

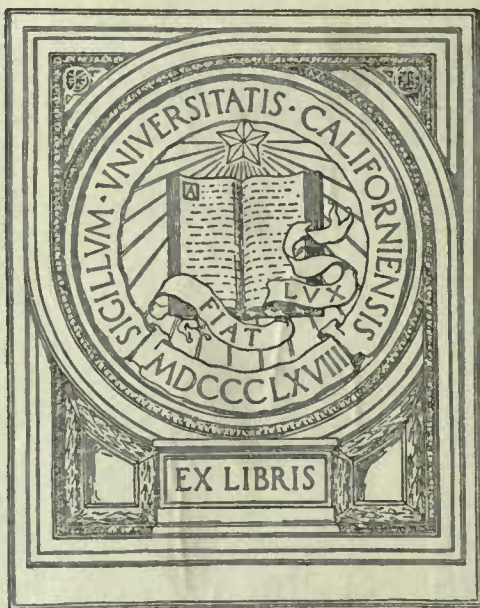
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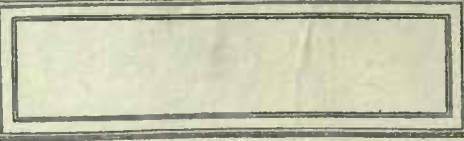
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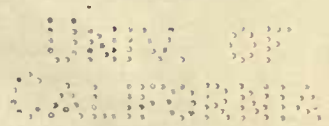


PAPER TESTING METHODS

Microscopical, Chemical, and Physical Processes Described
with an Account of the Apparatus Employed

By **FREDERICK C. CLARK**

Chairman of the Committee on Paper Testing of the
Technical Association of the Pulp and Paper Industry



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THE Paper Testing Committee of the Technical Association of the Pulp and Paper Industry presents the following revision of its previous work on Paper Testing, for the consideration of the association. This revision constitutes additions and corrections to the report of the Committee on Paper Testing as previously published.

Recent advances in paper testing methods have developed new procedures and shown the need of changes in some few of the previous methods.

All changes and additions to the previous report are presented to the association for comment, and it must be understood that some of them—for example: the qualitative tests for glue and rosin, can be only tentative until their application in general use has shown conclusively that they are worthy of adoption as standard methods.

Under stains there has been added what is known as the "Jenk's stain" as a supplement to the Herzberger stain for papers containing small amounts of rag.

Under testing devices, there have been included photographs of the Thwing paper scale, the new Perkins quadrant paper scale and the Thwing tearing tester.

A note on the meaning of "Substance Number" is also included by way of explanation.

Methods for determining the machine direction of paper are included.

Much discussion has taken place in the past regarding the use of the Schopper folding machine. In order to clarify certain points, there have been added detailed instructions for calibrating the machine, together with photographs showing the apparatus used in the calibrating method recommended.

The subject of "gloss" or "finish" has been taken up, and a method using the Ingersoll glarimeter is described in detail, together with a photograph of the apparatus. An article on the use of the Ingersoll glarimeter is in course of preparation, and will shortly be presented to the association through its official journal.

Under the head of "Specks in paper," there have been included detailed methods for identifying many kinds of dirt particles. These notes will prove helpful to many having work of this kind to do.

Under the head of "Fillers" have been added methods for the identification of various kinds of loading materials used in papermaking.

In order to simplify rosin determinations, there has been included a photograph and description of an

extraction apparatus that will be found not only to save time, but is also more handy to use and less expensive than the usual Soxhlet apparatus.

Recent work has been carried out for the purpose of determining the percent of starch in paper. The method included herein is recommended both because of its accuracy and its simplicity. Methods are also in process of completion to permit a differentiation between beater or pearl starch and tub sizing or treated and converted starches.

THE METHODS OF TESTING PAPER

The testing of paper is divided into three parts—namely: Microscopical, physical and chemical.

By microscopical examination the kind or kinds of fibers entering into the composition of a paper may be determined. The experienced observer is able to estimate the proportion of the various fibers used and such estimates are well within the limits of variation in the process of manufacture. By the use of the microscope it is often possible to indicate the presence of rosin size. It is also possible to distinguish different kinds of starches by the characteristic sizes and markings of starch grains. Some indication of the beating process is also secured as a light brushing action of the beater roll tends to fray out the ends of the fibers, whereas severe beating, with sharp tackle, cuts the fibers into smaller pieces without any tendency to fray out the ends.

The microscope is also of great assistance in helping to identify specks and dirt spots. A knowledge of the material that composes a dirt spot or specks will often give the clue as to where such spots originate, and means may thereby be taken to eliminate the cause.

Physical Testing—Under this heading are included such tests as are enumerated below, as follows:

- Area of the sample.
- Weight of the sample.
- Bursting strength per unit area.
- Thickness.
- Bulk or thickness of a number of sheets.
- Folding endurance.
- Tensile strength.
- Elongation at rupture.
- Absorption.
- Opacity.
- Gloss or glare.
- Translucency.
- Degree of sizing.
- Retention of loading.
- Breaking length.

Chemical Analysis—By chemical analysis it is possible to make the following determinations:

Percent of ash.
 Qualitative test to indicate kind of loading material used.
 Percent of paraffin.
 Sizing material used.
 Coating material used.
 Presence of acids, free chlorine, etc.
 (See chart of Paper Testing.)

MICROSCOPICAL EXAMINATION

Estimation of Fiber Content—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 percent 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 one minute, 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 No. 1) thoroughly dried on absorbent paper (Note No. 2) that is free from lint, placed on a microscopic slide and covered with several drops of Herzberg's stain. (Note No. 3.) 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 No. 4.) The slide is now ready for an estimation by the aid of the microscope. (Note No. 5.)

It is suggested that after the small sample of paper has been boiled with 0.5 percent caustic soda, that the sample be next washed with 0.5 percent 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 No. 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 $\frac{5}{8}$ -inch diameter and about 6 inches 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.

Second method. This was suggested by Ernest Mahler, general superintendent, and C. G. Bright, chemist, of the Kimberly-Clark Company, and later described in detail by George K. Spence, chief chemist, and John M. Krauss, assistant chemist, New York and Pennsylvania Company, in *Paper*, vol. xx, no. 11, page 12, May 23, 1917. 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 inches long. In place of the microscope needle a 10-inch 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 three minutes 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. (See also Notes 2, 3 and 4.)

NOTE No. 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 microscope 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 $1\frac{1}{4}$ inches 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 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.

Under Note No. 2 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.

HERZBERG'S STAIN

NOTE No. 3.—The Herzberg stain is made according to the following formula:

Solution A—20 grams zinc chloride.
 10 Cc. of water (preferably distilled).

Solution B—2.1 grams potassium iodide.
 0.1 gram iodine crystals.
 5 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 woodpulp, 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): 5.25 Gm. potassium iodide; 0.25 Gm. of iodine, and 12.5 Gm. 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 STAIN

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

TESTING OF PAPER TO DETERMINE QUALITY.

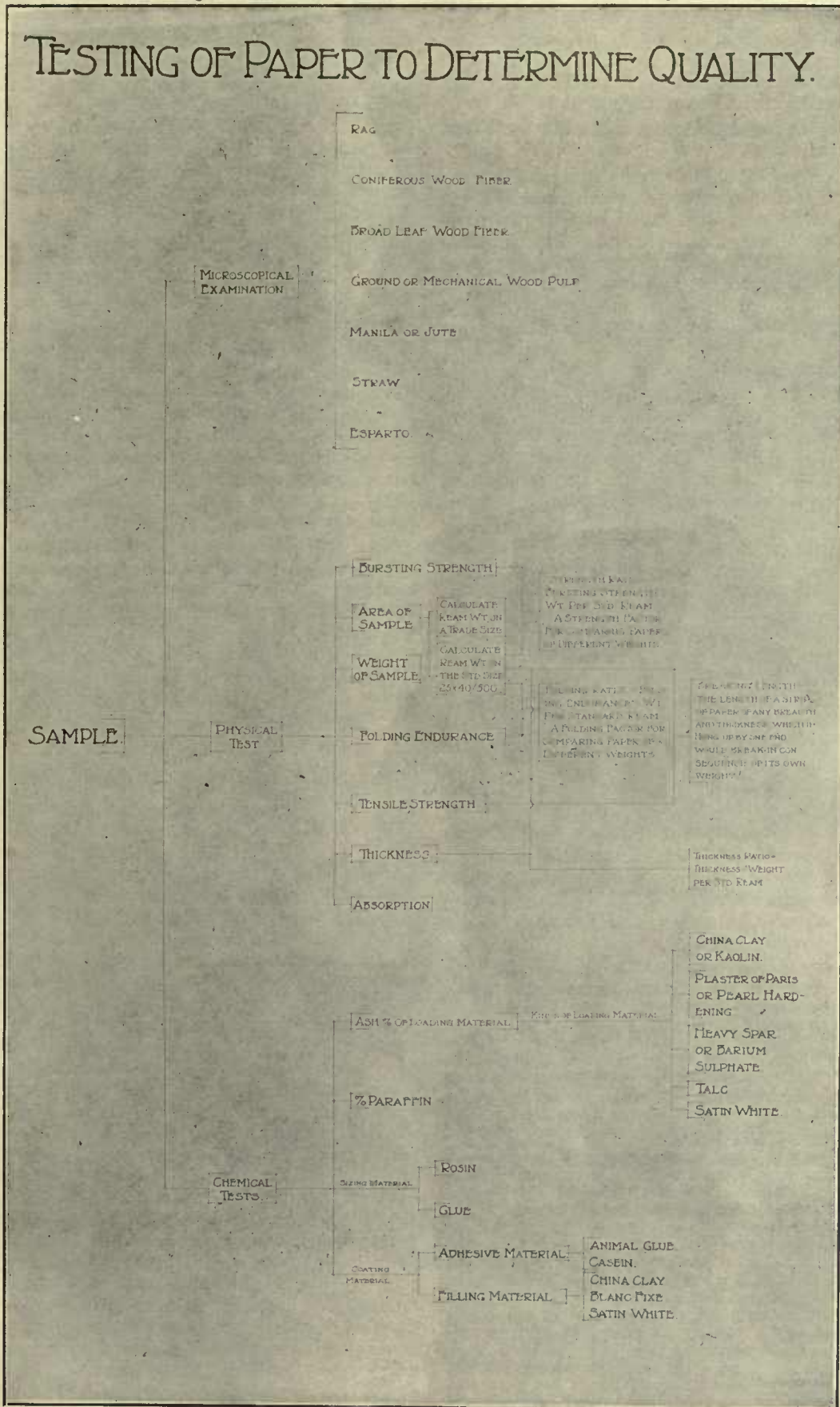


CHART OF PAPER TESTING SYSTEM OF THE TECHNICAL ASSOCIATION

chloride solution add $2\frac{1}{2}$ Cc. of iodine potassium iodide solution made up as follows: potassium iodide, Gm. 2; iodine, Gm. 1.15, 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.

The following suggestions are offered to those just beginning these tests.

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

It is the hope of the paper testing committee that it will very soon have a set of standard mixtures, available for anyone desiring to make use of them in comparing with unknown samples. These standards will also serve as a means of training the eye to judge proportion correctly.

NOTE No. 4.—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 diameter. 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 on each slide.

NOTE No. 5.—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 details 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 in

their article in *Paper*, vol. xx, no. 11, page 12 (May 23, 1917), recommend a magnification of 160 diameters.

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 percent. In making a fiber estimation no account is taken of the percent 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 (*Paper*, vol. xx, no. 11, page 11, May 23, 1917), which is worthy of description here and recommendation to the Technical Association. 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 percent 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 analysts. 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 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 $\frac{3}{8}$ inch thick by $\frac{1}{2}$ inch wide by 3 inches long (oak or maple may be used if the brass is not obtainable), then cut a groove $\frac{1}{8}$ inch wide by $\frac{1}{8}$ inch deep along one of the longitudinal edges of the brass strip. This groove then serves as a rest for the glass slides. (See Plate I.)

The pins (see sketch) 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.

STANDARD PAPERS FOR FIBER ANALYSIS

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

STAINS FOR SPECIAL PURPOSES

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

Phloroglucinol—Dissolve 5 grams of phloroglucinol in a mixture of 125 Cc. of distilled water and 125 Cc. of concentrated hydrochloric acid. The solution

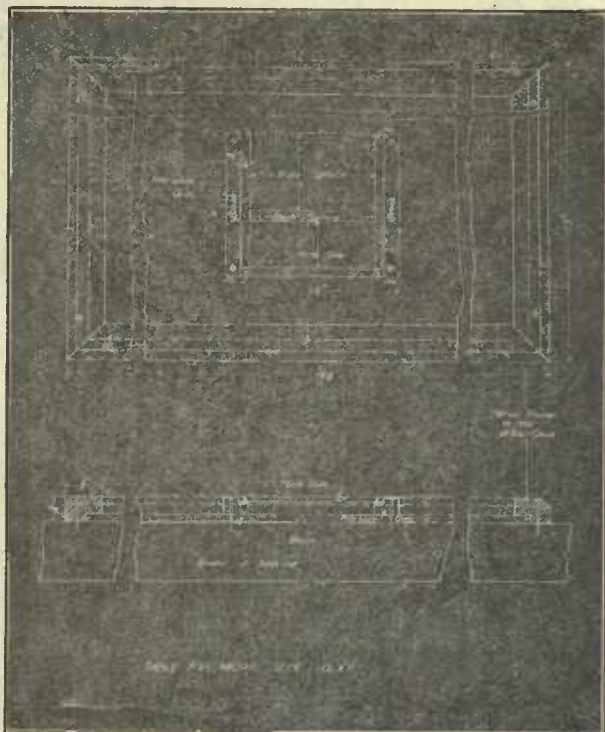


PLATE I

CLASSIFICATION OF VEGETABLE FIBERS USED IN PAPERMAKING

- A—Seed Hair Fiber
 - { Cotton
 - { Bombax Wool—East Indies
- B—Stem Fiber (Bast family)
 - { Flax or Linen
 - { Hemp—(Borders of Mediterranean)
 - { Jute or Calcutta Hemp
 - { Nettle Fibers
 - { Common Nettle
 - { China Grass
 - { Ramie—Water-resisting
 - { Sunn Hemp (Java)
 - { Manila Hemp
 - { Straw and Esparto
- C—Leaf Fiber
 - { New Zealand Hemp
 - { Manila Hemp
 - { Sisal or Domingo Hemp
 - { Aloe Fibers (South America)
 - { Pineapple Leaf Fiber
 - { Wool from green
 - { Vegetable Wool from green cones of Pine and Fir
- D—Fruit Fiber
 - { Coconut Fiber
- E—Wood Fiber
 - { Resinous or Coniferous
 - { Larch-Tamarack
 - { Fir
 - { Spruce
 - { Cedar
 - { Pine
 - { Hemlock
 - { Cypress
 - { Birch
 - { Mulberry
 - { Non-resinous or Broad-leaf
 - { Beech
 - { Gum
 - { Tulip Tree
 - { Poplar

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 groundwood pulp. The color may easily be noted by applying some of the stain to a piece of newsprint paper. There is approximately 80 percent of groundwood in newspaper so that a deep magenta color is developed. The depth of color is an indication of the amount of groundwood present. A very light shade of color, however, does not necessarily prove the presence of groundwood, as partly cooked jute, partly cooked unbleached sulphite pulp, and some other fibers are also slightly colored.

Aniline Sulphate—Dissolve 5 grams of aniline sulphate in 50 Cc. of distilled water and acidulate with one drop of concentrated sulphuric acid. This stain produces a yellow color on papers containing a large percentage of groundwood. This stain is not quite as sensitive to groundwood as phloroglucinol, but it is easier to obtain and prepare.

Para-nitroaniline—Saturated solution in concentrated hydrochloric acid. This stain produces an orange yellow color in the presence of groundwood and other lignified fibers.

The C. G. Bright Stain—This is used for distinguishing between bleached and unbleached pulps.

CLASSIFICATION AND CHARACTERISTICS OF THE MORE COMMON VEGETABLE FIBERS USED IN THE TEXTILE AND PAPER INDUSTRIES

Fiber	B. Stem or Bast Fibers (Dicotyledonous plants)										
	A. Seed Fibers or Seed Hairs	Flax (Linum usitatissimum)	Hemp (Cannabis sativa)	Jute (Corchorus capsularis)	Manila hemp (Musa textilis)	Straw (Wheat, oat)	Esparto (Stipa tenacissima)				
1 Shape	Cotton (Gossypium) Flat twisted ribbon	Cylindrical	Blunt end 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 pearshaped 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 $\frac{1}{4}$ 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"	$\frac{1}{5}$ " to $2\text{-}3\frac{1}{5}$ " average $1\text{-}1\frac{1}{5}$ "	$\frac{1}{20}$ " to $1\frac{1}{4}$ "	$\frac{1}{20}$ " to $1\frac{1}{5}$ "	$\frac{1}{10}$ " to $1\frac{1}{2}$ " average $1\frac{1}{4}$ "	$\frac{1}{250}$ " to $1\frac{1}{20}$ " average $1\frac{1}{35}$ "	$1\frac{1}{50}$ " to $1\frac{1}{10}$ "				
10 Breadth	.0047" 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 Chloride of Zinc	Wine red	Purple to yellow	Purple to yellow	Yellow	Uncooked-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	C. Leaf fibers (See B for Manila Hemp) (From leaves of many monocotyledonous plants)				E. Wood fibers from the trunk or stems of various coniferous and hard wood trees.		
	B. Stem or Bast Fibres	New Zealand Hemp (Phormium tenax)	Sisal or Domingo hemp (Agave rigida)	Pita fiber (Agave americana)	pineapple leaf fibre or silk grass (Ananas sativa)	Coniferous wood fibers	Broad leaf hard wood fibers
1 Shape	Kamic (Urticaceae) anettic fibre (Boehmeria tenacissima) Jointed and with transverse fissures	Regular and uniform	Stiff, Broad at middle	Wavy elastic fiber. Stiff and short	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	Thin			
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 par. enchyma and epidermis often on fibre	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				
6 Epidermal cells						None	None
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	Distinctly wavy and elastic. Similar to Sisal in appearance		Markings less distinct in well boiled pulps. Occasionally twisted	Always accompanied by short tubular vessels of large diam with numerous pits
9 Length	1/4" to 2.0" Average 1.0"	1/10" to 3/5" Average 1/3"	1/10" to 1/4"	1/10" to 1/5"	1/10" to 2 1/2"		
10 Breadth	.010" to .020" Average .0125"	.0020" to .0070" Average .0040"	.0050" to .0085"	.0040" to .008" Average .0055"	.0010" to .0020"		
11 Color Reactions with Chloriodide of Zinc	Dark red to green yellow	Dark yellow or brownish	Yellow	Yellow	Blue to greenish yellow	Blue	Blue
12 With Philoroglucine	Colorless	Reddish	Red	Red	Colorless to light red	Colorless	Colorless
13 With Aniline sulphate	Colorless	Yellow green	Yellow	Yellow	Colorless to yellow	Colorless	Colorless

Solution A: Tenth normal ferric chloride solution equal to 2.7 grams $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ per 100 Cc. distilled water.

Solution B: Tenth normal potassium ferricyanide solution equal to 3.29 grams $\text{K}_3\text{Fe}(\text{CN})_6$ per 100 Cc. distilled water.

Solutions A and B should each be filtered through a fresh filter into clear glass stoppered bottles. Equal volumes are mixed fresh whenever the reagent is used.

Solution C: Substantive Red—0.4 gram of benzo-purpurin 4B extra (Bayer Co), 0.1 gram of oxamine brilliant red BX (Badische Co.) and 100 Cc. of distilled water. Have water hot and stir in the dyes slowly.

The staining solutions are used in tall narrow cylindrical beakers, which are set into a water bath. The slides are suspended in the beaker by a clamp

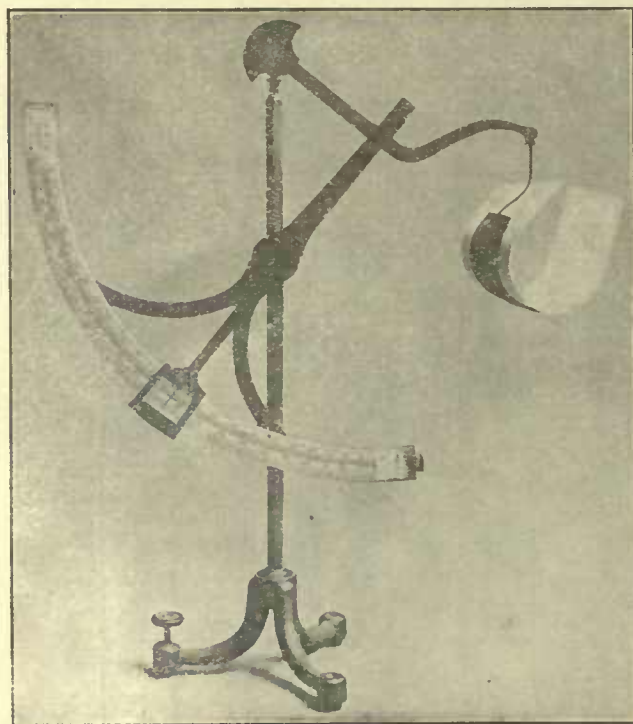


PLATE 2

which holds them at their upper ends, the clamps resting across the top of the beakers. The bath is heated by a small bunsen burner with a pilot flame, so that when the required temperature is reached the pilot flame may be used to maintain the temperature at the required amount. A thermometer should be suspended in the stain and the beaker containing the stain should be as small as possible so as not to use too much stain at one time.

In making up the slides for this staining method it will be necessary to use the dropper method as used by Bright and also by Spence. (See methods of making up microscope slides as outlined in first part of this report). This dropper method involves dropping a dilute mixture of water and fibers upon the slide and then evaporating the water. The dry slide is then ready for staining.

Method of using solutions A and B known as the potassium ferric ferricyanide stain.

Mix equal volumes of solutions A and B, heat to 35° Cent. in the water bath, regulating the pilot flame so that the temperature will remain constant within

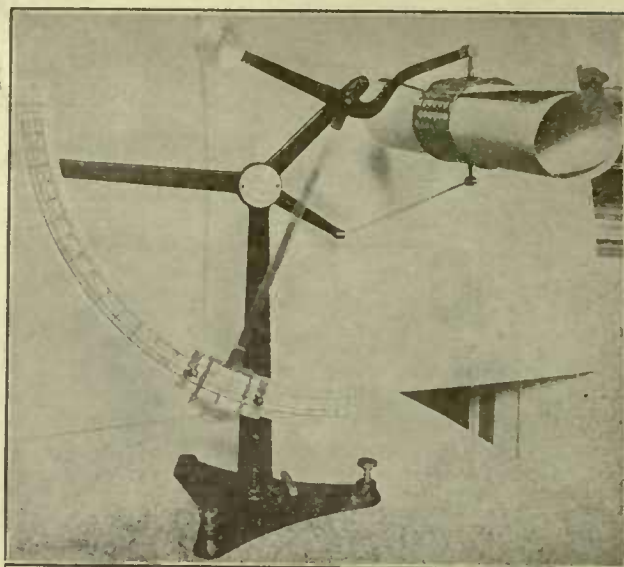


PLATE 2A

one degree for a period not less than fifteen minutes. The dry slide is then dipped in distilled water to moisten it uniformly, so that air bubbles will not be formed when it is immersed in the stain. If air bubbles are formed the fibers under the bubbles will not be stained. If dipping in water still leaves bubbles, they can be removed by blowing across the slide from the edge. The slide is then suspended in the stain and left there for fifteen minutes at 35° Cent. It is then removed and washed, by dipping in and out of a beaker of distilled water six times and repeating

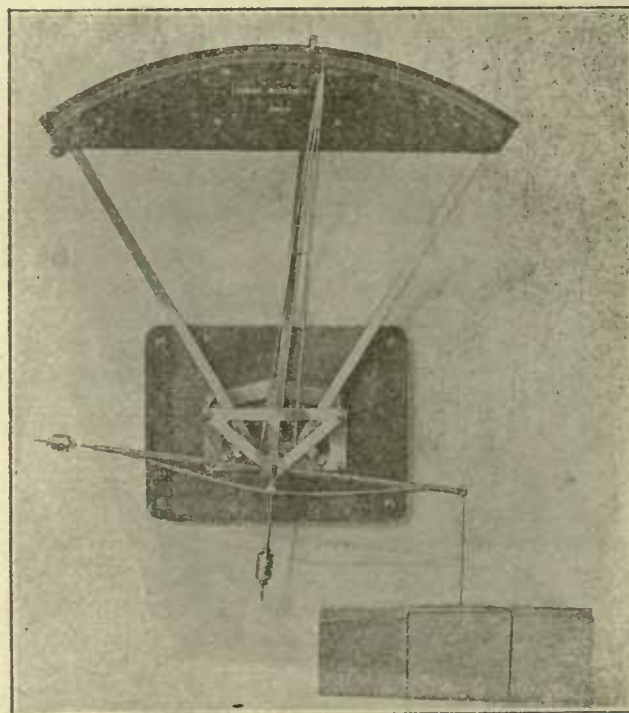


PLATE 2B

the process in a fresh beaker of water. The slide can then be placed wet into the red solution, but it is perhaps better to dry it out so that the fibers will be stuck together tightly again in case they have been loosened to any extent by the treatment.

Method of using solution C, the substantive red stain: A fresh solution is heated to 45° Cent., and the slide, after moistening and excluding bubbles as before, is suspended in the solution for five minutes at 45° Cent. and immediately washed in two beakers of distilled water.

The slide is then dried and a cover glass placed on with a drop of balsam.

Directions for assuring best results. To get the clearest, brightest results, distilled water must be used throughout, and the staining solutions must be fresh. The two solutions for making ferric ferrieyanide will keep well if placed in separate bottles. Equal volumes are mixed together immediately before using. The red solution should be freshly made each time for the best results, as it gets thick and stringy on standing, especially when it is being heated up continually.

Staining under the conditions described gives an unbleached sulphite perhaps the deepest blue it is possible to obtain without depositing blue on the slide and on the bleached sulphite; the method also produces the best red on the bleached fibers without turning purple the unbleached fibers. Unbleached sulphite from different mills varies considerably in lignin content, hence some samples stain a deeper blue than others. The foregoing conditions give a satisfactory blue on a sample of high grade imported unbleached pulp as well as a better color on the average run of unbleached pulps, the latter being not so well cooked as a rule. With pulp containing more lignin it is possible to use a slightly stronger treatment with the red and thus get a better color on the bleached without affecting the unbleached.

After a man has had a little experience with the method he can tell by the color of the unbleached fiber whether he may safely continue the staining with the red for six or possibly seven minutes at 45° Cent. At first, however, it is better to follow the directions as given. It is of prime importance to wash out or neutralize every trace of alkali in the fibers, as the blue is decolorized by alkali.

This method of staining will in general give a distinction between pure cellulose fibers and those which contain lignin. Rags, bleached sulphite, soda pulp or any thoroughly bleached material are stained red while unbleached sulphite, groundwood, jute, or any lignified materials are stained blue. The principal application lies in the estimation of unbleached pulp in book papers. A considerable saving can be made by using unbleached sulphite instead of bleached, hence it is important to know how much unbleached pulp there is in a sheet.

PHYSICAL TEST

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.

Weight of a sample—The sheet-weighing device that indicates the equivalent weight in pounds in terms of a 500 sheet ream, is most suitable for laboratory or mill use. The quadrant type scale, sold by Cornelius Kahlen, New York City, is most easily handled as no sliding poise is required. (See Plate 2.) 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 multi-

plied by 1.102, will give the equivalent weight of 500 sheets of the size weighed.

IMPROVED QUADRANT SCALE

Recent improvements in the quadrant type scale are shown in Plate 2-A. This scale is made by the B. F. Perkins Company, of Holyoke, Mass., and is designed to give the weight of a ream quickly without excessive loss of time due to swing of the basket or vibration of the indicator.

Plate 2-B shows another type of sheet weighing scale made by the Thwing Instrument Company, Philadelphia.

The weight of the sample should also be converted to the standard size (25x40, 500). This size is equal to an area of 1,000 square inches and is recommended for universal use in reporting all laboratory tests. To secure greater understanding of the sample in question, the weight should also be calculated on the trade size for that particular kind of paper.

Formula for small sample weighed on chemical balance:

$$\frac{(\text{wt. in grams}) \times 1.102}{\text{Area of the sample in square inches}} = \text{weight } 25 \times 40, 500.$$

$$\frac{(\text{wt. in grams}) \times (1.102) \times (\text{area of trade size desired})}{\text{Area of the samples in square inches.}}$$

weight on trade size desired.

Formula for sample weight on sheet paper scales:

$$\frac{(\text{wt. in lb}) \times (1,000)}{\text{Area of sheet in square inches.}}$$

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

There is a second type of sheet weighing scale on the market (See Plate 3) of the pea and beam type. This type of scales is customarily used in the paper mill, and is made by the Fairbanks Company.

TO DETERMINE THE SUBSTANCE NUMBER

The weight of a ream folio size, 17" x 22"—500, can be stated as substance number.

A method for determining 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" 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" weighed on the analytical balance has a substance number equal to the weight of the cut sample in centigrams.

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

$$\frac{\text{Weight in centigrams} \times 187000}{187110} = \text{substance number.}$$

BURSTING STRENGTH

There are two general types of apparatus used to determine bursting strength. 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 one square inch. The Mullen tester (See Plate 4), made by B. F. Perkins & Sons, Inc., Holyoke, Mass., and the District of Columbia Paper tester (See Plate 5), made by the District of Columbia Paper Manufacturing Company of Washington, D. C., are of the hydraulic type. The second type of bursting strength apparatus 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. The Ashcroft tester (See Plate 6) made by the Ashcroft Company, of New York, is the only one of this type now on the market.

The bursting strength 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

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 (See Plate 7) of thickness gauge 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 gauge: B. C.

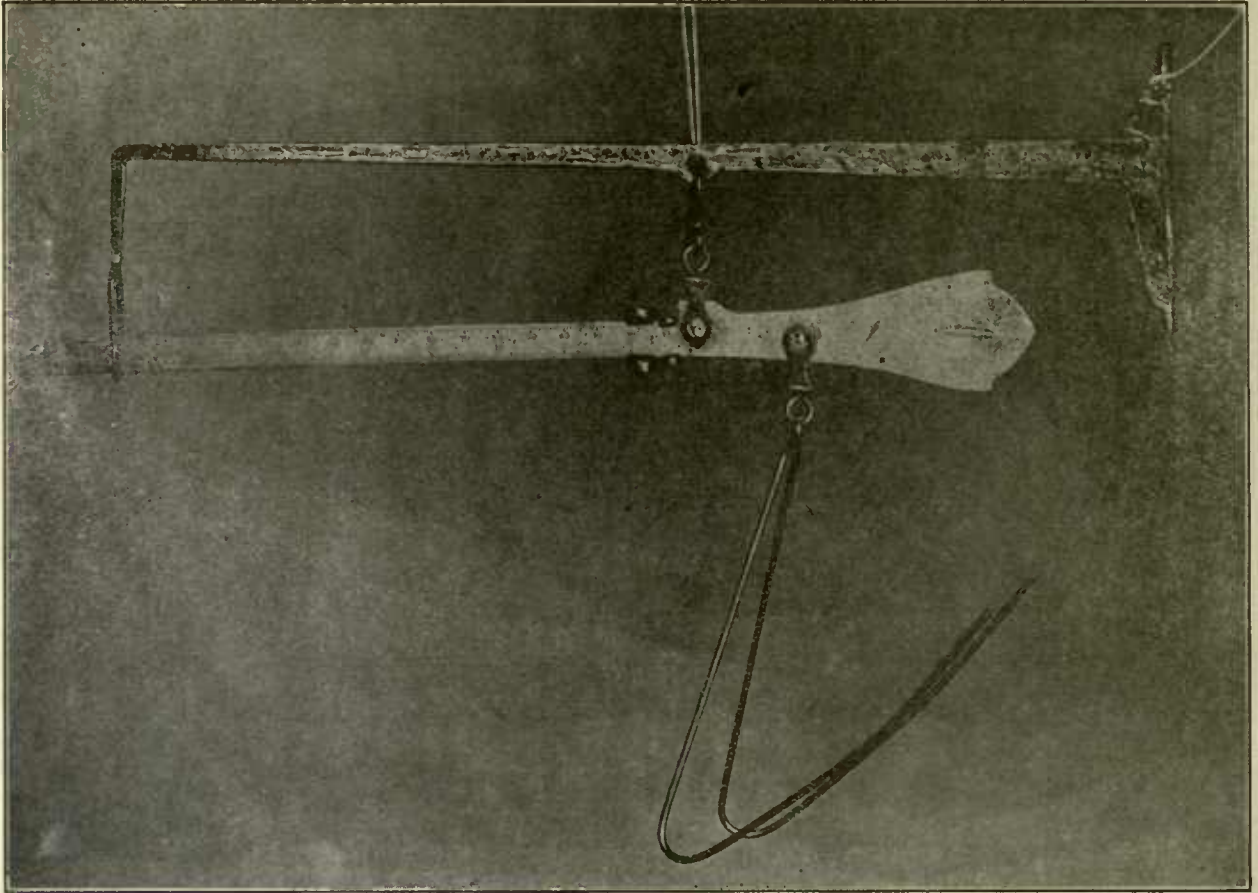


PLATE 3

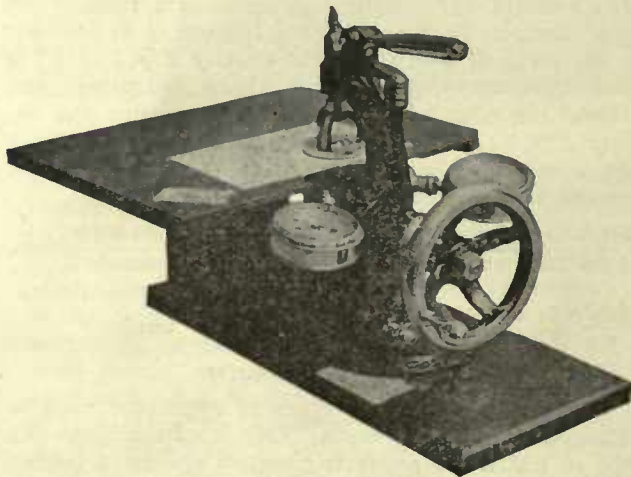


PLATE 4

compared with the strength ratio of any other paper if the same standard size sheet is used in each case. The strength ratio is expressed as a percentage.

$$\text{Strength Ratio} = \frac{\text{Bursting strength} \times 100}{\text{weight in pounds (on a size 25} \times 40,500)}$$

THICKNESS TESTER

The thickness of a paper may best be determined

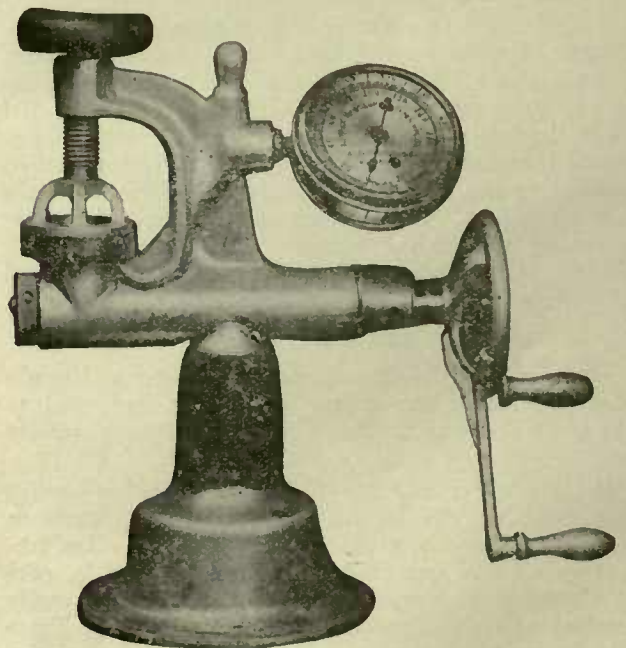


PLATE 5

Ames Co., Waltham, Mass.; B. F. Perkins & Son, Inc., Holyoke, Mass.; Storrs & Bement Co., 140-150

Federal Street, Boston, Mass.; The Ashcroft Mfg. Co., 85-89 Liberty Street, New York; Cornelius Kahlen, 349 Broadway, New York.

It is advisable to have all thickness gauges calibrated before use.. This may best be done by securing a set of standard sheet metal leaf gauges, which

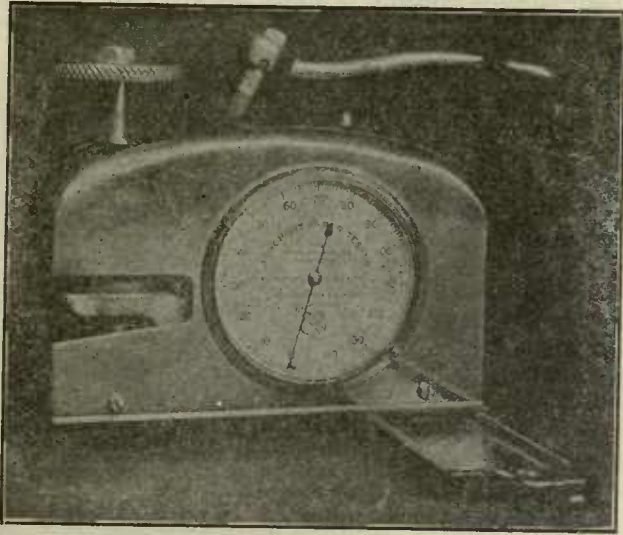


PLATE 6

range from 0.001 to 0.015 inches. This range of leaf gauges 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 compactness of several papers, the following formula is suggested:

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

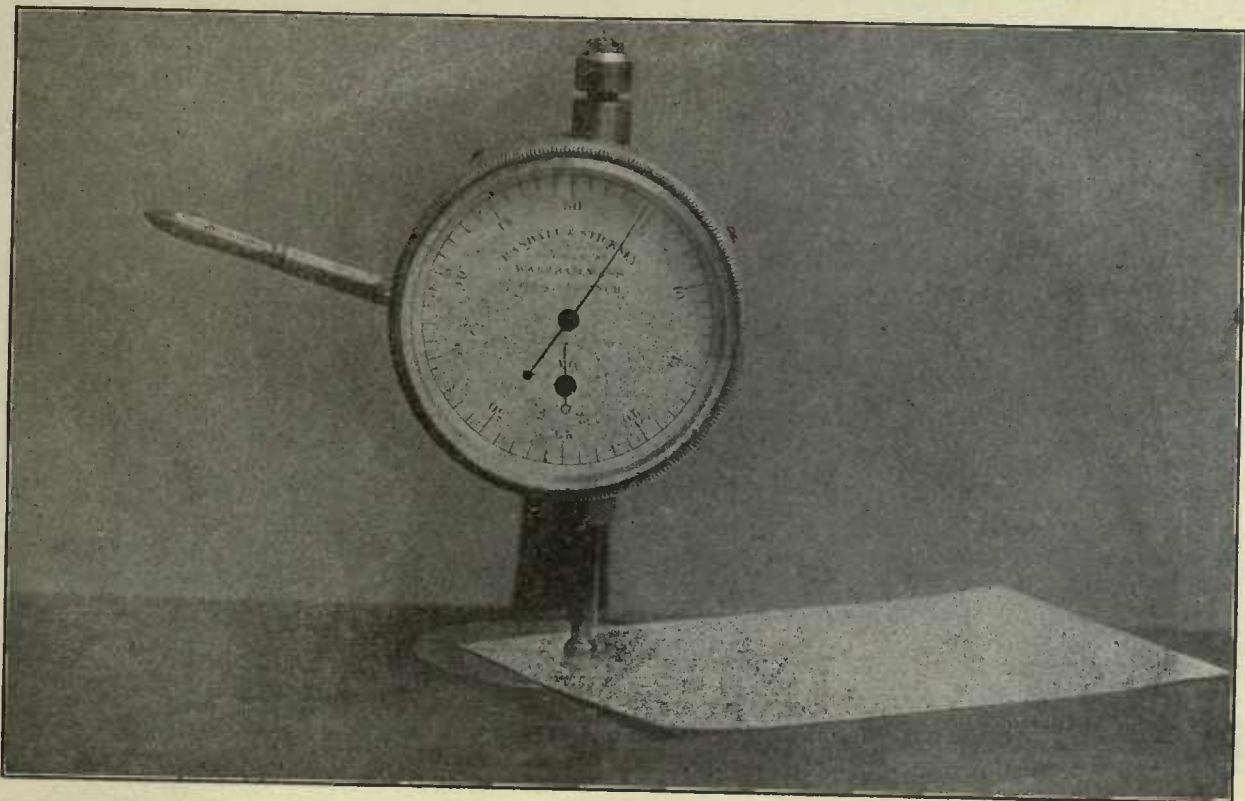
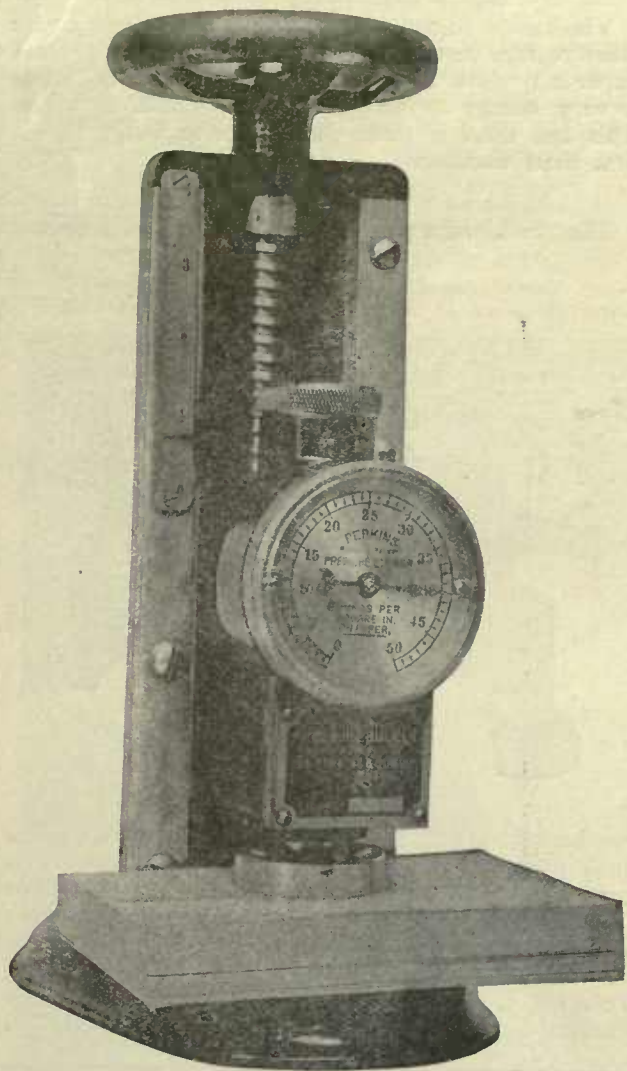


PLATE 7

The factor 10,000 serves to give a resultant figure more readily remembered. A very highly compressed paper may show a relative compactness=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 first sheet mentioned.

BULK TESTERS

The "bulk" of a paper is the thickness of a certain

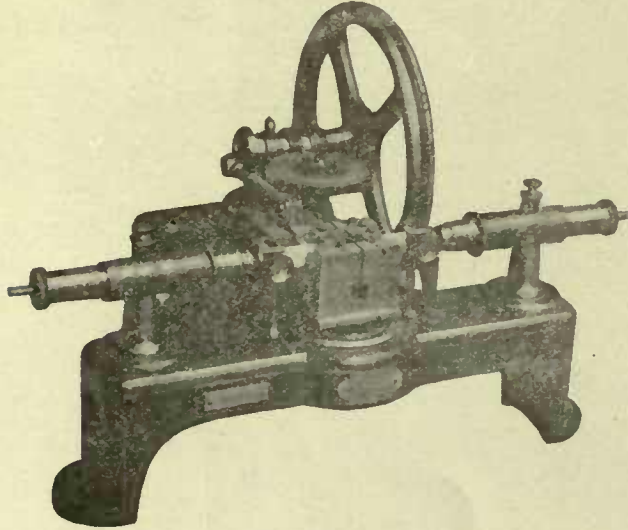


PLATE 9

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 (See Plate 8), made by B. F.

Perkins & Son, Inc., Holyoke, Mass. 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.

MACHINE AND CROSS DIRECTIONS

A disk of paper about 3 inches in diameter is dropped upon water. The valley formed by the curling of the disk lies in the machine direction.

Machine and cross directions may be distinguished by cutting two strips about 8 inches long and $\frac{3}{4}$ inch wide from the sample, the second one to be cut at right angles to the first. When these are put together and held upright by their lower ends one will bend lower than the other when tipped one way. By tipping back in the opposite direction the stiffer strip will support the other. The strip bending least by its own weight is cut in the machine direction. That bending most is cut in the cross direction.

FOLDING ENDURANCE

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. The Schopper folding machine, marketed by Cornelius Kahlen, New York (See Plate 9), is the only device so far made to carry out this test. A. B. Green, of Erie, Pa., has recently patented a new folding device.

The folding strength of paper is dependent not

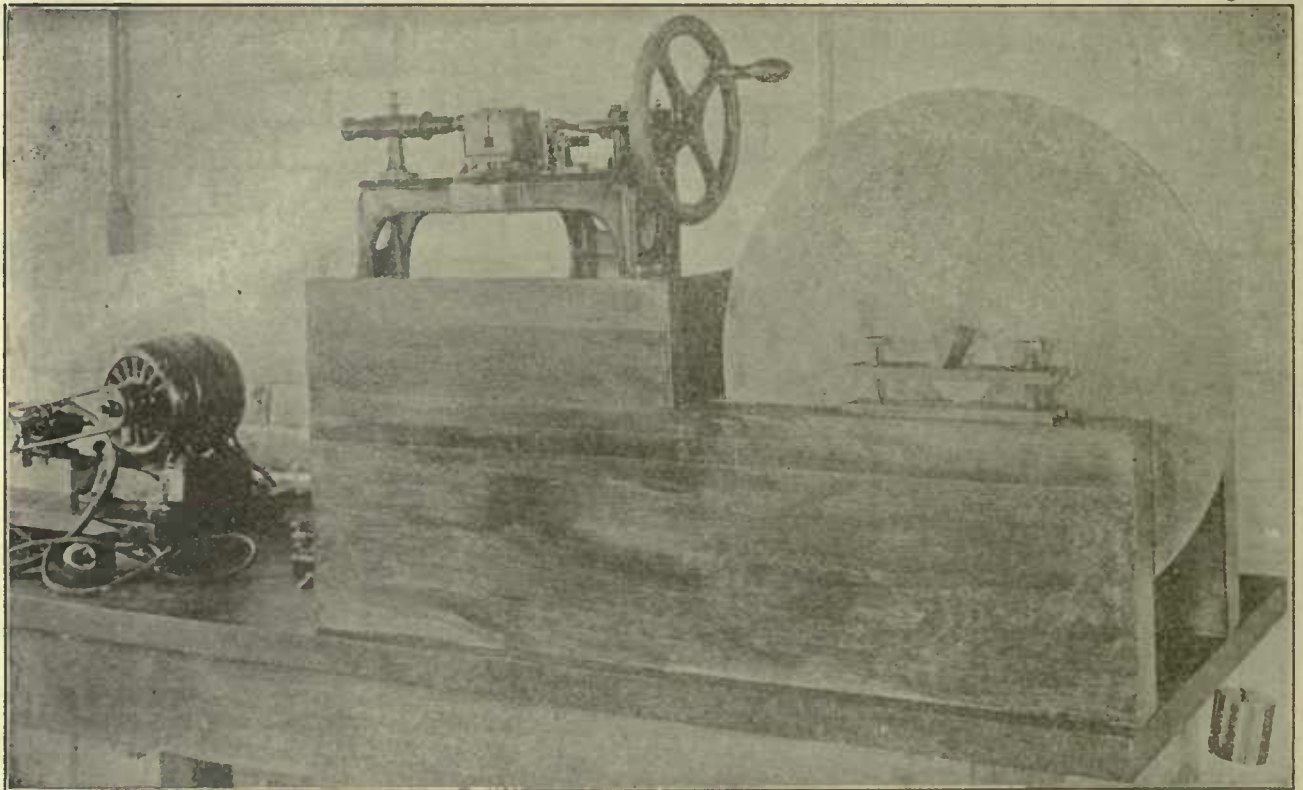


PLATE 9-A

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 the use of a room where the humidity is under control. Where such a room is not available then note must be made of the percent 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 percent 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.

TO CALIBRATE THE SCHOPPER FOLDING MACHINE

A machine along the lines of the one illustrated (Plate 9A) was designed at the Bureau of Standards for the purpose of calibrating the springs acting on the clamping jaws. This consists primarily 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 $\frac{1}{4}$ " 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° 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 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 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 done, it will be unnecessary to remove 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 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.

NOTE—In connection with the folding tester attention should be called to the fact that it is absolutely essential

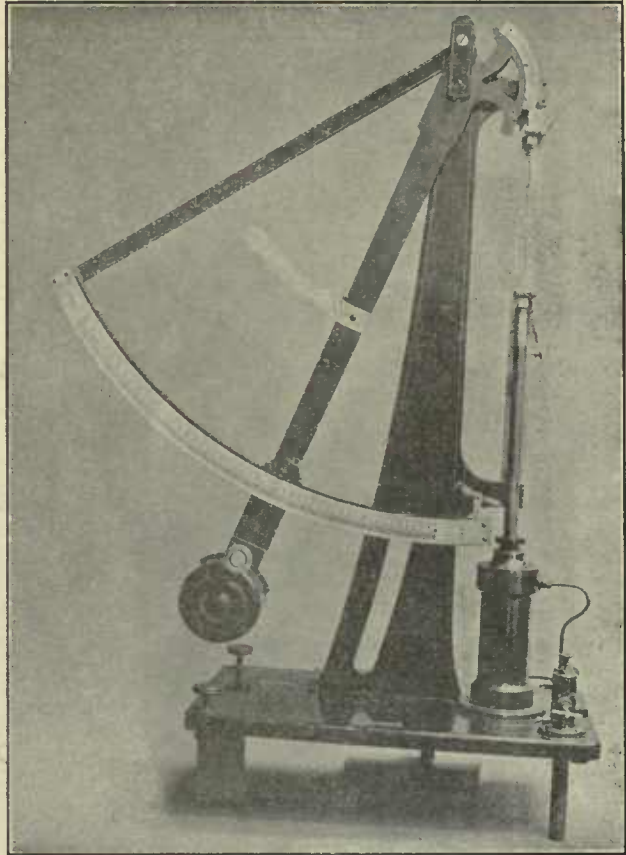


PLATE 10

that 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 percent in the results.

TENSILE STRENGTH

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, marketed by Cornelius Kahlen, New York (See Plate 10).

In this device (See Plate 10) a strip of paper 15 Mm. (approximately $19/32$ inch) wide by 180 Mm. long (approximately $7-3/16$ inches) 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 2.2lb) 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. See following formula:

$$(3.73) \times (\text{Tensile strength in Kg. per 15 Mm. width}) = \text{Tensile strength in lb per one inch width.}$$

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

$$\frac{(\text{Tensile strength in lb per 1 inch width})}{(\text{Weight } 25 \times 40, 500)} = \text{Tensile strength factor.}$$

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:

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

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

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

ELONGATION AT RUPTURE

The amount of elongation at the instant of rupture of a strip of paper under tension is measured on the

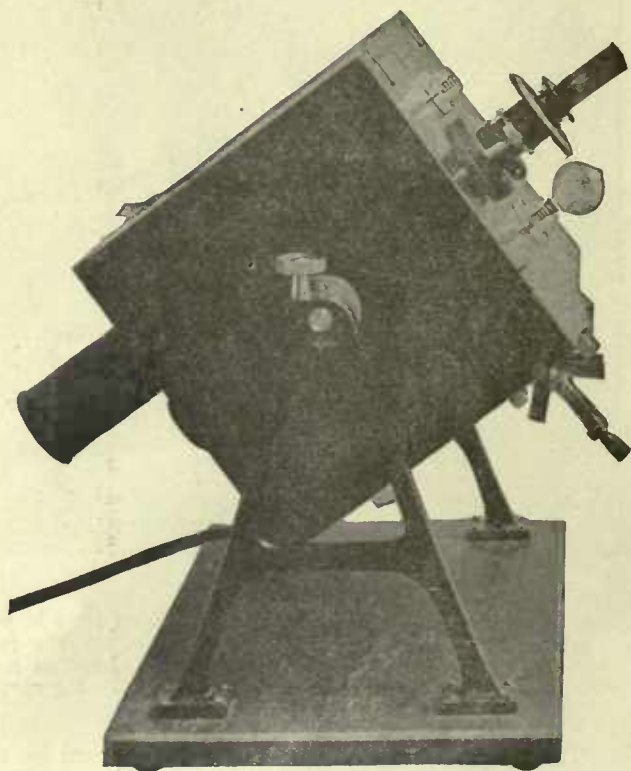


PLATE 11

Schopper tensile strength machine. The result is figured as a percent of the total original length.

ABSORPTION

The absorption of a blotting paper is indicated by the height in millimeters 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 millimeters to which the liquid (preferably water) will rise in ten minutes is taken as a measure of the relative absorption of the paper.

OPACITY AND TRANSLUCENCY

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

(Copy may be secured by addressing Bureau of Standards, Washington, D. C.) Briefly, the method involves a determination of the difference in photometric brightness, or contrast, between a black and white spot, when covered with the material to be tested. The instrument used is a Martens photometer in a specially constructed box. (See Circular No. 63.)

The figure expressing the "contrast ratio varies between zero and one. Values nearer zero indicate a more transparent paper, such as onion skin papers, tracing papers, etc., and larger values indicate greater opacity. The highest grades of tracing cloth will have a "contrast ratio" as low as 0.20, while a paper for envelopes, where opaqueness is essential, should have a "contrast ratio" of not less than 0.90 (See Plate 11 of apparatus.)

ESTIMATING TEARING STRENGTH BY THE THWING TEARING TESTER

A photograph of the Thwing tearing tester is shown in Plate 10C. From this photograph it can be seen that the test is carried out on a small sample of paper punched from a sheet by a special punch attached to the tester. The sample is slit part way and has six alined perforations following the slit to guide the tear. The sample is then pinned to two pins, one of which is attached to a movable weight on an arm carrying a recording pen, and the other attached to a sliding record card holder, which is moved by a motor drive. The machine is set in motion by electric contact, and as the record card-holder moves horizontally and tears the sheet along the alined perforations, the resistance of the sheet against tearing between the perforations swings the suspended weight and dips the attached pen, so that it makes a continuous record of peaks across the card; each peak being recorded as a definite force in grams required to tear the paper between two perforations. The average of the five different tears between the perforations is taken as the force required to tear this paper in grams. The weights governing the force applied can be varied by changing their position on the lever arm, or by substituting light and heavy weights, thereby adapting the machine to make uniform graphic records for a wide range of papers.

DEGREE OF SIZING

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 described by C. Frank Sammet, Circular 107 of the Bureau of Chemistry (The Detection of Faulty Sizing in Paper) also published in *Paper*, vol. x, 9, pp. 15-16, Feb. 12, 1913. 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 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.

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

Tannic acid (dry)	23.4 grams
Gallic acid (crystals)	7.7 grams
Ferrous sulphate	30.0 grams
Dilute hydrochloric acid (U. S. P.)....	25.00 Cc.
Phenol	1.0 grams
Blue Dye (Bavarian Blue S. & J. No. 478) .	2.2 grams
Water to make up to 1,000 Cc., allow to settle, and decant from any sediment.	

NOTE—Any water-soluble aniline blue, as methylene blue, may be used in place of Bavarian blue.

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

MEASUREMENT OF GLOSS

The percentage of gloss or glare on papers of any furnish or color can be determined very readily by means of the Ingersoll glaremeter. See Plate 10A. A sample of the paper to be tested should be cut with such dimensions as to allow it to cover the whole area included by the spring clasp which is located at the foot of the polariscope. When the sample is clamped properly in the field of light, the case should be closed, and all unnecessary light excluded from the room. Then, by rotating the eyepiece somewhere between the scale readings of 75° and 115° , a point can be found where the two fields

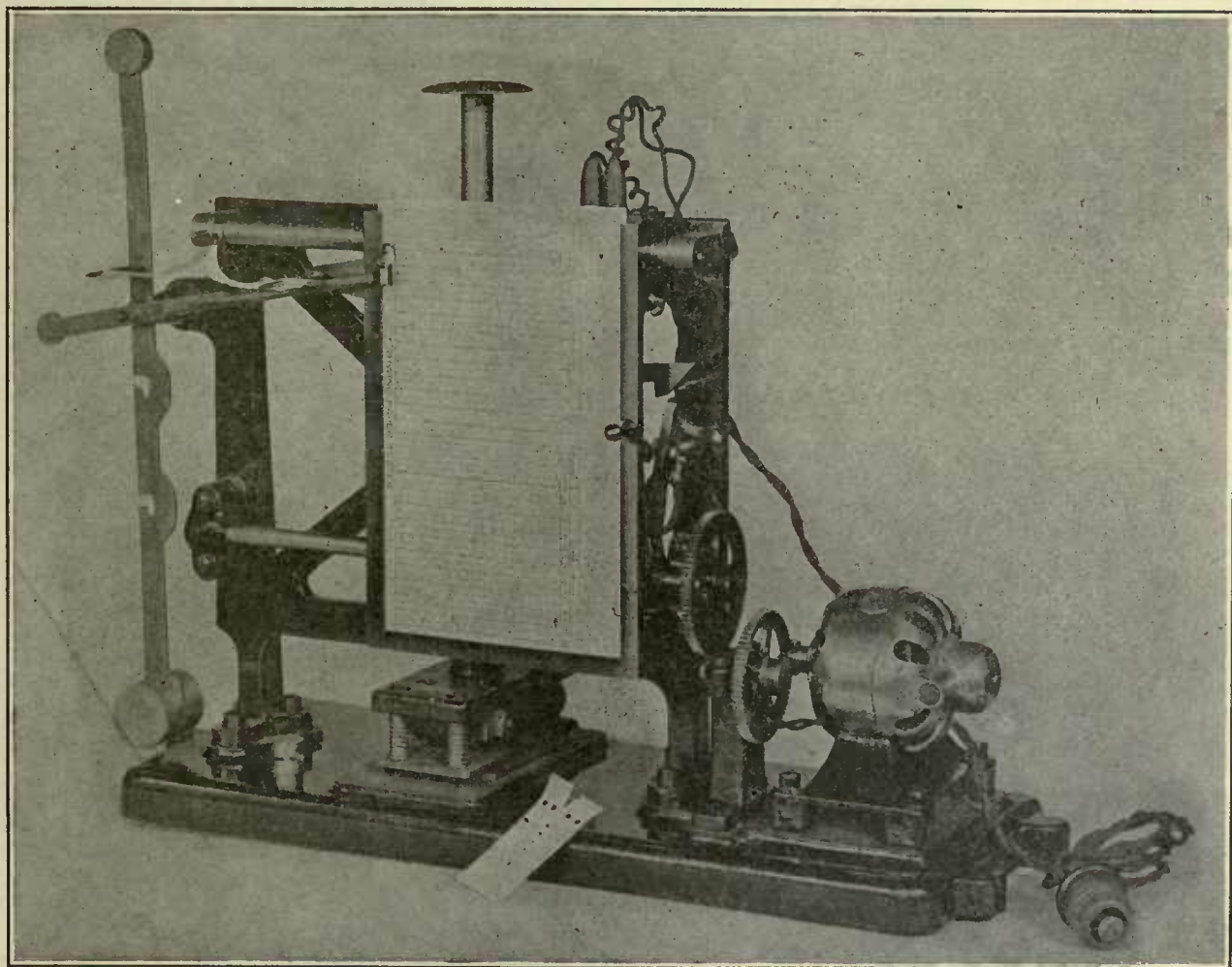


PLATE 10-C

SIZING EFFECT—ALTERNATIVE METHOD

For comparative sizing effect, squares 2"x2" are cut from each sample. These are subjected for at least thirty minutes 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 moisture content influences the penetration of the paper and different people have different judgments as to when the ink is "through."

as seen through the polariscope are of equal brilliancy. This is the desired reading and should be checked within half a degree at least three times before being taken as final. When this reading in degrees is located on the graph or table which accompanies the instrument, the corresponding percentage of glare can be obtained directly.

When difficulty is encountered in matching the fields while testing various colored papers, a light red stained glass should be interposed between the eye of the observer and the eyepiece. This eliminates all color differences and allows the light intensities to be matched very readily.

NOTE—The Ingersoll glaremeter was devised by Dr. L. R. Ingersoll of the Forest Products Laboratory, University of Wisconsin. The original article appeared in the *Electrical World* for March 21, 1914.

RETENTION OF LOADING

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

Secure about a five-pound 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 grams of loading material is obtained, which is then placed in a bottle for further use. From this bottle, remove a 1-gram sample, dry at 105° Cent. to constant weight and calculate percent 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 percent 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 percent of moisture and percent of ash. Weigh pulp added to the beater. Weigh 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

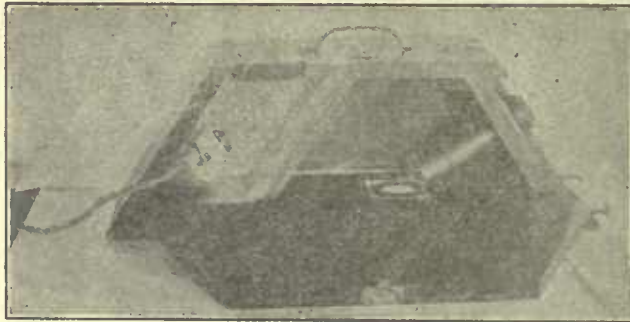


PLATE 10-A

above paper. The above mentioned data used in the following formulas will give the percent of clay used and the percent retention.

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

The formulae for percent of clay should be as follows:

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

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

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

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

The value of K is the percent 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

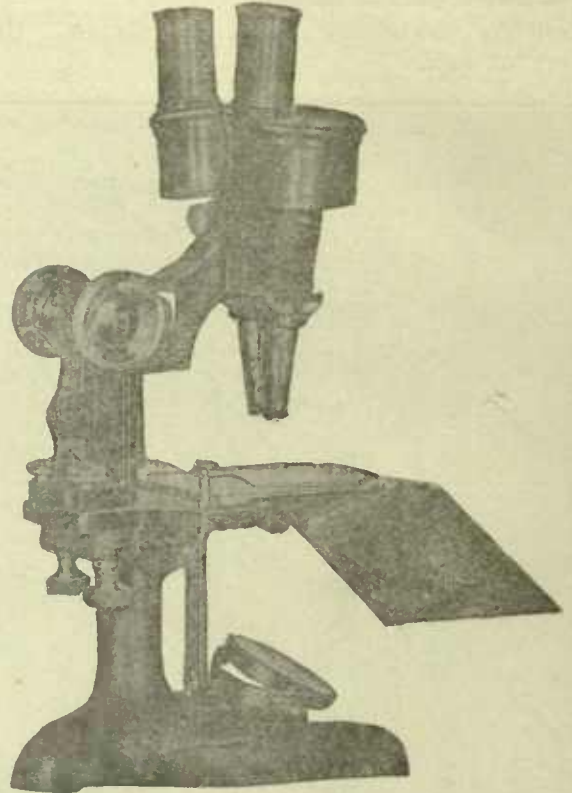


PLATE 10-B

not take into consideration the percent water of composition in the loading. Where this 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 percent and therefore negligible. An ash determination need not be calculated beyond the first place. (See ash determination under chemical testing.)

SPECKS 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 Plate 10B and a set of dissecting needles are useful. For chemical tests on small particles small test tubes made by sealing up 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 percent 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 percent potassium ferrocyanide, then in 2 percent 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 undefibred 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 defibred 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.

CHEMICAL ANALYSIS

Ash determination—A one gram (Note No. 6) 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.

NOTE No. 6—The sample of paper need not be weighed closer than 0.005 gram since a one percent variation in the moisture content will introduce an error of 0.01 gram. If the maximum error in the weight of the paper is 0.01 gram then the maximum error in the weight of the ash will be 0.01 gram for every 10 percent of ash present. Therefore in a paper containing 10 percent ash the results will be reported to the nearest tenths. If especial 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 ± 0.0005 gram and the error will be 0.0001 gram for every 10 percent of ash. The results may then be reported to the nearest hundredths. This latter result will of course be 1 percent lower than the ash results on a 1 gram sample containing 10 percent of moisture.

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 portion of it. While cooling they may be kept in a desiccator, but

this is not necessary, since the ash may be poured into a counterpoised aluminum (Note No. 7) 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 No. 7—Aluminum is recommended as being less easily broken as well as lighter, than glass.

The ash as finally obtained includes all nonvolatile and noncombustible 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 size, 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 casinates 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. glue or casein) of the coating. (Note No. 8.)

NOTE No. 8—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 percent without being loaded. This might be due to the ash in the pulp, as well as to the ash derived from water, alum and sizing materials.

Where the ash is 5 to 20 percent the paper is loaded. A list published in *Paper* for April 25, 1917, 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.

KIND OF FILLER

Burn enough paper to obtain at least 0.2 Gm. ash in a platinum or nickel crucible. Separate $\frac{1}{3}$ of the ash from the main portion; to this $\frac{1}{3}$ add 5 Cc. water and boil until well extracted; filter; add a drop of hydrochloric acid to the filtrate and then 3 Cc. 10 percent 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 $\frac{2}{3}$ portion of the ash add 1 Gm. 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 precipitate 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 to near 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 filler. 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 flocculent 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.

DETERMINATION OF 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 (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 No. 9.)

NOTE No. 9—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 two grams of paper should be sufficient.

Place the paper in a Soxhlet or in an ordinary Erlenmeyer flask fitted with a reflux condenser, cover with gasolene 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.

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.

Quantitative Rosin Determination — Sammet Method: Alcohol-ether Method.

Cut five grams of paper into strips approximately one-half inch wide and fold them into numerous small crosswise folds. Place the folded strips in a Soxhlet extractor and fill with acidulated alcohol diluted to approximately 3 percent made by adding to 900 Cc. of 95 percent alcohol, 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° Cent. 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 percent of cases a single extraction would be sufficiently accurate.

NOTE—For extracting rosin, the apparatus shown in Plate 11a 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.

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

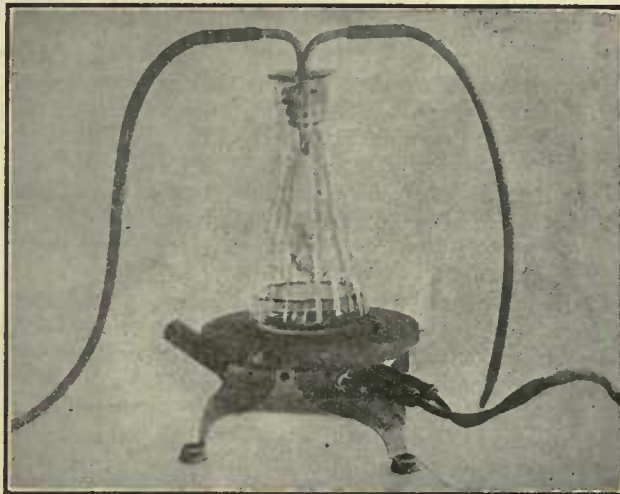


PLATE 11A

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 three to five grams of the paper which has been cut into small pieces in a Kjeldahl digestion flask, add ten grams potassium sulphate, 0.7 gram 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 grams of sodium sulphate crystals be used in this case.

Heat gently at first to prevent 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 percent solution of potassium sulphide are added.

The potassium sulphide 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 a 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 percent) 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 grams 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. tenth normal acid should be ample.) A few drops of indicator should be added to this solution. Sodium alizarin sulphonate 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 forty-five minutes and the distillate should equal 200 Cc. Titrate with tenth 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 tenth normal alkali required to neutralize the distillate, from the number of Cc. required by the blank. This difference is the number Cc. of tenth normal alkali equivalent to ammonia.

$$\text{No. Cc.} \times 0.014 = \text{Gm. nitrogen.}$$

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.

NITROGEN DETERMINATION

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.

Starch Determination—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 one gram 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.

QUANTITATIVE ANALYSIS FOR STARCH

Method of Kamm and Voorhees as described in *Paper* for August 27, 1919.

Preparation of Reagents—The usual Fehling's solution is employed.

Sol. A—69.3 Gm. of crystallized copper sulphate are dissolved in water and the solution diluted to 1000 Cc.

Sol. B—346 Gm. of Rochelle salt and 120 Gm. of sodium hydroxide are dissolved in water and the solution also diluted to 1000 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 percent solution of $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$ is used.

Acetic acid solution. A 50 percent solution of acetic acid is found convenient.

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-gram sample is cut into small pieces and placed in a 500-Cc. round-bottom flask. Two hundred Cc. of water is added, and 5 Cc. glacial acetic acid is run in, making a 2½ percent solution. The flask is connected with a reflux condenser by means of a clean rubber stopper and the contents boiled vigorously for 1½ hours. 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 percent) and boiling continued for thirty minutes, 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 one minute. 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 the 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 sharper and more distinct end-points 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.

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, fuculose and dextrin.

Example—A sample of corn-starch was dried at 105° Cent. for three hours. An 0.500-gram portion was then weighed out and hydrolyzed with about 190 Cc. of a 4 percent HCl solution during a period of thirty minutes. 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 $20 \times 0.50 = 0.050$ gram starch.

200

In an analysis of a 5-gram 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 percent of starch in the sample of paper is therefore:

$217 \times \text{Value of Fehling's solution expressed in grams of starch}$

$\frac{39}{100} \times \text{Wt. of sample of paper} = 5.5 \text{ percent.}$

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

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

39

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.

ANALYSIS FOR DEXTRINS IN PRESENCE OF BEATER STARCH

(Method of Kamm and Tendick.)

The procedure adopted consists in the removal of the surface sizing by a 45-minute leaching of the sample of paper with water at a temperature of 60° Cent. For a 5-gram sample 200 Cc. of water is used. The extract 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 Determina-

tion 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.

CHLORINE DETERMINATION

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.

FREE ACID DETERMINATION

Weigh 10 grams of the paper to be tested, tear into small pieces, place in a 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 same to water extract. Make up to 100 Cc. according to directions given on page 103 of Cohn's *Indicators and Test Papers*.

The solution is then poured into a 100 Cc. Nessler tube (long form). A similar tube is filled with 100 Cc. of distilled water to which has been added two drops of the litmus solution. 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 SO_3 .

CONCLUSIONS DRAWN FROM TESTS

For the general information of those who may undertake the laboratory testing of paper, it is especially pointed out, that the most complete series of tests, will not alone tell all the important properties of a paper. In order that correct conclusions may be drawn, it is necessary to know something of the use to which the paper is to be put. This knowledge may be obtained only by experience, and by close observation and continual testing of the material used. Conclusions should not be drawn without full information regarding both the tests results and the actual working conditions of the paper.

Complete laboratory records should be kept of all tests and in such a manner as to be always available. The accompanying 5x8 inch record cards are offered as a suggestion, though individual requirements may necessitate certain alterations. (See Plate 12.)

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