, the other clamp being removed entirely, will form a horizontal line. After the jaws have been properly marked for maximum extension according to Reid, Veitch and Sammet, one of the jaws with its spring holder and stand should be removed

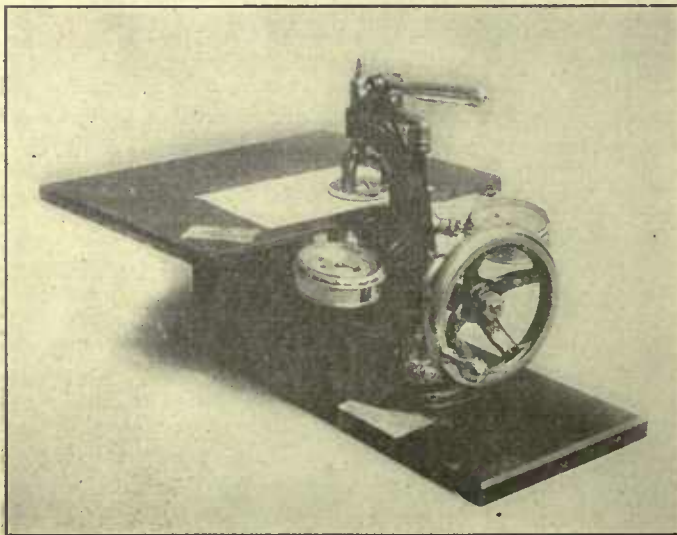


FIG. 22. MULLEN BURSTING TESTER \*(89)

A hydraulically operated bursting tester in which the pressure is applied to the paper through a rubber diaphragm and indicated on a gage. (B. F. Perkins & Sons, Holyoke, Mass.)

entirely from the machine and the latter should then be mounted with the end without the jaw and spring toward the grooved pulley. A thread or very fine wire should be attached to the center of the clamping jaw, passed through the reciprocating slot (the latter being locked in its neutral position) over the wheel, and tied to a

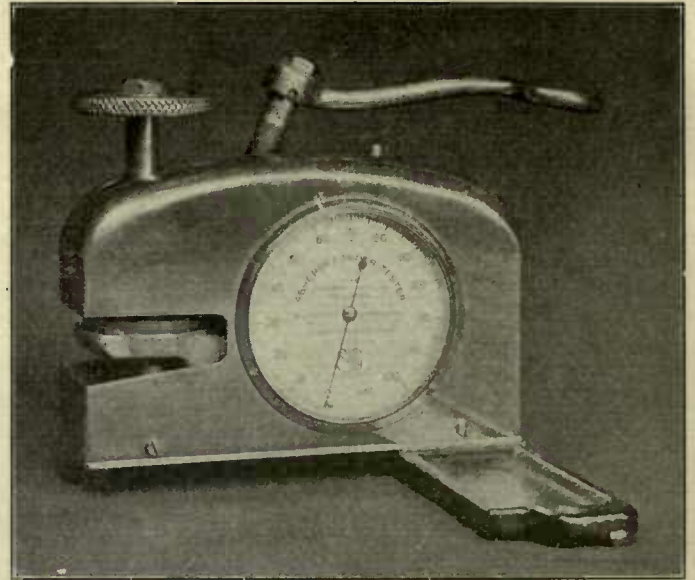


FIG. 23. ASHCROFT TESTER

A spring operated metal plunger bursting tester. (Ashcroft Manufacturing Co., New York City).

1 kg. weight so that the latter swings free, and the thread falls entirely in the plane of the groove in the pulley. If the reciprocating part be locked in its neutral position and the alignment of the machine and pulley be carefully made, it will be unnecessary to re-

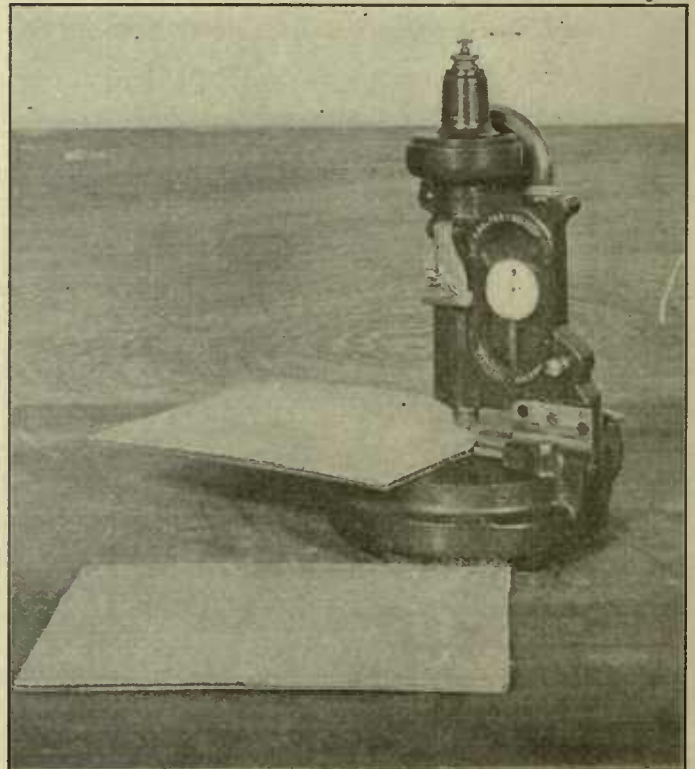


FIG. 24. WEBB TESTER

A device developed for testing corrugated and solid fiber container board. It is of the spring operated metal plunger design. (Container Club, Chicago.)

move that part of the machine which carries the four small rollers and through which the reciprocating part passes. After setting up, care should be taken that the thread holding the weight does not touch any part of the folding tester and that the square shank of the jaw is entirely free from any contact with the sides of the

square opening into which it fits. If the spring tension is correct the jaw will be drawn out by the action of the 1 kg. weight so that the mark previously made to the square shank will be just visible. If this is not the case the spring tension should be adjusted by loosening the small set screw holding the knurled collar on the end opposite the jaw, after which the tension is adjusted by revolving this knurled collar. The other jaw should be adjusted in the same way after reversing the machine, removing the jaw just calibrated and replacing the other.

*c. Accuracy.* In view of the fact that the folding test is practically confined to a test strip 15 mm. long and about 0.25 mm. wide, that wearing parts tend to make it difficult to maintain uniform conditions of the tester and because of the very marked effect of changes of relative humidity, it is probable that the variations between averages of ten tests, either on the same machine or on different machines, will vary from 5 to 15 per cent under ideal conditions and that this variation will be considerably greater under the normal testing methods. It is recommended that an average of not less than ten tests on the sample in one direction be obtained to indicate the folding endurance in that direction.

*Note.*—In connection with the folding tester, attention should be called to the fact that it is absolutely essential that the small steel wheels supporting the clamping jaws be perfectly round, well oiled and revolve easily, as the jaws move back and forth. In one case the fact that one of these did not revolve caused an error of 25 per cent in the results.

GREEN FOLDING TESTER

This folding tester (Fig. 29) was originally devised to apply to a pulp sheet made either in the hand mold or by means of the standard pulp sheet mold. The folding tester is, therefore, applicable to any sheet product, irrespective of bulk or tensile strength. It consists of two planes of hardened steel, ground true, which abut along a straight line, accurate to ground fit. These two planes have a slight inclination downward from the line along which they abut. They are pressed together along this line by means of springs, set to a specified tension. Running over these planes are two rollers, each parallel with the line along which the planes abut, set a fixed distance apart, and pressing downward on the planes by a known force, which is fixed by the adjustment of springs. By means of constant speed electric drive, the rolls are run backward and forward across

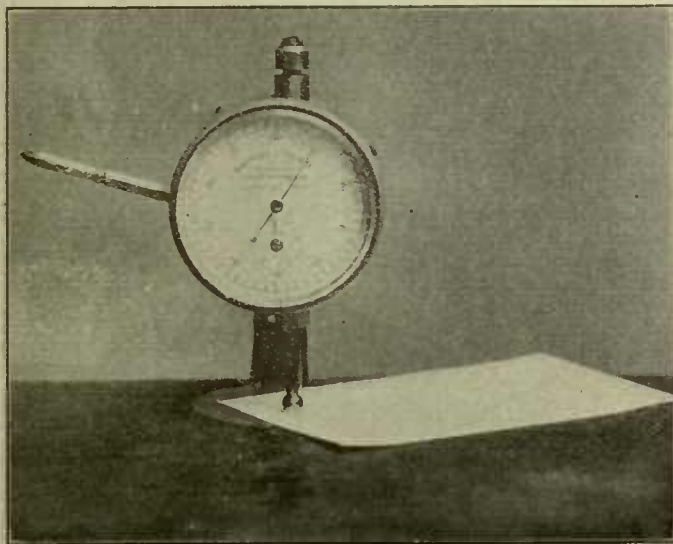


FIG. 25. THICKNESS TESTER \*(89)

A typical micrometer gage for testing the thickness of paper. (A. Storrs & Bement Co., Boston, Mass.)

the line along which the two inclined planes abut and the specimen under test is held between the planes under known and constant pressure. There is an automatic counting attachment which registers the number of double folds of the specimen. The end point

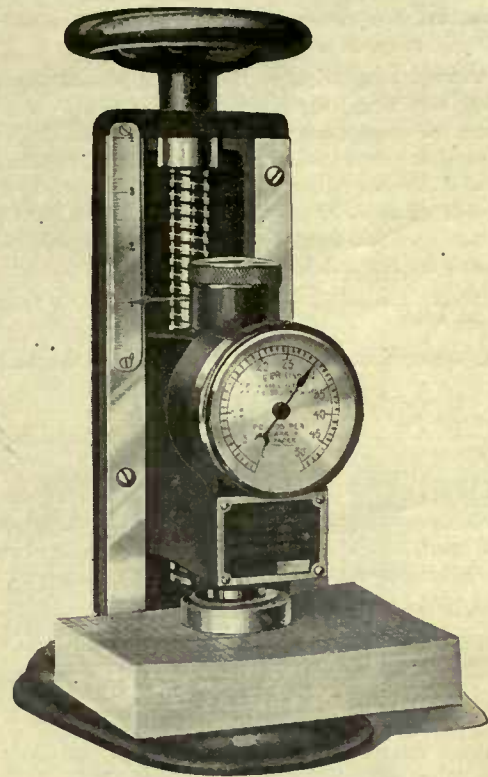


FIG. 26. BULK TESTER

A useful apparatus for determining the bulk or the number of pages to an inch of a particular kind of paper. (B. F. Perkins & Sons, Inc., Holyoke, Mass.)

is the moment when the specimen has been severed at its center, not when it has been severed entirely.

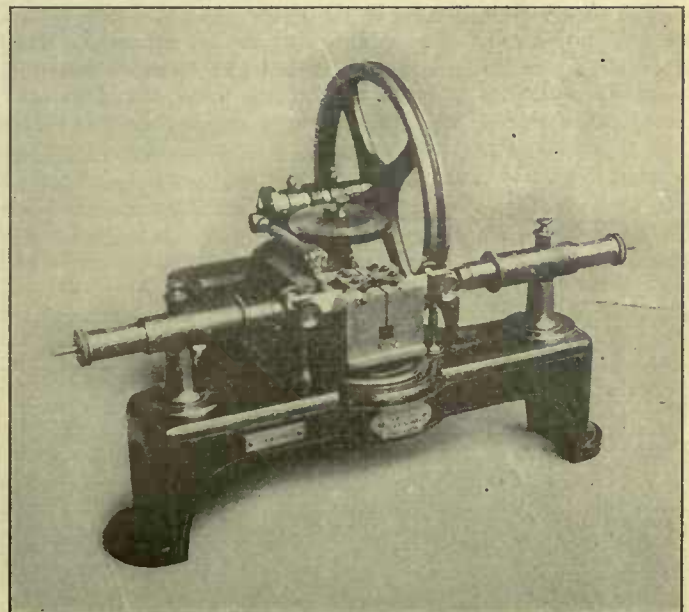


FIG. 27. FOLDING TESTER

A device for determining the folding endurance of a strip of paper 15 mm. wide under a tension of 1 kg. (Foreign Paper Mills, Inc., New York)

9. Tensile or Breaking Strength

*a. Description.* The tensile strength of paper is determined by the load, in pounds, required to break a strip of paper. The tensile

strength machine, best known in the paper industry, is the Schopper tensile machine (Fig. 30).

In this device a strip of paper 15 mm. (approximately 19/32 in.) wide by 180 mm. long (approximately 7 3/16 in.) is clamped at each end and the clamps are moved apart until the strip is broken. A suitable device indicates the pull in kilograms (approximately 2.2 lb.) required to break the strip. As the English units of measurements are used on all other tests, it is recommended that the load in kilograms per 15 mm. width strip, be converted into pounds per inch of width by the following formula:

$$(3.73) \times (\text{Tensile strength in kg. per 15 mm. width}) = \text{Tensile strength in lb. per 1 in. width.}$$

A tensile strength factor may be determined by the following formula:

$$\frac{(\text{Tensile strength in lb. per in. width})}{(\text{Weight } 25 \times 40, 500)} = \text{Tensile strength factor.}$$

The usual factor for tensile strength is known as the breaking length. This is the length of a strip which, if suspended at one end, would break of its own weight. The following formula may be used to determine the breaking length of a sample:

$$\frac{(\text{Tensile strength per 1 in. width}) \times (13,889)}{(\text{Weight of a sheet } 25 \times 40, 500)} = \text{Breaking length in yds.}$$

$$\frac{(\text{Tensile strength per 15 mm. width}) \times (13,889 \times 3.73)}{(\text{Weight } 25 \times 40, 500)} = \text{Breaking length in yds.}$$

The breaking length factor will range from a maximum of about 11,000 yards down to approximately 2,000 yards.

In this test certain precautions should be observed in order to get accurate results. The width of the sample, the rate of applying the load and the alignment of the sample in the jaws will affect the test. Care should be exercised that the test strip should be cut accurately to the prescribed width and that it should be cut accurately parallel to either the machine or cross direction of the paper. It is recommended that the lower jaw be moved at a rate of 12 in. per minute for the 50 kg. tester. The test strip should be carefully inserted in the jaws, so that the pull is straight, since otherwise, a tearing strain will be introduced that will produce an error. It is further recommended that the tester should be calibrated and, if necessary, a correction curve and formula be derived.

*b. Wet Tensile\** (122). This test may be performed on the same apparatus as for the dry tensile strength with certain modifications. Due to the weakness of paper when wet, it is desirable to make the apparatus more sensitive and this is done by removing the weight at the bottom of the moving arm, calibrating the tester

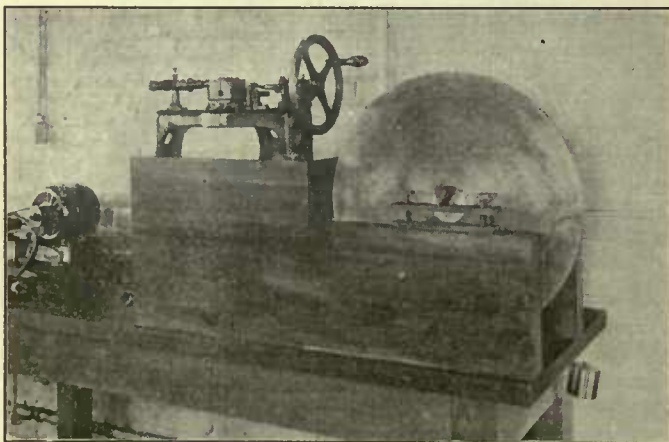


FIG. 28. CALIBRATING DEVICE

An arrangement designed for the purpose of calibrating the spring tension of the Schopper folding tester. (Bureau of Standards, Washington, D. C.)

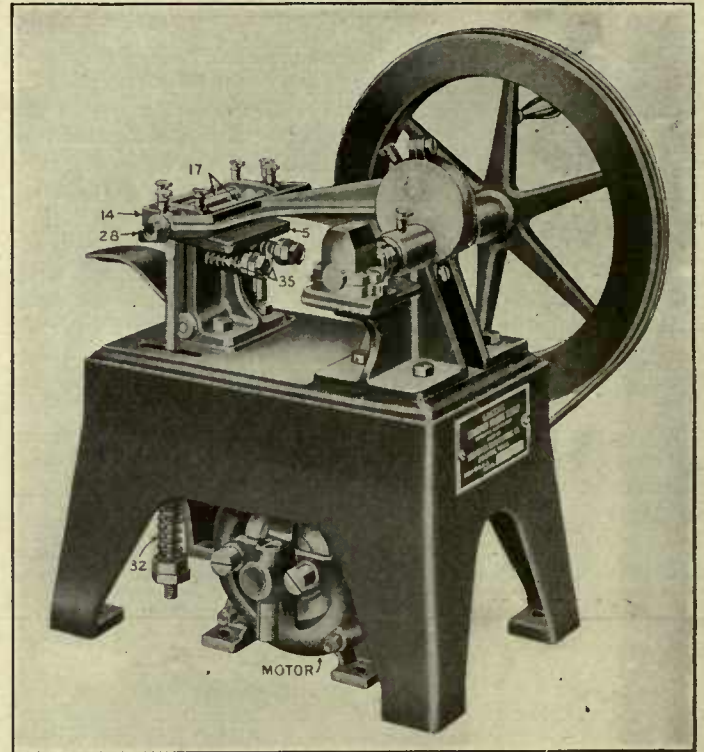


FIG. 29. GREEN FOLDING TESTER  
Star Brass Manufacturing Co., Boston, Mass.

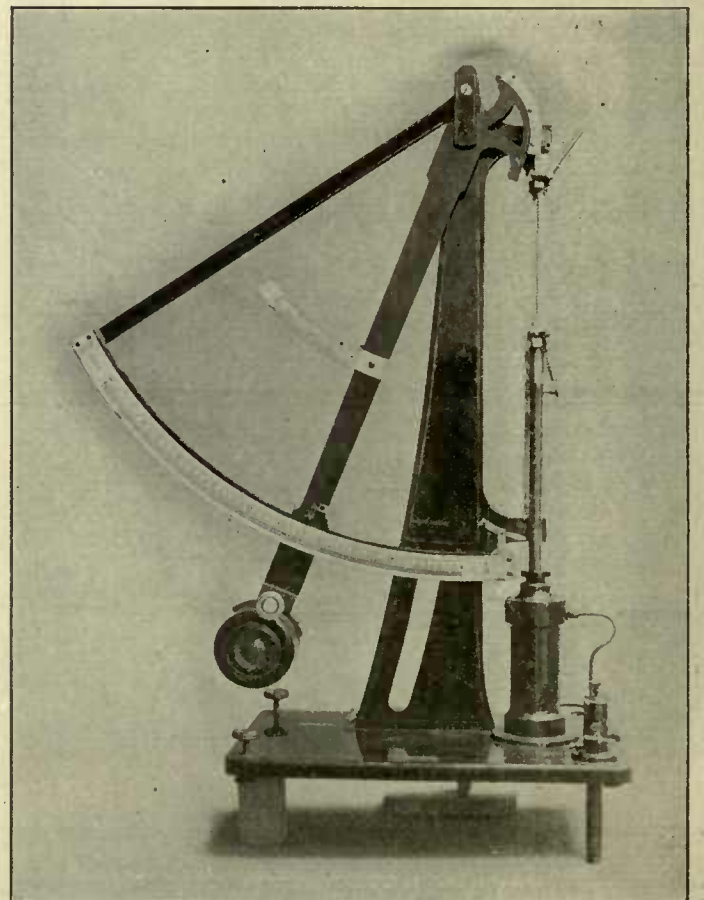


FIG. 30. SCHOPPER TENSILE TESTER

Tensile tester operated hydraulically with device for determining the elongation of the paper. (Foreign Paper Mills, Inc., New York)

under this new condition and obtaining a correction curve or factor for conversion. It is recommended that a speed of 6 in. per minute be applied to the lower jaw and that this be kept uniform. It is recommended that before testing, the strips shall be immersed in water at 70° F. for 20 minutes and that these conditions be rigidly observed, as the test is markedly influenced by both the temperature of the water and the time of immersion. Since the test strips, after wetting, are easily injured and are difficult to place in the jaws in true alignment, extra care is necessary.

c. *Stress Strain\** (20). In connection with the determination of the breaking strength of heavy bag papers, it has been indicated that a study of the stretch due to repeated application of load is of considerable importance in indicating the quality of the sample for this purpose. The tensile strength of the paper is determined, using a strip 1 in. wide and 12 in. long, the load being applied at the rate of 12 in. per minute. By means of a recording device, shown in the accompanying photograph (Fig. 31), the stretch of the sample under repeated loads (10 per cent less than the average breaking strength) is indicated and it is possible to determine the stretch, regain and elasticity of the sample by this method.

d. *Elongation at Rupture*. Most of the tensile test devices are equipped with a secondary scale to indicate the stretch or elongation at the time of rupture. This secondary quadrant has two scales and gives the stretch in mm. or, if a test strip of 180 mm. between jaws was used, in percentage stretch. Data available seem to indicate, however, that this elongation at rupture has little significance in evaluating a test sample.

10. Absorption

a. *Strip\** (6). The absorption of a blotting paper is indicated

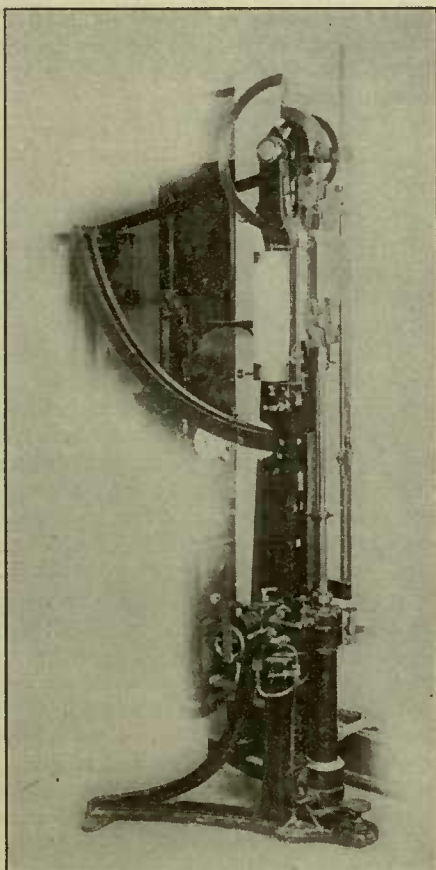


FIG. 31. STRESS STRAIN TESTER

A hydraulically operated 200 kg. Schopper tensile tester with recording drum to indicate the stretch on the repeated load. (Bureau of Standards; Foreign Paper Mills, Inc., New York.)

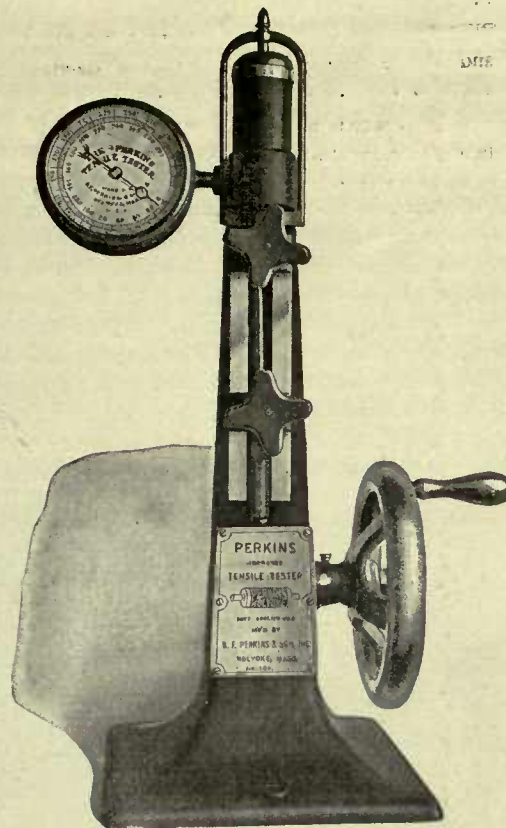


FIG. 32. PERKINS TENSILE TESTER

A tensile tester recently brought out by B. F. Perkins & Son, Holyoke, Mass.

by the height in mm. to which, in a given time, a liquid will rise by capillary action, when one end of a strip of paper held vertically is immersed in water. The height in mm. to which the liquid (preferably water) will rise in 10 min. is taken as a measure of the relative absorption of the paper.

In making this test, using the 'strip' method (Fig. 33), a strip of blotting paper 15 mm. (about 3/5 in.) wide and 150 mm. (about 6 in.) long is suspended, so that the lower end dips 3 mm. (about 1/8 in.) into a pan of distilled water. Besides the strip is a scale reading in mm. (fractions of an inch), and at the end of each minute for 10 minutes readings are taken of the height to which the liquid rises in the strip. Five tests are made in both the "machine" and "cross" direction and an average obtained. The result is reported as the height to which the liquid will rise in 10 min. When necessary, or advisable, the same strips may be subjected repeatedly to the test, which will indicate the decreasing ability to absorb water or ink. In addition, a standard ink of the following formula may be used:

	Grams
Tannic acid (dry).....	23.4
Gallic acid (crystals).....	7.7
Ferrous sulphate (crystals).....	30.0
Dilute hydrochloric and (U. S. P.; sp. gr. 1.049; 10% HCl by weight	25.0
Phenol .....	1.0
Bavarian blue, S. & J. No. 478 or similar suitable dye.....	2.2
Water to make a volume of 1,000 cc. at 15.6° C.	

Note—Any water-soluble basic aniline blue, as Niagara 3B, National Aniline Company, may be used in place of Bavarian blue.

b. *Pipette\** (13). In this test a 1 cc. pipette is employed and is suspended in such a way that the end of the pipette is 1/2 in. from the surface of the test sample of blotter. The test sample cut 4 in. square is laid felt side up upon a coarse wire screen, which is supported by a large beaker. This is done to prevent as far as possible the blotting paper from caving in at the center where the liquid fell upon it. (The felt side of paper is the top side of the paper as it leaves the paper machine wire.) Both distilled water

and the above-mentioned government standard ink are used at the three temperatures of 60, 70, and 80° F.

A stop watch is used to measure the time it took the 1 cc. of liquid to leave the pipette until it is totally absorbed by the paper. Also the diameter of the circular spot on the paper was measured immediately at the completion of the time reading.

c. *Total Absorption\** (23). By means of this test, test samples cut 2 in. square are first weighed on a chemical balance and then dropped with the felt side down on a trough of distilled water and also on a trough of government standard ink. The same temperatures are used for the ink and water as in previous absorbency tests. After a 10 minute period of absorption, the samples were taken out, drained ½ minute and again weighed to determine the amount of liquid absorbed.

d. *Blotting Test\** (23). In this test, small strips of blotting paper cut ½ in. wide by 4 in. long are used to blot signatures that are

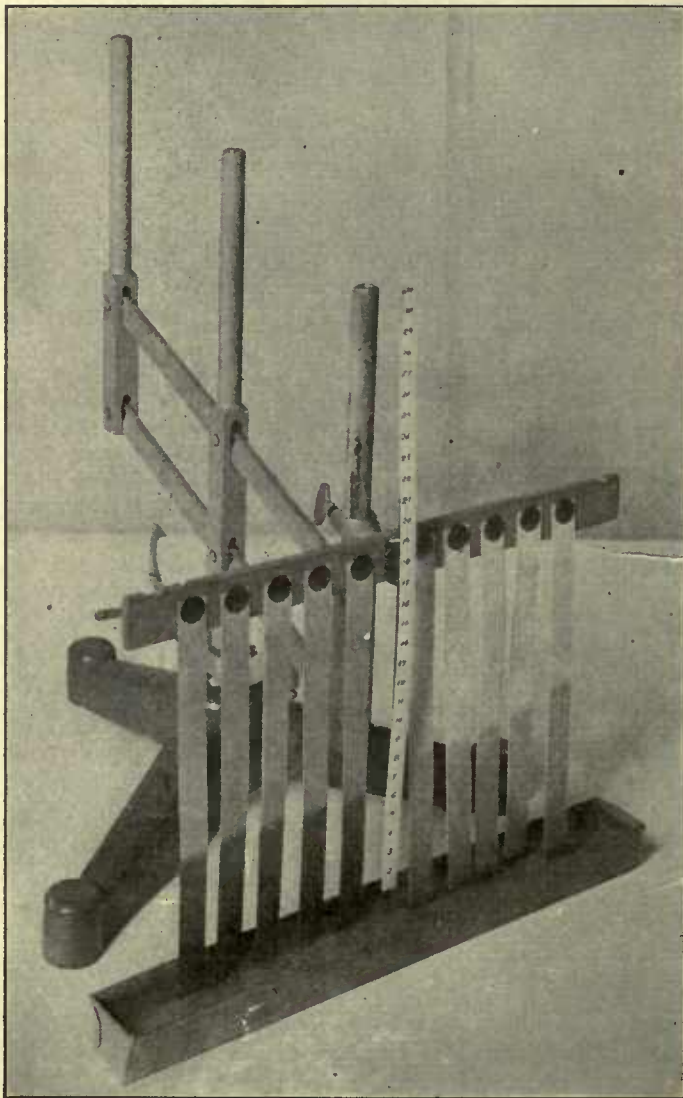


FIG. 33. ABSORPTION TEST

A convenient method of making a number of absorption tests of blotting paper by the Klemm method. (Bureau of Standards)

written with a stub pen on ordinary bond paper. The same signature is used throughout the test and only one signature is blotted at a time. The small size of the test sample causes each blot to be made on almost the identical spot in the blotting paper. Gov-

ernment standard ink is used as in previous tests and a record is kept of the number of times each test sample will blot the signature before the ink shows signs of spreading on the paper. The felt side of both blotting and bond paper is used throughout the tests, and

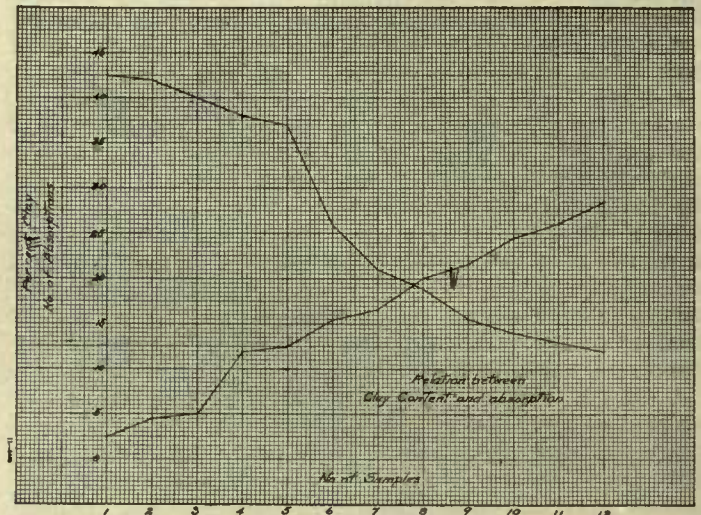


FIG. 34. RELATION BETWEEN FILLER AND BLOTGING QUALITY

Curves representing the variation of a practical blotting test with the percentage of clay in the paper

an average of three tests is taken as a final result for each blotting paper.

It is interesting to note in the accompanying curves (Fig. 34) that there seems to be an inverse proportion between the amount of ash of the paper and the number of times that the test strip may be used before the ink begins to spread.

#### 11. Opacity and Translucency\* (142)

The opacity or translucency of a paper may be measured by the "contrast ratio" method as described in Bureau of Standards Circular No. 63 (Fig. 35).

The Bureau of Standards has developed and adopted a standard method for determining the transparency of paper and tracing cloth, which is described in detail in Circular No. 63. Briefly, this method consists in placing a sample of the paper or cloth to be tested over two adjacent surfaces, one white, and the other black, and measuring the reduction in contrast of the appearance of the two surfaces. If the material in question is quite transparent, the contrast between the black and white surfaces, as seen through the material, will be quite noticeable; but if the material is opaque, none of the light incident upon its surface will be transmitted and absorbed by the black surface beneath, and consequently, there will be no contrast between the parts of the material covering the black and white surfaces.

In making the measurements, one must use a photometer having a divided photometric field, one-half of which is illuminated by the light coming from the material over the white surface, while the other half is illuminated by the light coming from the material over the black surface. The two halves of the photometric field are then "matched" by usual observation and properly setting the photometer, and the indicated results recorded. A slight computation based on these observations, gives the numerical measurement sought, which is called the contrast ratio. It varies between zero and unity, larger values indicating less transparency.

#### 12. Tearing Test

A continued and increasing interest in the determination of the tearing strength of paper by some mechanical means has produced

various devices for this purpose. A brief description of several is given, but there is not as yet sufficient data available to make any recommendations in regard to those which are most satisfactory.

a. *Knife Edge Method\** (133). In the tester perfected at the A. D. Little, Inc., Laboratory, the sample to be tested is clamped to the baseboard of the instrument. The clamp has a knife edge along one side and the paper is torn along this edge. The tear is first started slightly and the end of this strip is clamped to the movable arm which is connected to a bellows arrangement with a pressure gage. The arm, bellows and gage are mounted on a movable carriage, operated on an inclined track by means of a lug screw. As this screw is made to revolve, the carriage moves up the incline and, as one end of the paper is attached to the movable arm, the tearing is accomplished. The force necessary to tear the paper is determined by the pressure shown on the gage.

b. *Witham and Case Testers*. Both these testers are based on the principle that the force necessary to tear the sample is applied by running water into a beaker which may be connected to the sample in any one of several ways. In the Witham tester\* (136, 148), this load is applied by means of running water from a burette into a beaker, set on one end of the pivoted arm, the opposite end of which is attached to the sample. In the Case tester\* (128), the movable clamp is merely a spring clothes-pin, to which is attached a small bucket. Water is run into the bucket until the sample is torn.

c. *Schopper\** (129). The heavy counter-weight of the Schopper tensile tester (Fig. 38) is removed, the test sample is cut 1 in. wide and 4 in. long, slit down the middle about 2 in., and one of the slit ends is placed in each of the jaws. The pawls on the rack are raised, the lower jaw lowered at a uniform rate and readings taken at intervals of 10 sec. As in the case of the wet tensile test, the tester must be calibrated for the new conditions and to avoid a rubbing effect when several plies are used, the lower jaw may be offset as indicated in the accompanying photograph.

d. *Elmendorf\** (131, 134). The principle upon which this tester (Fig. 37) is based is the fact that the tearing force is determined

test sample is recommended and care must be exercised that the width of the test sample is exact and the placing of it in the jaws is carefully performed. The tester is calibrated for 16 plies of paper and if any other number is used, the necessary factor must be applied.

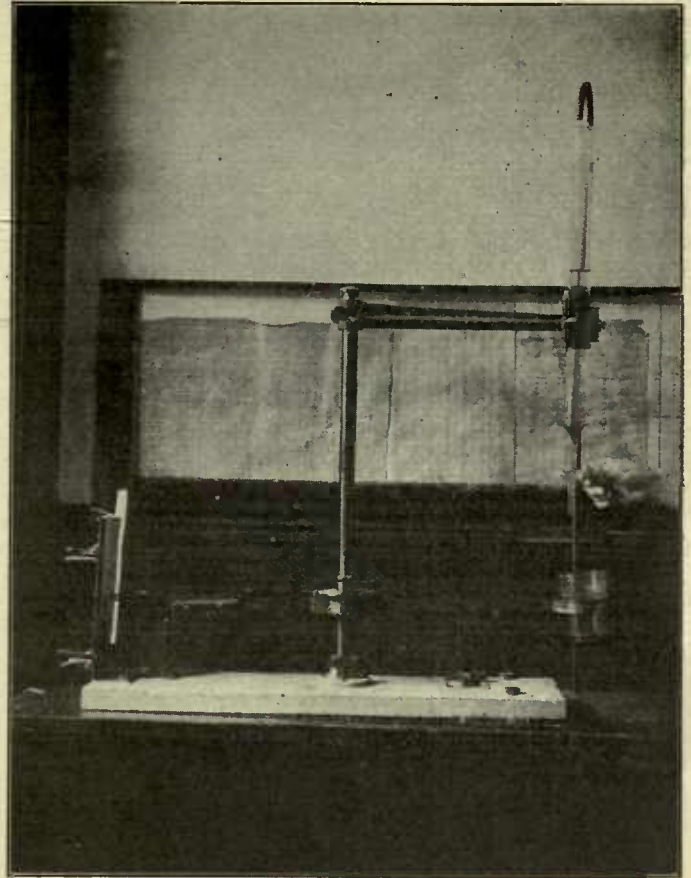


FIG. 36. WITHAM TEARING TESTER

An early type of tearing tester developed for the use of testing bag paper. (G. S. Witham, Sr., Hudson Falls, N. Y.)

e. *Discussion of the Tearing Test*. Available data seem to indicate that there are a number of factors which influence the results obtained in making this test by any of the above-mentioned methods. These factors are: (1) width of sample and width of paper on either side of the tear, (2) the number of sheets or plies torn at one time, and (3) the relative humidity at which the tests are made. Sufficient data are not available for conclusions to be reached in regard to these factors and it is, therefore, impossible at this time to make any recommendations in regard to the efficiency or accuracy of these methods or to interpret the data obtained.

### 13. Degree of Sizing

Several methods have been proposed for determining the sizing quality of paper. Practically all of these methods are merely comparative and a recent series of tests indicated that no two laboratories or observers would grade various samples even in the same order. The chief criticism of the majority of the methods used is that no account is taken of the thickness of the paper.

a. *Flotation Methods*. A simple qualitative test to indicate the effectiveness of the sizing as a preventive of the absorption of ink, may be made by using the Ink Flotation Test\* (110). This method involves the drawing of a strip of paper over the surface of an iron tannate ink and allowing it to drain and dry naturally. Upon examination of the surface with a low-power microscope, a well-

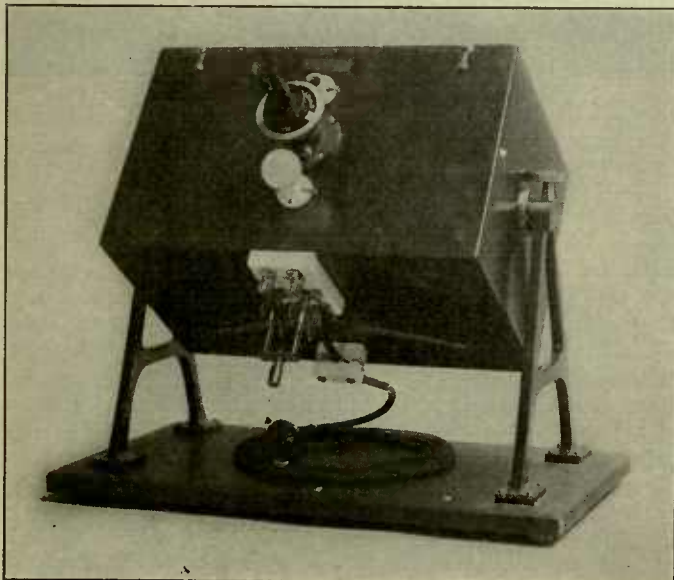


FIG. 35. OPACITY APPARATUS

A device for measuring the opacity or translucency of paper by contrast-ratio method. (Bureau of Standards)

indirectly by means of the work required to tear a sheet of paper. The moving segment of the instrument may be considered to be a pendulum, whose swing is retarded by the tearing of the paper. The tearing force is determined from the relation existing between work, length of tear and force. More than one ply of paper in the

sized paper will show no indication of the fiber having absorbed the ink. Any variation in the depth of color on the surface will indicate a lack of uniform sizing. This test may be still further developed by erasing the surface with an ink eraser (a spun glass eraser is most suitable) and again dipping the sheet as before. A paper well sized throughout the sheet will show little or no additional absorption of ink at the erased spot. This test is only comparative but may be valuable to a mill in checking the daily progress.

For comparative sizing effect, squares 2 by 2 in. are cut from each sample. These are subjected for at least 30 min. to the same atmospheric conditions. Each square is then dropped upon an ink bath and the time in seconds recorded from the moment the sample touches the ink to the penetration of ink through the upper surface of the sheet. The average of an equal number of determinations, at least six, is used for comparative sizing effect in each sample. It is absolutely essential that comparative tests be made under identical atmospheric conditions and by the same operator, because atmospheric moisture and the ink bath temperature may greatly influence the penetration of the paper and different people have different judgments as to when the ink is through.

The ink used for the above test is made as follows:

Tannic acid (dry).....	23.4 gr.
Gallic acid (crystals).....	7.7 gr.
Ferrous sulphate (crystal).....	30.0 gr.
Dilute hydrochloric acid (U. S. P.; sp. gr. 1.049; 10% HCl by weight).....	25.0 cc.
Phenol.....	1.0 gr.
Blue dye (Bavarian Blue S & J No. 478).....	2.2 gr.
Water to make up to 1,000 cc., allow to settle, and decant from any sediment.	

Note—Any water-soluble basic aniline blue, as Niagara 3B, National Aniline Company, may be used in place of Bavarian blue.

Keep the temperature of the ink constant. Use the ink but once.

b. *Electrolytic Method.* \*(105, 112). Since 1917 there has

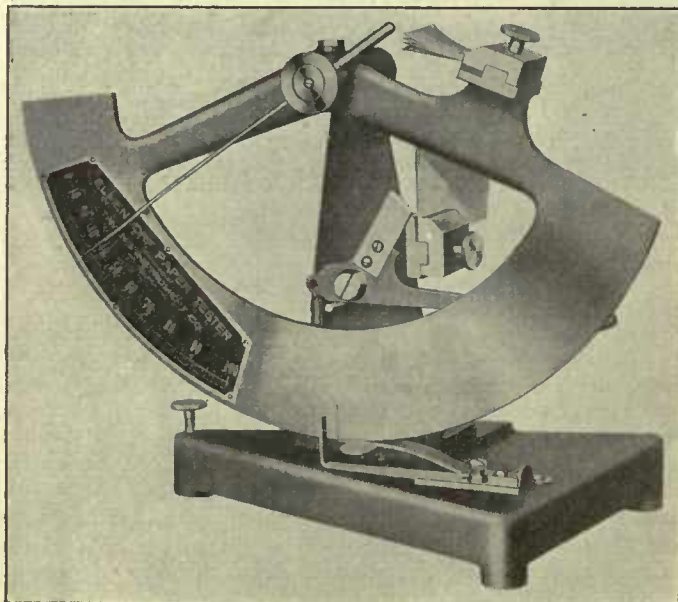


FIG. 37. ELMENDORF TEARING TESTER

A pendulum type of tearing tester which measures the work done in tearing several strips of paper. (Thwing Instrument Company, Philadelphia, Pa.)

been an increasing interest and use of the conductivity or electrolytic method for determining the sizing quality of paper. There have been a number of variations of the principle first proposed by Okell but they are all modifications to attempt to make the method of more value and to make it possible to interpret the data obtained.

When a sheet of paper between two electrodes is surrounded by an electrolyte and an alternating current is passed through the whole, there follows a decreasing resistance or increasing conduct-

ance as the electrolyte penetrates the sample. The sample of paper is clamped in the cell unit, which is itself a part of a wheatstone bridge. The accompanying photograph (Fig. 40) illustrates the various parts of a rather elaborate outfit. In this case, a recording

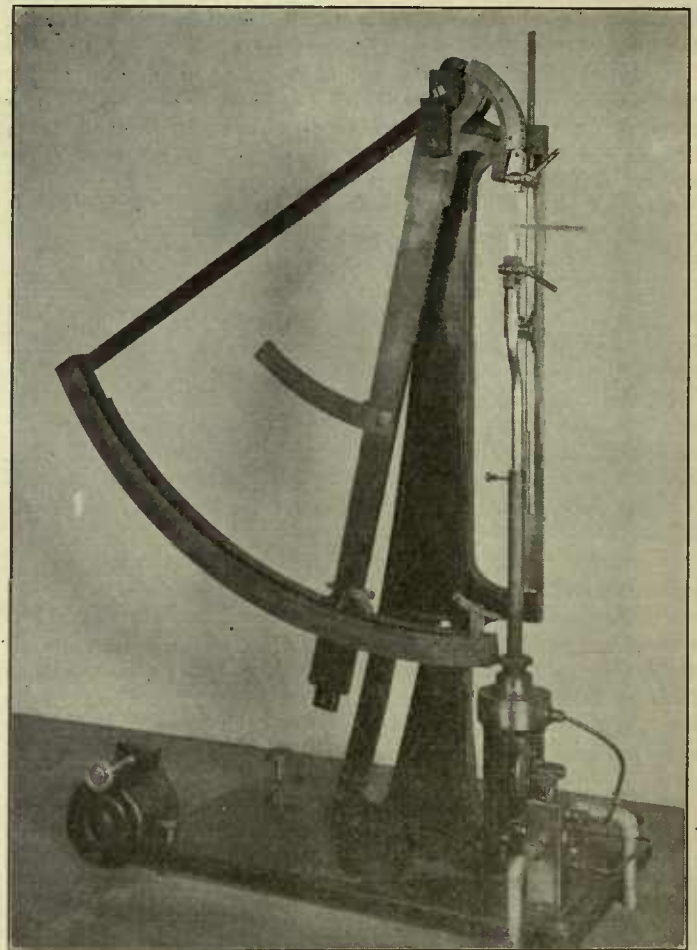


FIG. 38. SCHOPPER TEARING TESTER \*(129)

A tensile tester adapted for making tearing tests with the heavy weight removed and with an off-set lower jaw. (Foreign Paper Mills, Inc., New York.)

drum is attached so that the data are plotted as a curve. The value of this method is that, in principle it may be made very accurate, the curves may be reproduced with test samples from the same sheet, and the curve always presents a very regular aspect. The method is of considerable value as a control method during mill runs, after a curve has been determined for a particular kind and weight of paper. It does not seem possible, however, with the limited data available to recommend the method for general use and it is doubtful that the data can be interpreted in terms of any fundamental unit.

A practical laboratory instrument recently developed is shown in Fig. 39.

c. *Stöckigt Method.* Reference is made to a method for determining sizing quality found in *Wochenblatt für Papierfabrikation*, 1920, p. 39 (translation, *Paper*, March 10, 1920), by Fritz Stöckigt. The method involves floating a piece of paper to be tested on a 2 per cent solution of ammonium thiocyanate ( $\text{NH}_4\text{CNS}$ ) and applying to the upper surface a 1 per cent solution of ferric chloride ( $\text{FeCl}_3$ ) as an indicator.

14. Finish or Gloss\* (56)

a. *Ingersoll Glarimeter.* The glarimeter (invented and developed by L. R. Ingersoll for the Forest Products Laboratory), is an in-



strument for measuring the gloss or degree of finish of paper. It depends in principle on the fact that light reflected at an oblique angle from a sheet of paper is partially polarized, the degree of polarization depending on the gloss and being taken as a measure of it.

The way in which this principle is applied is made clear by the diagram (Fig. 42). Unpolarized (ordinary) light from the lamp is partially polarized on reflection from the sample of paper, P, and then enters the polarimeter or "glariscopes," which consists of a slit, S, quartz Wollaston double-image prism, W, lens, L, and small nicol prism, N, mounted in a divided circle. The eye at E sees a field of view divided into two parts illuminated respectively by the diffusely and specularly reflected light from the paper, and the diffusely reflected light alone. A setting is made by turning the nicol until the two halves are equally bright when the gloss may be read at once from the divided circle. On the scale chosen white blotting paper reads about "20 degrees gloss," ordinary machine finish around 30 and high supercalendered about 40. The highest gloss on white paper runs about 50 degrees.

The instrument furnishes a ready means for the control of the supercalendering process and renders possible uniformity of product. Readings require only a few seconds of time and may be made by an almost inexperienced operator and in an ordinarily lighted room. Colored papers may also be tested if a suitably colored glass is placed in the eyepiece.

*b. Martins-Koenig Photometer.* The accompanying cut (Fig. 43) of the type of photometer used in connection with the determination of finish and translucency of paper is given because of the



FIG. 39. VALLEY SIZE TESTER  
(Valley Iron Works, Appleton, Wis.)

increasing interest in the use of these instruments. The scale, which is engraved on a platinoid circle, is divided into angular degrees and densities. It is understood that this type of photometers is to be produced in this country.

### 15. Volumetric Composition\* (3)

The determination of the volume composition of a paper is at best only an approximation but it is at times desirable to carry it

out. The weight of a cubic centimeter of the paper is first ascertained by calculation from the thickness of the sample and the weight of a measured area. The percentage by weight of the various materials present, fibers, clay, size, etc., is then determined in the usual way and from this the weight of each in a cubic centi-

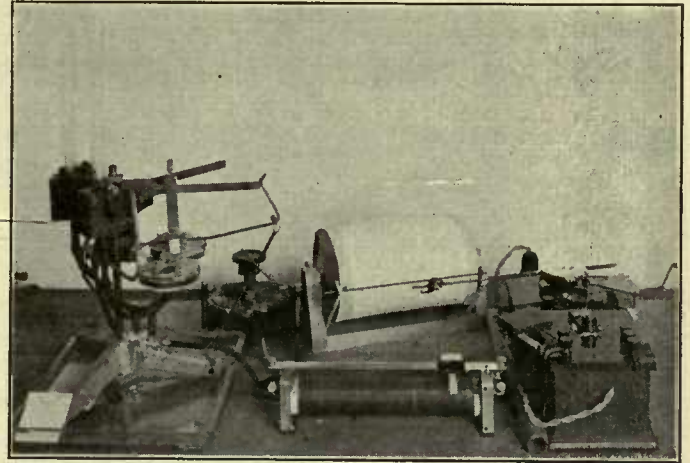


FIG. 40. SIZING TEST APPARATUS\* (112)

An elaborate set-up with recording drum for the study of the electrolytic method of determining the sizing quality of paper. (Bureau of Standards.)

meter of the paper is calculated. The weight of each substance in grams divided by its specific gravity gives the volume occupied by it, and the sum of all of these volumes subtracted from 1.0 gives the volume of air per cubic centimeter of paper. This method is fairly accurate when only fibers, clay and rosin are present but when other substances are added as in coated papers, the problem becomes more complex and the results less reliable.

If the volume of air per cubic centimeter of paper is the only information needed it may be obtained by determining the actual specific gravity by weighing in air and then in oil of known density exactly as in making specific gravity determinations in water. It will be found necessary to expose the paper, submerged in oil, to reduce pressure for some time in order to be sure that all air is removed and replaced by oil.

### 16. Retention of Loading

By retention of loading is meant that per cent of the entire amount of loading material added to the beater, that is retained in the finished product.

Secure about a 5-lb. sample of the filler to be used, being careful to select a representative sample. Break up all lumps, spread on a flat surface, divide into four parts, by dividing the pile by two lines at right angles to each other crossing at the center of the pile. Select two opposite quarters, mix and proceed as before. This is known as the "quartering method of sampling." This quartering method is continued until about 25 g. of loading material is obtained, which is then placed in a bottle for further use. From this bottle, remove a 1 g. sample, dry at 105° C., to constant weight and calculate per cent of moisture in the loading material. Place the dried residue in a crucible and heat at the full heat of a meker burner until a constant weight is secured, then calculate the per cent of water of composition in the dry clay.

(Have clay in a finely divided state and stir frequently during burning.)

Secure sample of pulps to be used and determine per cent of moisture and per cent of ash. Weigh the pulp added to the beater. Weigh the clay added to the beater. After running the paper over the paper machine, secure several pieces as a representative sample, dry and make the ash determination on the paper. The above

mentioned data used in the following formula will give the per cent of clay used and the per cent retention.

Let P = weight of pulp added (in lb.)  
 C = weight of clay added (in lb.)  
 A = Per cent ash in the finished paper.  
 Ap = Per cent ash in the pulp.  
 Wc = Per cent water of composition in the clay.  
 Mp = Per cent moisture in the pulp.  
 Mc = Per cent moisture in the clay.

$$(1) \% \text{ of clay used} = \frac{100 \times C}{P}$$

$$(2) \% \text{ retention} = \frac{100 A \times P}{C (100 - A)}$$

$$(3) \% \text{ of clay used} = \frac{100 C (1 - Mc)}{P (1 - Mp)}$$

$$(4) \% \text{ retention} = \frac{100 P \times (A - K)}{C (100 - A - K)}$$

The value of K is the per cent of filler not derived from the loading added. An average value of K is 0.50 so that the formula (4) may be used as above or as follows:

$$(5) \% \text{ retention} = \frac{100 P (A - 0.5)}{C (100 - A - 0.5)}$$

Formulas (3) and (5) are recommended for use by the Technical Association of the Pulp and Paper Industry, though (1) and (2) may be used when accuracy is not essential or when the values for moisture content are unknown. Formula (4) does not take into consideration the per cent water of composition in the loading. Where this is known suitable correction may be made.

No account is taken of the ash from alum or rosin size as the maximum amount from these factors is probably under 0.05 per cent and therefore negligible. An ash determination need not be

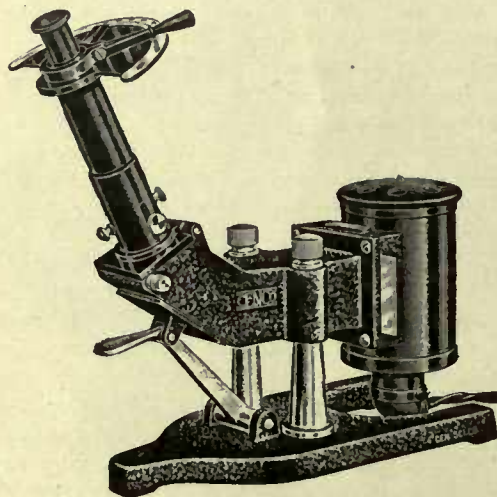


FIG. 41. GLARIMETER

A device developed by L. R. Ingersoll for the purpose of measuring the finish or gloss of paper. (Central Scientific Company, Chicago.)

calculated beyond the first decimal place. (See ash determination under chemical testing.)

An alternate retention formula, developed in the laboratory of the S. D. Warren Company, Cumberland Mills, Maine, is suggested as being of value, as it may be used without making tests that interfere with production of paper.

A = Per cent of ash in air-dry stock going to machine.  
 B = Per cent of ash in air-dry paper at reel.  
 C = Per cent of bone dry filler lost on ignition.

$$\text{Retention} = \frac{0.94 B (100 - 100 C - A)}{A (100 - 100 C - B)}$$

A and B are considered as whole numbers and C as a decimal.

## 17. Conducting Particles

To show the presence of conducting particles in paper 0.5 or 0.75 mils thick, the sample is placed upon a metal plate which has been polished to a smooth plane surface. This plate is connected in series with 3 dry cells, a model 280 Weston voltmeter of 3-volt range or a similar instrument, and a metal piece which has a perfectly flat under surface and will be in contact with all parts of the plate upon which it rests. This metal piece is about 1 in. long and 1/2 in. wide and is attached to a handle for convenience in using.

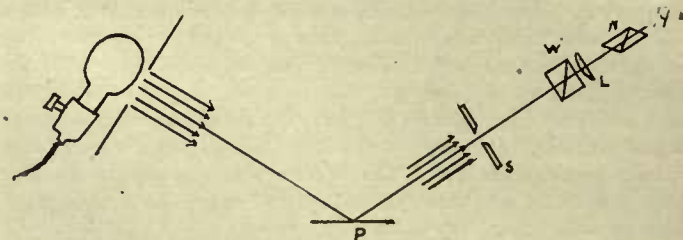


FIG. 42. GLARIMETER PRINCIPLE

Cut showing the arrangement of lenses and the principle of the Ingersoll glarimeter. (L. R. Ingersoll.)

It is called the detector. To test paper, place a measured area upon the plate, make contact with the metal detector and the plate and if there is deflection of the voltmeter indicating that the voltmeter will show any drop in potential that occur when a conductor is between the plate and the movable detector, the instrument is ready to use. Pass the detector slowly over the paper on the plate, using light pressure. When a deflection of the voltmeter indicates that there is a conducting particle in the sheet between the detector and the plate, the position of the detector is marked and it is then moved over the spot at right angles to its former position and the paper marked and it is then moved over the spot at right angles to its former position and the paper marked again when deflection occurs. This locates the particles within a half-inch square and makes it available for microscopic study. Results are expressed in terms of number of conducting particles present per sq. ft. of paper. This instrument is intended for papers of 0.75 mil thickness or less. With thicker papers, the particles cannot be registered with dependable accuracy because they seldom extend through the full thickness of the sheet. Comparison of iron particles present as shown by chemical tests give numbers far in excess of the number of iron particles that are actual conductors in the sense of spoiling the paper for electrical purposes. This instrument is intended for use in testing papers specified for use in electrical equipment.

An additional method is indicated by the accompanying photograph (Fig. 44). The small metal piece in the foreground is used as a detector and the presence of a conducting particle is indicated by a click in the telephone receivers.

## 18. Resistance to Water Penetration

Various simple methods have been proposed for this purpose and they are included as they may be of some assistance.

A quart mason jar is used in the test. A circular hole of 1 in. in diameter is cut in the metal top and the bottom of the jar is broken out. The sample to be tested is placed in the metal top, between the rubber washer and the metal and firmly screwed in place. The jar may either be filled with water or may be reversed and partially immersed in water. The temperature of the water should be 75° F. The length of time for penetration of the water will indicate a relative resistance.

As in the above case, a circular hole of 1 in. in diameter is cut in the metal top, the sample placed in the top between the rubber washer and the metal, a bone-dry weighed sample of absorbent cotton or paper is suspended in the jar, the lid put on and the jar reversed and partially immersed in water at 70° F. After a prede-

terminated length of time, the absorbent cotton or paper is removed and immediately weighed. The increase in weight will indicate a relative value for moisture penetration.

A modification of the Stöckigt method may also be used as a means of determining the relative water resisting quality of paper. For routine tests, the samples may conveniently be cut about 2½ in. square and molded into a cup-shape in the top of a bottle by depressing the paper into the top of the bottle with the stopper. The top of the bottle should be about 1½ in. inside diameter. A ground glass bottle and stopper are best suited to the purpose. At least three test "cups" should be prepared from each sample to be tested and the average result taken as an indication of the degree of water-proofing. A number of samples may be tested at the same time by using a wide flat-bottom pan or dish. Enough of the 2 per cent ammonium thiocyanate ( $\text{NH}_4\text{CNS}$ ) solution to cover the bottom of the dish and float the "cups" is used. After the "cups" have been floated and the time recorded, place three or four drops of the 1 per cent ferric chloride ( $\text{FeCl}_3$ ) solution in the middle of each cup as quickly as possible, taking care not to drop any of it into the other solution. A large dropper is convenient to use. Spread the ferric chloride out over the bottom of each cup with a glass rod, taking care not to spread it out far enough to touch the bend. For where the paper is bent or folded, the solution will penetrate more rapidly and give erroneous results. The layer of ferric chloride solution should be fairly thin because it has a reddish color and may interfere with one's judgment of the reaction if it is too thick. The time required for the pink or red coloration to set in is taken as a measure of the degree of water-proofing. On samples it will be found that only a single point of color is seen. This is probably due to a "pin hole" or fault and should be noted, but not taken as the "end point." The color should be fairly general and pronounced before the solution is considered through.

Note—Fritz Stöckigt, *Wochenblatt für Papierfabrikation*, 1920, pg. 39; translation Paper, March 10, 1920.

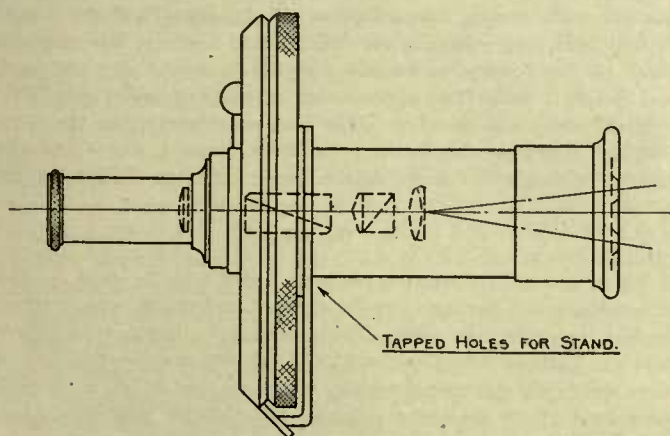


FIG. 43. MARTINS-KOENIG PHOTOMETER

A sketch of a photometer used for Ingersoll glarimeter. (Courtesy of Adam Hilger, Limited, London.)

#### IV. CHEMICAL ANALYSIS

##### 1. Ash Determination

(a) *Quantitative*—A 1 g. sample of the paper to be tested is burned in a porcelain or nickel crucible. A Meker burner is very convenient for this purpose, as some heavily loaded papers require considerable time and heat to burn the last traces of carbon. Ordinarily a white paper will give a white ash, but if mineral pigments have been used the ash is likely to be colored. In any case the ash should be free from specks of unburned carbon.

During the burning care must be taken that a portion of the ash

is not lost by air currents. The ash is often light and fluffy, and the strong currents of air from the burners may blow away a por-

Note—The sample of paper need not be weighed closer than 0.005 g., since a 1 per cent variation in the moisture content will introduce an error of 0.01 g. If the maximum error in the weight of the paper is 0.01 g. then the maximum error in the weight of the ash will be 0.01 g. for every 10 per cent of ash present. Therefore in a paper containing 10 per cent ash, the results will be reported to the nearest tenth. If special accuracy is required, the paper may be weighed in the bone dry condition. Then with the error due to moisture eliminated it is possible to weigh the paper to  $\pm 0.0005$  g. and the error will be 0.0001 g. for every 10 per cent of ash. The results may then be reported to the nearest hundredth. This latter result will of course be 1 per cent lower than the ash results on a 1 g. sample containing 10 per cent of moisture.

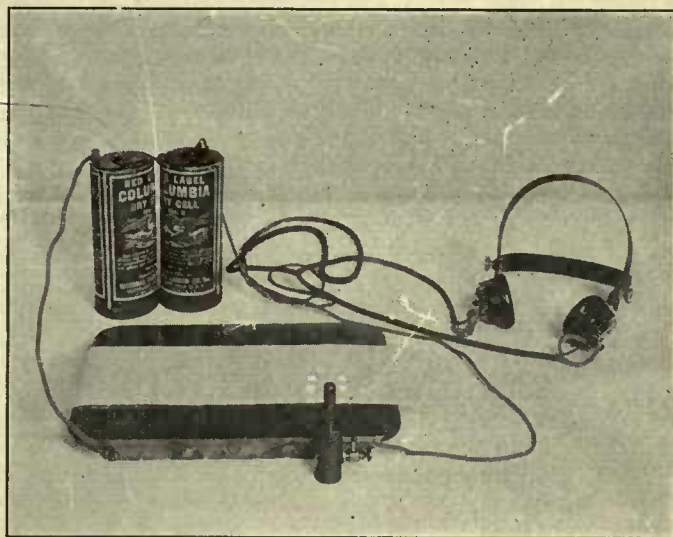


FIG. 44. CONDUCTING PARTICLES

An arrangement for easily determining the presence of conducting particles in thin paper, such as condenser paper. (Pittsfield Works Laboratory, General Electric Company.) (Bureau of Standards.)

tion of it. While cooling they may be kept in a dessicator, but this is not necessary, since the ash may be poured into a counterpoised aluminum pan as soon as the crucible is cool enough to avoid the danger of loss from convection currents. The ash will cool almost instantly and may be weighed at once. This saves the time required for the crucible to cool and also avoids the necessity of weighing the crucible.

Note—Aluminum is recommended as being less easily broken as well as lighter, than glass.

The ash as finally obtained includes all non-volatile and non-combustible matter in the paper. It may be derived from at least five sources:

(1)—The ash of the pulp from which the paper was made; (2) the ash from the various loading or filling materials added; (3) the ash from any surface coating or sizing, and (4) the ash of mineral coloring materials or pigments, and (5) the ash derived from alum used, though the amount traceable to this cause is very small and may be neglected. The complete quantitative analysis of an ash is a time-consuming and also a rather complicated process. It is possible, however, to obtain some idea of the composition of the ash by a few comparatively simple tests.

Once the paper is burned it is impossible to tell which portion of the ash is derived from the coating and which portion is derived from the filler. Therefore, if anything more than the total ash content is desired the coating must be stripped from the paper before ashing. In the case of coated papers where casein has been used as the adhesive, this can often be done by the use of dilute ammonia. The insoluble material may be filtered off, dried and weighed. The filtrate may be evaporated to dryness and the residue weighed. This will include the casein (or soluble caseinates if such be present) as well as any soluble material present. The difference between the weight of the total ash and the ash of the

paper from which the coating has been stripped plus the weight of the coating will give the weight of the combustible portion (i. e. glucose or casein) of the coating.

Note—Provided the insoluble portion of the coating has been ignited to the same extent as the total ash.

It is quite possible for a paper to have an ash of 3 to 5 per cent without being loaded. This might be due to the ash in the pulp, as well as to the ash derived from water color, alum and sizing materials.

Where the ash is 5 to 20 per cent the paper is loaded. A list published in *Paper* \*(70) gives the names of twenty-one loading materials. However, from the chemical standpoint many of these are practically the same material sold under different names. They are all silicates, sulphates or carbonates of aluminum, magnesium, barium or calcium. While an analysis will give the composition of the ash, it will not tell under what trade name the material may have been bought.

In this connection it is interesting to note the following percentages of ash in fibrous raw materials as given by Wrede. (*Paper*, Jan. 31, 1912)\* (3).

Stock	Ash %
Bleached linen half stuff.....	0.12—1.86
Bleached cotton half stuff.....	0.24—0.79
Unbleached cotton half stuff.....	0.24—1.12
Sulphite, unbleached.....	0.48—1.25
Soda.....	0.36—1.40
Adansonia.....	5.70—7.19
Japanese fibers.....	2.5

(b) *Qualitative* \*(4, 11, 69).—To determine the kind of loading or coating material used, it is necessary to test the ash qualitatively, for which purpose at least 0.2 g. of ash is desirable. Briefly, tests should be made for the substances indicated in table, in which are also given the fillers that the presence of these substances would indicate.

#### PAPER FILLERS AND THEIR INDICATORS \*(91)

Substance	Filler indicated
Calcium sulphate.....	Crown filler
Calcium carbonate.....	Chalk
Barium sulphate.....	Blanc fixe
Magnesium silicate.....	Talc
Aluminum silicate.....	China clay

These fillers have various trade names and do not in all cases have definite chemical formulas, but the presence of any great amount of any of the materials in the first column would indicate the kind of filler used, and further confirmatory tests may be made.

Burn enough paper to obtain at least 0.2 g. ash in a platinum or nickel crucible. Separate 1/3 of the ash from the main portion; to this 1/3 add 5 cc. water and boil until well extracted; filter; add a drop of hydrochloric acid to the filtrate and then 3 cc. 10 per cent barium chloride solution. A white precipitate is due to *calcium sulphate* or *crown filler* in the paper. To the residue from the water extraction add dilute hydrochloric acid. Effervescence of carbon dioxide gas is due to *chalk* in the paper. This test for chalk may be applied directly to the paper before ignition if the presence of chalk is suspected at the start.

To the 2/3 portion of the ash add 1 g. sodium carbonate and mix well. Fuse the mass in a platinum crucible until it becomes a clear quiet liquid. Cool and dissolve in boiling dilute hydrochloric acid. This solution should be clear. If an undissolved white residue remains, filter this off. It is probably due to barium. Dip a clean platinum wire in this residue and hold it in a bunsen flame. Barium will give a characteristic green color. This shows the presence of *blanc fixe*.

If the previous hydrochloric acid solution was clear evaporate nearly to dryness. Dip a clean platinum wire in this mass and test for *barium* as given above. Then take up the mass with dilute hydrochloric acid; boil; filter. The residue is *silica* from silicates in the filter. A portion of this filtrate can be used as a confirmatory test for *sulphates*. To the filtrate from the silica separation add ammonium hydroxide until slightly alkaline. A white floccu-

lent precipitate shows the presence of *aluminum*. Filter off this precipitate and make the filtrate acid with oxalic acid. Make alkaline slowly with ammonium hydroxide. The formation of a white precipitate shows the presence of *calcium*. Filter off this precipitate and make the filtrate alkaline with ammonium hydroxide. Add 5 cc. saturated solution of sodium acid phosphate and stir with a rod. There will be a crystalline precipitate formed if *magnesium* is present. It forms slowly and is best brought down by an occasional rubbing of the sides of the beaker with a stirring rod.

These tests indicate the possible combinations of elements in the filler. Where there are several names for one chemical combination—such as *talc*, *asbestine*, *agalite*, etc.—for various magnesium silicates a microscopic analysis and comparison of the crystal form with known samples is necessary. Quantities of aluminum invariably indicate *clay*. Silica and magnesium indicate *talcs*, *agalites* or *asbestine* and water-soluble sulphates from filler point to *calcium sulphate*.

\*(12).—If the paper contains *calcium sulphate*, the ash obtained may consist partly of calcium sulphide, due to reducing action of the carbon found on ignition, and the amount will, therefore, not represent the true amount added. The ash should be moistened with a few drops of sulphuric acid, and again ignited, in order to reconvert it into calcium sulphate. It should also be borne in mind that the sulphate of lime as present in the paper is combined with two atoms of water ( $\text{CaSO}_4 + 2\text{H}_2\text{O}$ ), and, therefore, that every part of calcium sulphate obtained represents 1.26 parts of *pearl-hardening* actually in the paper.

(c) *Amount of Coating*. \*(11).—Weigh a piece of the paper cut exactly 2 x 5 in. and place in a flat glass dish. The dishes used for developing in photography are convenient for this purpose. Cover with water containing 1 per cent of  $\text{NH}_4\text{OH}$  and set aside in a warm place (2 or 3 hrs. is generally sufficient to loosen the coating). Remove the paper to a large watch glass, rub the surface with a small camel's hair brush cut off square, and wash the coating into a beaker. If the paper is double-coated, turn it over and repeat on the other side. Continue the operation until all the coating is washed into the beaker. Dry the paper and weigh it under the same conditions as those under which the original paper was weighed. The loss in weight is the weight of coating. Calculate this to per cent of the original sample and also figure the weight of coating on the basis of a ream of 25 x 40, 500.

#### 2. Paraffin

There are several paraffin solvents which may be used for this determination. Gasoline is easily obtained and comparatively cheap. It has, however, the serious disadvantage of being very inflammable. Carbon tetrachloride ( $\text{CCl}_4$ ) is not combustible. It is superior to chloroform, since the fumes are not likely to produce anesthesia. Both gasoline and carbon tetrachloride have been found satisfactory.

Note—Carbon tetrachloride cannot be kept in ordinary "tin" cans on account of its action on iron.

Enough of the paper must be taken to obtain a weighable amount of paraffin. One or 2 g. of paper should be sufficient.

Place the paper in a soxhlet or in an ordinary erlenmeyer flask fitted with a reflux condenser, cover with gasoline or carbon tetrachloride and extract until the paraffin is all dissolved. If the erlenmeyer flask be used it will probably be necessary to make a second extraction with a fresh amount of solvent.

The solution may then be evaporated to dryness and the paraffin weighed. If the paraffin shows a tendency to "creep" over the edge of the dish it may be easier to weigh the paper before and after extraction and consider the loss in weight as *paraffin*.

The following qualitative test for paraffin known as the *Dunlop*

method may be of value for determining the presence of *paraffin* in the presence of rosin:

It consists in boiling the sample with acetic anhydride and observing the behavior of the solution on cooling. If *paraffin* is present the anhydride becomes turbid and the *paraffin* separates out on the top in a white precipitate. Less than 1 per cent of *paraffin* may be detected in this manner. (*Allen's Commercial Organic Analysis*.)

### 3. Sizing Materials

a. *Rosin*.—*Sammet Method*; *Alcohol-ether Method*. \*(103).

Cut 5 g. of paper into strips approximately  $\frac{1}{2}$  in. wide and fold them into numerous small crosswise folds. Place the folded strips in a soxhlet extractor and fill with acidulated alcohol. Acidulated alcohol solution is made by adding 900 cc. of 95 per cent alcohol to 95 cc. of distilled water and 5 cc. of glacial acetic acid. Place the soxhlet flask directly in the boiling water of a steam bath and extract by siphoning from six to twelve times, according to the nature of the paper. Wash the alcoholic extract of rosin, which may contain foreign material, into a beaker and evaporate to a few cc. on a steam bath. Cool, take up in about 25 cc. of ether, transfer to a 300 cc. separatory funnel containing about 150 cc. of distilled water to which has been added a small quantity of sodium chloride to prevent emulsification, shake thoroughly and allow to separate. Draw off the water into a second separatory funnel and repeat the treatment with a fresh 25 cc. portion of ether. Combine the ether extracts which contain the rosin and any other ether-soluble material and wash twice or until the ether layer is perfectly clear and the line between the ether and the water is sharp and distinct, with 100 cc. portions of distilled water to remove salts and foreign matter. Should glue which is extracted from the paper by alcohol interfere by emulsifying with the ether, it may be readily removed by adding a strong solution of sodium chloride to the combined ether extracts, shaking thoroughly and drawing it off, repeating if necessary before washing with distilled water. Transfer the washed ether extract to a weighed platinum dish, evaporate to dryness and dry in a water oven at from 98 to 100°C. for exactly one hour, cool and weigh. This length of time is sufficient to insure complete drying. Prolonged heating causes a continual loss of rosin.

Some objections have been made to portions of the foregoing method. It has been stated that the sodium chloride is sufficiently soluble in the ether to produce high results. Some also prefer to carry the evaporation of the alcohol extract to complete dryness and then take up in ether and in water. The residue as obtained is only partially soluble in ether, but in case the entire amount of ether-soluble material should not be secured, after as much has been dissolved by the ether as possible, the remainder of the residue is taken up in water. The ether and water is then separated in a separatory funnel in the usual manner. There appears to be no reason why a glass dish should not be as satisfactory as a platinum dish. It is also asserted that the extraction may be carried out in an erlenmeyer flask instead of a soxhlet. The number of extractions required depend upon the character of the paper used. In some individual cases it has been found that a single extraction took out practically all the rosin. This extraction was done on a hot plate and the alcohol was in contact with the paper for about half an hour. It is not known to what extent this time could be shortened or in what per cent of cases a single extraction would be sufficiently accurate.

Note—for extracting rosin, the apparatus shown in Fig. 43 will do the work of a soxhlet extractor with greater convenience. It is essentially the same as the soxhlet in principle, but can be set up very quickly, takes less solvent, keeps the condensed solvent surrounded by hot vapors, occupies less space, and is less liable to breakage. The time of extraction is lessened because of more frequent flushing of the small well with the condensed solvent. This apparatus is listed as an *Underwriter's Extractor*, and has been extensively used in the extraction of rubber.

#### QUALITATIVE TEST FOR ROSIN.

Boil a small portion of the paper in 5 cc. acetic anhydride in a

dry test tube. Cool. Add carefully down the side of the test tube a small amount of concentrated sulphuric acid. The development of a pink ring shows the presence of *rosin*.

*Rosin* \*(3) is used almost exclusively in the beater to impart waterproof properties to the paper. There is no single test of a simple nature which will demonstrate positively the presence or absence of rosin and any judgment regarding it must be based on the indications of a number of different tests. If a little ether is dropped onto a sheet of paper and allowed to evaporate there will be formed, in the case of rosin-sized paper, a ring of rosin at the edge of the zone where the ether evaporated. This will be absent in most unsized papers, and it will, of course, be formed in any paper which contains any ether soluble material besides rosin.

Another test is made by boiling a little of the paper for a few minutes in glacial acetic acid and pouring the acid into a little

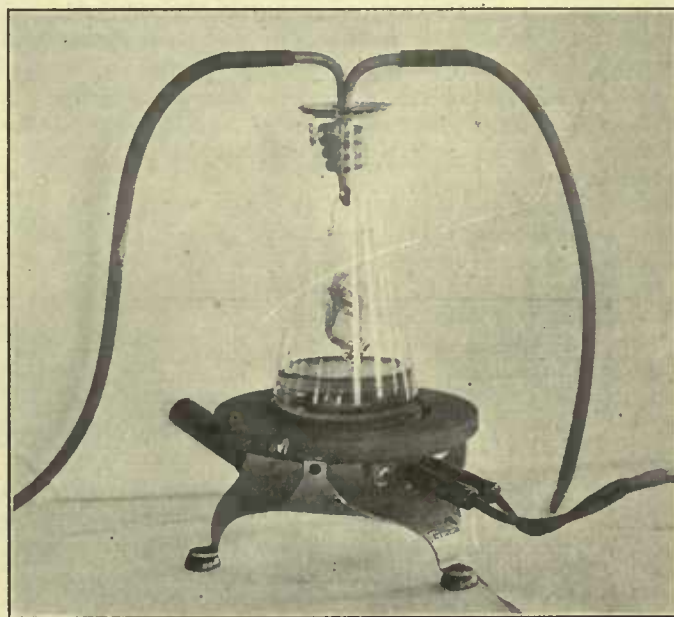


FIG. 45. ROSIN EXTRACTION

A simple apparatus for determining the rosin content of paper (*American Writing Paper Company, Holyoke, Mass.*)

distilled water. A pronounced turbidity indicates rosin, but a slight opalescence may be caused by other soluble substances and must be disregarded.

A third test is that known as the *Raspail reaction*. If a drop of concentrated sulphuric acid be placed on the paper and a grain or two of sugar added a pronounced *raspberry red color* will develop with rosin-sized papers, while with unsized papers red color is also formed when albuminous materials are present so they must first be proved absent before the test can be considered indicative of rosin.

(b) *Glue and Casein*.—There appears to be no quantitative method known for the determination of these materials in the presence of each other. Both substances contain nitrogen. If only one be present and the nitrogen content of the original material as added to the paper be known, then by means of the nitrogen determination the content of glue or casein may be determined.

#### QUALITATIVE TEST FOR GLUE.

Boil a small portion of the paper with 10 cc. of water in a test tube. Decant the extract to another test tube and cool. Then add 5 cc. of ammonium molybdate solution, followed by a few drops of nitric acid. The formation of a white amorphous precipitate shows presence of *glue*.

## DETERMINATION OF NITROGEN

Place from 3 to 5 g. of the paper which has been cut into small pieces in a kjeldahl digestion flask, add ten g. potassium sulphate, 0.7 g. of mercury and 25 cc. of concentrated sulphuric acid.

The mercury acts as a catalytic agent aiding in the decomposition of the nitrogenous material. The potassium sulphate serves to raise the boiling point of the sulphuric acid. It is probable that sodium sulphate can be used in place of potassium sulphate, but it is recommended that 15 g. of sodium sulphate crystals be used in this case.

Heat gently at first to avoid frothing and finally increase the heat as the digestion proceeds. At the finish the solution should be colorless, or of a pale straw color, and of a syrupy consistence. At the completion of the digestion, which may require one and a half to two hours, the contents of the flask are allowed to cool and 30 cc. of a 4 per cent solution of potassium sulphate are added.

The potassium sulphate is necessary to break up nitrogen compounds of mercury. Other materials than potassium sulphide have been used for this purpose, but are not recommended.

Before the distillation can be made the mass must be rendered alkaline. First dilute with about 200 cc. of distilled water and then neutralize by adding an excess of saturated solution of sodium hydroxide.

The volume of the solution after the sodium hydroxide has been added should be about 400 cc., therefore the volume of water added must be calculated so that just enough room would be left for the sodium hydroxide solution. Commercial sodium hydroxide (95 per cent) has been found satisfactory.

There should be an excess of caustic soda equal to about 5 cc. of a saturated solution. It is convenient to add a few drops of methyl orange indicator or phenolphthalein indicator solution to the flask before adding the sodium hydroxide. The solution will become yellow or red respectively when it becomes alkaline.

The sodium hydroxide solution is carefully poured down the side of the flask so that it does not mix with the contents. The flask is immediately connected to the condenser and then the flask is shaken in order to thoroughly mix the contents.

If about 5 g. of granulated zinc or a few small pieces of pumice stone are added to this flask just before the sodium hydroxide, they will help to prevent bumping.

The distillate is caught in a flask containing a known amount of standard acid diluted to a volume of 100 cc. with distilled water. (The equivalent of 30 cc. *n*/10 normal acid should be ample.) A few drops of indicator should be added to this solution. Sodium alizarin sulphate and methyl red have been recommended as indicators. The end of the condenser tube should dip beneath the surface of the acid. The distillation should continue for 45 min. and the distillate should equal 200 cc. Titrate with *n*/10 normal alkali.

This same operation of distillation should be carried out, using only the chemicals involved in order to have a check on their purity. This is known as the "blank."

Subtract the number of cc. of *n*/10 normal alkali required to neutralize the distillate, from the number of cc. required by the blank. This difference is the number cc. of *n*/10 normal alkali equivalent to ammonia.

$$\text{No. cc.} \times 0.014 = \text{g. nitrogen.}$$

The following factors should be used on unknown samples: For *casein* use the factor 6.3 and for *glue* use the factor 5.6. In all cases this factor should be determined wherever possible, as those values will vary, depending on the grade of material used.

Note—Copper sulphate, weight for weight, can be substituted for the mercury as a catalytic agent in this determination; it serves as an indicator for alkalinity by turning a characteristic blue when the solution is made alkaline previous to distillation. Small glass beads can be effectively substituted for granulated zinc to prevent bumping in the distilling flask.

*Casein* \*(3) may be detected in paper by moistening the sample with Millon's reagent and warming gently either over a flame or over an open steam bath. If casein is present a brick-red color will develop. In the case of coated paper in which much satin white is used, the alkali present determines the formation of a yellow color. In this case proof may be obtained by moistening the paper first with dilute nitric acid, to neutralize the alkali, and then applying the Millon's reagent as before; tested in this way satin white coated paper will give the usual red color. Casein may also be detected by boiling the paper with water and a few drops of ammonia, filtering and adding to the filtrate dilute acetic acid very gradually. Casein will precipitate when the solution becomes very faintly acid, but it may redissolve on adding a considerable excess. This test is also given, though usually less strongly, by rosin, so the precipitate should be tested with Millon's reagent to confirm the presence of casein. Casein is seldom used except in the coating; cases of surface sizing or of its use in the beaters are very rare.

*Glue* \*(3) is sometimes used as an adhesive in coating papers and in rare instances in the beaters; the better grades known as gelatines are used in surface sizing. If glue is present alone it may be detected by boiling the sample of paper in water, filtering if necessary, and adding a little dilute tannic acid solution; a grayish, flocculent precipitate indicates glue. Casein is also precipitated by tannic acid and the presence of starch prevents the precipitation of glue so that when either casein or starch is present there is apparently no means of proving the presence or absence of glue.

*c. Starch: Procedure for Analysis.*—The paper to be analyzed is tested with the usual iodine reagent. If but a trace of starch is present, no acetic acid is required in extraction. A 5-g. sample is cut into small pieces and placed in a 500-cc. round-bottom flask. 200 cc. of water is added, and 5 cc. glacial acetic acid is run in, making a 2½ per cent solution. The flask is connected with a reflux condenser by means of a clean rubber stopper and the contents boiled vigorously for 1½ hrs. The extract is decanted through a Büchner funnel equipped for suction filtration and the pulp washed with about 50 cc. of hot water. To the filtrate is added 15 cc. of HCl (37 per cent) and boiling continued for 30 min., the volume of the solution being permitted to decrease by evaporation to about 200 cc. The hot acid solution is neutralized by the addition of solid sodium carbonate until effervescence ceases and the volume is determined. This solution is titrated into a measured quantity of Fehling's solution (2 to 10 cc., according to the amount of starch present). After each addition of sugar solution the mixture is heated to the boiling-point and maintained at that temperature for 1 min. The reaction mixture may be diluted if this is considered desirable. The end-point is determined on a spot plate with a potassium ferrocyanide-acetic acid solution and is that point at which no immediate color is produced on the plate; it may be determined to within ½ to 1 cc. of sugar solution, depending on the volume of solution employed. It was found that the potassium ferrocyanide became colored when allowed to remain a number of days with the acetic acid, and that a sharper and more distinct end-point can be obtained if the acid is added separately to the spot plate when the test is to be made. One drop of each solution is used for a test.

## QUANTITATIVE ANALYSIS FOR STARCH\* (120)

*Method of Kamm and Voorhees.*

PREPARATION OF REAGENTS—The usual Fehling's solution is employed.

Solution A—69.3 g. of crystallized copper sulphate are dissolved in water and the solution diluted to 1,000 cc.  
Solution B—346 g. of Rochelle salt and 120 g. of sodium hydroxide are dissolved in water and the solution also diluted to 1,000 cc.

Solutions A and B are kept separate and equal volumes mixed when ready to be used. In a given experiment, where it is reported

that 10 cc. of Fehling's solution is used, it is understood that 5 cc. of solution A is added to 5 cc. of solution B. According to the literature, 10 cc. of such a solution should be equivalent to 0.05 grams of dextrose when an analysis is run in a specified empirical manner. It is found more convenient to standardize the solution with a known quantity of starch, the latter being hydrolyzed and titrated under the same conditions used later for the hydrolysis and titration of starch in paper. The advantage is obvious.

*Potassium ferrocyanide solution.* A 10 per cent solution of  $K_4Fe(CN)_6 \cdot 3H_2O$  is used.

*Acetic acid solution.* A 50 per cent solution of acetic acid is found convenient.

#### METHOD OF CALCULATION OF RESULTS

It has already been suggested that Fehling's solution be standardized against one of the ordinary starches used in paper manufacture. Such a procedure is justified by the close agreement in the reducing values of corn-starch, Hercules gum, feculose and dextrin.

*Example*—A sample of corn-starch was dried at 105° C. for 3 hrs. A .05-g. portion was then weighed out and hydrolyzed with about 190 cc. of a 4 per cent HCl solution during a period of 30 min. After neutralization with solid sodium carbonate, the final volume was adjusted to 200 cc., and the solution titrated against 10 cc. of Fehling's solution; 20 cc. of sugar solution were required and 10 cc. of Fehling's solution are therefore equivalent to  $20/200 \times 0.50 = 0.050$  g. starch.

In an analysis of a 5-g. sample of paper the volume of the final hydrolysis mixture was 217 cc. Of the latter solution 39 cc. were required for reaction with 10 cc. of Fehling's solution. The per cent of starch in the sample of paper is therefore:

$$\frac{217}{39} \times \frac{\text{Value of Fehling's solution in g. of starch} \times 100}{\text{Wt. of sample of paper}} = 5.5 \text{ per cent}$$

Since, however, a 5-g. sample of paper is used, and since our Fehling's solution is equivalent to 0.05 g. starch to 10 cc. of solution, the calculation is simplified thus:

$$\frac{217}{39} = 5.5 \text{ per cent starch.}$$

Mention might be made of the polarimetric method of Dr. C. E. G. Porst and H. A. Crown. See *Journal of Industrial and Engineering Chemistry*, vol. 5, No. 4, April, 1913.

#### QUALITATIVE TEST TO INDICATE ITS PRESENCE IN PAPER

Make a dilute solution of iodine in potassium iodide by adding a small amount of water to a mixture of three or four crystals of iodine and 1 g. of potassium iodide, stirring until the iodine is completely dissolved, and then diluting the solution with pure water until a pale straw-yellow color is obtained. Add a drop of this solution to the paper under examination, a blue color indicates the probable presence of starch. If this blue coloration is obtained it is well to confirm the test by boiling the paper with water and testing the water extract with the iodine solution, because cellulose in the presence of water when subjected to certain mechanical processes gives rise to modifications known as hydrocelluloses. These hydrocelluloses are not soluble to any great extent in boiling water, but they will give rise to a blue coloration when brought into direct contact with the iodine solution.

*An alternate procedure is as follows:* The universal test for starch \*(3) is to apply a dilute iodine solution to the paper when a blue to violet color will appear if starch is present. It is well to confirm this test by boiling some of the paper with a little water, filtering and testing the filtrate, after cooling, with a few drops of iodine solution. This is necessary because hydrocelluloses, which are only slightly soluble in boiling water, also give a blue color when brought into direct contact with iodine solution. Microscopic examination will show whether the starch granules have

been burst by boiling or whether the starch was used without cooking. If the paper to be tested is torn so that it splits on the edge before being moistened with the iodine solution it is generally possible to tell whether it is surface sized or not. If it is surface sized only, the interior of the sheet will remain white while the surface will turn blue; if, however, considerable starch was used in the beater, this is in part cooked and drawn to the surface by the heat of the driers so that the paper has the appearance of being surface sized when in reality it was not. Microscopic examination of the papers after treating with iodine will sometimes enable an opinion to be formed though it is seldom possible to prove positively in such a case whether the paper is surface sized or not.

*d. Dextrine in Presence of Beater Starch. Method of Kamm and Tendick.* \*(119).—The procedure adopted consists in the removal of the surface sizing by a 45-min. leaching of the sample of paper with water at a temperature of 60° C. For a 5-g. sample 200 cc. of water is used. The extra is removed by suction filtration and the soluble carbohydrate material hydrolyzed and estimated according to the procedure already described in detail. See *Method for quantitative determination of starch*. The starch remaining in the paper may then be isolated by the dilute acetic acid extraction method recommended in the article on starch determination.

#### 4. Chlorine

The determination of free chlorine in paper is carried on in a manner similar to that used in testing half-stuff; namely, take a small mass of the stuff to be tested, from the beater, press it with the hand and test with a few drops of potassium iodide starch solution. If free chlorine is present the characteristic blue color will be developed.

For the testing of finished paper the determination is best carried out as follows. Cut the paper into small pieces, moisten with distilled water, and test with starch iodide paper; this is best done on a glass plate.

Instead of starch iodide paper one may mix a small piece of starch to a paste with cold water, and mix it with a solution of potassium iodide.

#### 5. Sulphur \*(127)

The apparatus consists of a 500 cc. round bottom flask with a neck about 2 in. long and 1 in. in diameter. The mouth of this neck is ground to a flat surface and on this is placed a glass tube about 4 in. long and 1 in. in diameter, the lower end of which is also ground flat to fit tightly upon the upper surface of the neck of the flask. The whole is so arranged that after placing a piece of filter paper between the two ground surfaces, the tube and flask can be securely clamped together so that all gas generated in the flask must pass through the filter paper and then up through the superimposed glass tube.

The procedure for the testing of tissue papers is as follows: A sample of 25 sq. in. is taken and its weight determined. It is then shaken up in a wide mouth, glass-stoppered bottle with 10 cc. of distilled water; when partial disintegration has taken place, another 10 cc. of water is added and the shaking continued until the paper has been completely reduced to pulp. The larger part of the pulped mass is now transferred to the flask described above, and the residue which is left in the bottle is rinsed into the flask with a mixture of 10 cc. of water.

Prepare turnings from the highest grade, pure stick zinc, which must be free from sulphur and arsenic. Treat 1 g. of these turnings with 10 cc. of a dilute solution of copper sulphate containing about 0.002 g. actual copper. After a few minutes all the copper will have deposited and the turnings are then thoroughly washed to remove every trace of zinc sulphate.

The turnings are added to the flask and a wad of cotton inserted in its neck. Between the two ground glass surfaces is then clamped a piece of filter paper about 2 in. square which has been perforated with small pin holes about  $\frac{1}{8}$  in. apart and which just

before use is moistened with several drops of lead acetate solution. Finally a loose wad of cotton is placed in the tube above the paper.

The flask is placed on the steam bath and allowed to stay, with occasional shakings, for an hour. The filter paper is then removed from the neck of the flask and air dried. It is best compared with the standard test pieces by placing them side by side on a piece of white paper and covering them with a thin piece of clear, white glass. The standard test pieces are prepared by using sulphur-free cotton in the flask instead of the disintegrated paper and adding to this definite volumes of a very weak solution of sodium thiosulphate whose strength is accurately known. The sulphur-free cotton is prepared by boiling absorbent cotton in weak caustic soda solution and washing thoroughly with distilled water.

The sensitiveness of this test is such that the presence of 0.000001 g. of sulphur in the flask will give a distinct color on the lead acetate paper. From tests of a considerable number of papers which have been found satisfactory in actual practice it has been proved that tissue paper is safe for wrapping silverware if it does not contain more than 0.000002 g. of sulphur per 25 sq. in. of paper (about 0.25 g.)\*

#### 6. Coloring Matter \*(28)

*Smalts*, existing as it does in high-class papers, usually without admixture with loading materials, can be estimated with sufficient accuracy by incinerating the paper, weighing the ash, and making a correction for the small proportion of the latter due to the fiber, etc. This proportion does not usually exceed 2 per cent.

The *ultramarines* are of variable and even doubtful composition, and are, therefore, best estimated by comparing the depth of color of the ash with that of standard mixtures of the pigment with known proportions of china clay.

*Chrome yellow, orange*, etc., also of variable composition, may be determined, if necessary, by estimating the lead and chromium separately, and calculating the results to the nearest indicated composition. It is scarcely necessary here to describe the full gravimetric process as it is likely to be but rarely required. It will be sufficient to say that the lead is precipitated and estimated as the sulphate, and the chromium as chromic oxide.

*Prussian blue* may be determined approximately by estimating the iron by igniting the paper, fusing the ash with sodium carbonate, treating the fused product with hot water, filtering, and boiling the residue with dilute hydrochloric acid and a drop or two of nitric acid. The solution is then again filtered, and the iron and alumina precipitated with ammonia in the presence of a little ammonium chloride. The precipitate of iron and aluminum hydrates is washed, filtered off, and digested with excess of caustic soda, then filtered again and carefully washed. The residue, which consists entirely of iron, is washed, dried, ignited, and weighed as the oxide. This process also serves for the estimation of all other iron pigments except the *natural pigments, ochres*, etc.

#### 7. Tests for Special Materials \*(28)

Oils and fats can be estimated by extracting with ether, evaporating the solvent, and weighing the residue.

*Paraffin-wax*.—Similar to the above, using benzine or petroleum spirit.

*Salicylic Acid*.—This substance is used as a preservative in papers required for wrapping foodstuffs. It is extractable with petroleum ether, and may be estimated in the solution by diluting the latter with an equal volume of 95 per cent alcohol and titrating with  $n/10$  normal alkali, using phenolphthalein as indicator. Each cc. of  $n/10$  normal caustic soda is equivalent to .0138 g. of salicylic acid.

*Carbolic Acid*.—The estimation of carbolic acid in carbolized wrapping paper is frequently required. Commercial carbolic acid consists chiefly of cresylic acid with higher phenols, but little real

phenol being usually present. Since, however, cresol is probably as efficient an antiseptic and insecticide for ordinary purposes as phenol, the absence of the latter body is of little importance. Carbolic acid may contain tar oils, which are, however, quite inert. Naphthalene is also liable to be present.

For the estimation of commercial carbolic acid the bromine-absorption method in use for the determination of phenol is valueless. The writer has found the following method, which is based on a process originally described by Muter, quite satisfactory:

From 10 to 20 g. of paper (according to the probable proportion of acid present) are cut into pieces and extracted with a sufficient quantity of alcohol (95 per cent) in a Soxhlet. The extract is transferred to a basin, mixed with about half its volume of a 10 per cent solution of caustic soda, and the mixed liquids evaporated in the water bath to small bulk. Tar oils and naphthalene, if present, here separate out and may be removed by filtration. The liquid is now transferred to a separating funnel and hydrochloric acid added cautiously and with gentle shaking until the liquid shows an acid reaction. Means should be taken to prevent the mixture becoming too hot during the process. A little brine is now added. The liberated tar acids rise to the surface of the liquid which also becomes milky from the precipitation of rosin. The whole is now set on one side for a short time to complete the separation of the layer of tar acids, after which the resinous liquid is drawn off as completely as possible. The residue of oil is shaken up with ether or petroleum spirit, transferred to the weighed flask, the solvent evaporated off, and the residue weighed.

#### 8. Free Acid in Paper

Weigh 10 grams of the paper to be tested, tear into small pieces, place in a 250 cc. porcelain casserole, and cover with a small amount of distilled water. Heat gently for an hour over water bath or electric hot plate. Pour off water and wash with small quantities of distilled water, adding it to water extract.

Another casserole is filled with an equal amount of distilled water, to which is added two drops of a methyl orange solution (0.1 per cent solution in water). To the former is then added tenth normal standard solution of caustic soda until the color matches the sample. The acidity is then expressed in terms of sulphuric acid ( $H_2SO_4$ ).

An alternate method is as follows: Take a piece of the paper six inches square, place in a saucer, and pour over it distilled water, and work about with a glass rod for 5 or 6 min. Now take a blue litmus paper or a little tincture of litmus and test the extract, when if either turn red it shows the presence of acid. Divide the extract into two parts; to one add a few drops of nitric acid, then nitrate of silver solution, when if a white curdy precipitate is formed, it proves the presence of *hydrochloric acid* or *chlorides*. To the second portion add a few drops of hydrochloric acid, heat to boiling in a test tube, and add a solution of barium chloride; a white precipitate indicates the presence of *sulphuric acid* or *sulphates*.

#### 9. Tarnishing Test \*(11)

A paper which is to be used for wrapping silverware should be essentially free from active sulphur compounds. The method of testing so called "anti-tarnish" paper consists, in general, of comparing the sample to be tested with special papers impregnated with 0.001 per cent and 0.0001 per cent  $Na_2S$  solutions, the sulphide test in each case being made under prescribed conditions by a hydrogen evolution method and lead acetate paper.

*Preparation of Special Impregnated Papers*.—Make the special papers from 10 cm. best white filter paper, each of which weighs approximately 0.6 gram. Prepare the following solutions:

a. Dissolve 3 grams of fresh sodium sulphide crystals in 100 cc. of distilled water. (3 g. of  $Na_2S \cdot 9H_2O$  are equivalent to 1 g of  $Na_2S$ .)



b. Dilute 1 cc. of solution (a) to 1 liter to make a 0.001 per cent  $\text{Na}_2\text{S}$  solution.

c. Dilute 10 cc. of solution (b) to 100 cc. to make a 0.0001 per cent  $\text{Na}_2\text{S}$  solution.

Saturate the filter paper in solution (b) and (c) and dry in air. Considerable quantities of these papers may be made at one time and stored in separate, tightly stoppered bottles labeled:

"0.001 per cent  $\text{Na}_2\text{S}$  paper for tarnishing test."

"0.0001 per cent  $\text{Na}_2\text{S}$  paper for tarnishing test."

The papers may also be torn into four equal segments, each segment (0.15 gram) being sufficient for one test.

*Materials Required*—(1) Four 500-cc. flat bottom flasks, approximately 7 inches high; (2) Granulated zinc (arsenic free); (3) 15 per cent HCl solution; (4) Lead acetate test paper, moistened; (5) Absorbent cotton.

*Method*—Into each flask put 2 grams of granulated zinc and 0.15 gram of paper torn into small pieces. The four flasks are for the following papers: (1) Sample; (2) Pure filter paper (for a "blank"); (3) 0.001 per cent  $\text{Na}_2\text{S}$  paper; and (4) 0.0001 per cent  $\text{Na}_2\text{S}$  paper.

Add to each flask 25 cc. of 15 per cent HCl (free from As). Into the neck of the flask insert a loose plug of cotton to a depth of about 1.5 in. Above the cotton place a piece of moistened lead acetate test paper about one inch square, and cover this loosely with a plug of cotton. Set the four flasks in a pan or tub containing water at room temperature to a depth of 0.25-0.50 inch, or in order to prevent any considerable rise of temperature of the contents of the flask. The liberated hydrogen will carry any  $\text{H}_2\text{S}$  evolved up to the lead acetate paper, which will darken. Examine the four lead acetate papers at the end of 30, 60 and 90 minutes and record their comparative appearances.

*Interpretation of Results*—It has been found that the 0.001 per cent  $\text{Na}_2\text{S}$  paper causes some tarnishing when held in contact with a polished 10 cent piece for five weeks. Commercial papers known to have caused tarnishing of polished metal goods have been found to be more reactive under this test than the 0.001 per cent  $\text{Na}_2\text{S}$  paper. Therefore, a paper to be acceptable should show up as well as the 0.0001 per cent  $\text{Na}_2\text{S}$  paper (which should show slight discoloration in about sixty minutes). A paper between 0.0001 per cent and 0.001 per cent  $\text{Na}_2\text{S}$  papers is dangerous; while those that are inferior to 0.001 per cent  $\text{Na}_2\text{S}$  paper should be unquestionably rejected.

In reporting, a paper superior to 0.0001 per cent  $\text{Na}_2\text{S}$  paper should be classed as "safe"; those between 0.0001 per cent and

0.001 per cent  $\text{Na}_2\text{S}$  as "questionable"; and those inferior to 0.001 per cent  $\text{Na}_2\text{S}$  as "unsafe."

Note—\*The practical use of paper for wrapping polished metal seems to indicate that sulphur in forms other than sodium sulphide will produce a tarnishing effect. This subject should be investigated before the method is adopted generally.

## V. INTERPRETATION OF DATA

The technique of paper testing has developed in no orderly or systematic manner and, in nearly all cases where paper is being tested, the methods are used chiefly for mill control purposes. In such cases, comparative results only are necessary and few attempts have been made to interpret the data obtained in any fundamental units. Little attention has been given the calibration of testing instruments or the experimental error of the methods. Provision for the proper error of sampling and testing is generally overlooked and considerable friction has at times developed for these reasons.

### 1. Relation of Various Tests

Some attempts have been made to draw a relation between some of the physical qualities of paper, with especial reference to *bursting, folding, tearing, and breaking strength*. Although there is a wealth of such data available, both published and in laboratory files, the only conclusions so far reached are entirely negative. In a given paper, it is quite possible to have a strong *bursting strength* but a weak *tearing and folding strength* and vice versa. It is interesting to note, however, that both the bursting and breaking strength are similarly affected by relative humidity, *i. e.*, a maximum strength occurs at about 35 per cent relative humidity. The amount of *rosin, glue or starch* present in a paper does not seem to have any relation to sizing quality, except in a very general way. It is recommended that this question of the relation of various tests be studied.

### 2. Quality Indicated by Tests

It is quite general practice to make certain tests on paper whether or not the test indicates the quality in question. This has been due to lack of test methods to some extent but discrimination should be exercised in the choice of the proper test. Folding endurance seems to be the best method of determining the durability and probable life of a paper, while the bursting strength is so affected by various factors that the data obtained with it are often misleading. It is recommended that data be collected and suggestions made as to the proper tests for various kinds of paper.

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