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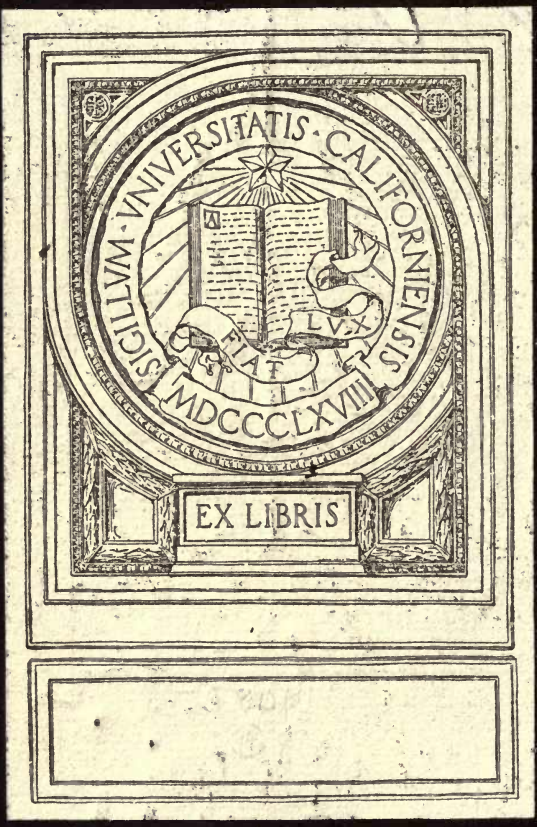
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OUTLINES OF STATIONERY TESTING.

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STATIONERY TESTING.

A Practical Manual.

BY

HENRY ALDOUS BROMLEY,

ASSISTANT EXAMINER OF PAPER TO HIS MAJESTY'S STATIONERY OFFICE, LONDON.

With 6 Plates and other Illustrations.

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PREFACE.

THE object of this little book is to present in a concise and thoroughly practical form the essential points of stationery testing. All the works with which I am acquainted on the subjects dealt with in the following pages contain a considerable mass of detail which, though no doubt of interest from the point of view of the analyst, is yet both unnecessary to the ordinary practical worker and likely to confuse by obscuring the essentials.

As far as possible all purely theoretical considerations, as well as descriptions of any but the most definitely satisfactory methods, have been eliminated. Descriptions of apparatus are only intended as a guide to the general principles of their operation. In all cases the chemical processes described are those which require the simplest possible apparatus and the least possible knowledge and experience of analytical manipulation.

I am indebted to my Publishers for the loan of several of the illustrations, and I express my thanks to them and to Mr. A. W. Leddington, of H.M. Stationery Office, who has very kindly prepared the Index.

H. A. B.

February, 1913.

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OUTLINES OF STATIONERY TESTING.

CHAPTER I.

INTRODUCTORY.

On the Use of the Balance.

On the integrity of the balance depends the value of all the quantitative work of the chemist. It should, therefore, be treated with a corresponding degree of care. As its employment is required for several operations, to be described hereafter, it may be well to give here a few hints on manipulation.

Never leave the beam resting on the knife edge. See that the plumb line hangs true, and that the pointer indicates the centre division on the scale before commencing to weigh, and, if necessary, adjust by means of the milled nut provided on the beam. Always put what you are weighing in the left-hand pan, and the weights in the right. Commence with the largest weight likely to be required ;

if too heavy substitute the next, and so on, always picking up and *replacing* each weight *with the forceps* before trying the next. Raise the beam gently and slowly, and allow the pointer to swing twice each way before deciding that no more weights are required. Make your last reading with the glass front down to avoid influencing the balance by draughts. Never put chemicals on the pans without placing them in a watch-glass first, and never lift weights off without first lowering the beam.

The following weights are usually provided—viz., 50 grms. to ·001 grm. More accurate weighing than is thus provided for is unnecessary for the operations treated of in this book.

On Certain Simple Chemical Operations.

Filtration consists in the separation of insoluble precipitates from the liquid in which they are suspended by pouring the mixture on to papers specially sold for the purpose, whereby the precipitate remains behind on the paper and the liquid passes through clear. It is well to buy as good filter paper as possible, as there are great differences in efficiency in good and cheap varieties. Moreover, the ash itself of the filter paper, which might confuse the results of quantitative work, is negligible in the better papers.

The illustrations show the proper way to fold a filter paper, the resulting cone being placed in a glass funnel, and moistened with water, so that it adheres closely to the glass.

Always pour the liquid you are filtering down a glass rod on to the paper. If the liquid shows a tendency to come through the paper turbid, it is usually permissible to boil it before filtering. This helps to keep the particles on the paper.

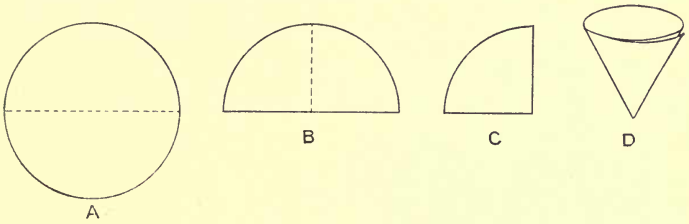


Fig. 1.

Decantation consists in pouring off a clear liquid after a sediment has been allowed to subside. It should always be done by pouring down the side of a glass rod.

Evaporation is performed by heating a liquid until any required amount has passed off in the form of vapour. Where the liquid contains substances which are volatile, or will char if exposed to more than a certain degree of heat, the evaporation must be conducted over the water bath—*i.e.*, a vessel containing water maintained at the boiling point, and of such dimensions that it will just hold the evaporating basin exposed to the steam from

the water. An ordinary tin, into the top of which the basin just fits, forms a very good bath. The solid substance left after evaporation of a liquid is called the "residue."

Ignition is conducted by heating an infusible body to redness in a crucible in the flame of the Bunsen burner.

Fusion consists in liquefying a solid body by the aid of heat. It is performed in a crucible either of porcelain or platinum. The latter is the most generally useful, but very expensive. Small quantities of substances can be fused on platinum foil.

Note on Weights and Measures.

In chemical operations the unit of weight is the gramme, shortly written grm. The unit of volume is the cubic centimetre, written c.c. The unit of length is the metre. Small measurements of length are expressed as millimetres, written mm. The millimetre is the thousandth part of the metre.

CHAPTER II.

PAPER: ITS PHYSICAL EXAMINATION.

THE physical examination of paper consists in the examination of the colour, surface or finish, substance, texture, opacity, bulk, handle, cleanliness, and freedom from defects of manufacture, its ink-bearing properties, and determination of its strength. In the following paragraphs only the usual and most practical operations are considered.

Colour.—Paper should be examined for colour lying in good diffused day light from a window at the back of the observer. The paper should not be examined in bulk, since the underlying sheets are apt to give a false impression of the depth of colour. A single sheet should be selected from the ream and laid side by side with the sample. Every manufacturer has his own ideas as to the exact shade of colour meant by the terms cream, blue, etc., and different makings of the same colour frequently vary considerably in shade. This latter point is important where more than one delivery is required for the completion of an edition of a printed book. In the case of blue papers, it is well to examine the under-side of the sheet. Where heavy blue pigments are used, considerable variation in the depth

of the colour of the top and bottom sides of the paper frequently exists. Variation in shade in different parts of the sheet should be looked for.

Surface and Finish.—The degree of finish will depend on the purpose for which the paper is required. A drawing paper may be either very rough, rough ("not" surface), or smooth (hot-pressed) as desired. A good writing or ledger paper should be highly rolled both sides or "plate glazed." In the latter case the plate mark can generally be seen along the edge. Typewriting papers are best unglazed. The surface of printing papers should not be crushed by too heavy rolling. The fingers and palm of the hand drawn across the surface of the paper gives a good idea of its nature. Looking along the surface with the eye nearly on a level with the sheet will give a measure of the light reflected from the surface, and will, at the same time, detect any fibres sticking up which may have escaped the calenders. A more scientific method of judging and comparing surface in papers consists in attaching sheets to glass plates, placing a smooth known weight on each, tilting, and noting the angle of the glass to the horizontal at which the weights begin to slide.

Substance.—This is expressed in terms of the weight of the paper and of the thickness of the sheet. The weight of the ream is usually marked on the wrapper, and individual sheets may be

weighed by means of the special quadrant paper scales sold for the purpose. The sheet is placed in a balanced pan connected with a pointer, which indicates the weight *per ream* of 480, 500, or 516 sheets respectively on a graduated quadrant. For use with very small pieces a specially sensitive instrument can be obtained showing the weight per ream in *demy* ($17\frac{1}{2}'' \times 22\frac{1}{2}''$). The pieces are cut to a certain definite size (100 mm. square) by means of a metal template.

The *thickness* of the sheet is determined by means of the automatic micrometer. The paper is placed under a plunger, which is connected by suitable gearing to a pointer travelling round the face of a dial graduated in thousandths of an inch. With practice considerable skill may be acquired in estimating thickness by feel alone.

The thickness of cards is usually expressed as so many "sheets." The numerical value of sheet thickness varies from 3 to 5 thousandths of an inch with different makers, and even in different supplies by the same maker. About 3 thousandths is the most usual. Thus an "eight-sheet" board will be about 24 thousandths of an inch in thickness.

Texture is examined by holding the paper to the light and observing the "look through." The sheet should present a uniform closeness of mesh and wire marks. "Wildness"—*i.e.*, an irregular

patchy or cloudy appearance—is objectionable. Any water mark should be clear and well defined.

Opacity is dependent upon the nature of the fibre and its length of treatment during making, on the sizing, loading, and finish of the paper. Transparency is, of course, desirable in a paper used for tracing purposes, but objectionable in a thin paper intended for printing both sides. The degree of opacity of two or more papers may be roughly compared by holding them to the light side by side with a pencil at the same distance behind each, and judging the difference in depth of the shadow seen through each. A more scientific way is by observing the relative thickness required to cut off the rays of a source of light of known power. A piece of black opaque paper is pasted on to a sheet of glass, which is then secured vertically at a distance of 1 foot from a source of light of known candle power. A number of pieces of the paper under examination are now placed together behind the glass and between the latter and the light, until on looking through the screen the light is found to be obscured. The total thickness of the sheets is then determined with a micrometer, and the result expressed in terms of the thickest paper as unity—*e.g.*, if two sheets of a notepaper of ·12 mm. thickness, and twelve sheets of cigarette paper of ·01 mm. thickness are required respectively to produce obliteration, then their

relative opacities are as (2 × ·12) to (12 × ·01)—that is, as 2 : 1.

General Considerations.—Pure sulphite wood pulp usually makes a transparent paper. Conversely an *opaque* paper known to contain sulphite wood is probably heavily loaded or contains "mechanical" pulp. Linen and straw produce similar degrees of transparency. A *cheap* "loan" paper may be suspected of containing straw. An excessively transparent paper may be correspondingly low in *strength*. Too great density suggests excessive mineral matter present.

Bulk is the ratio of *fibre* volume of paper to the *total* volume (total volume representing, of course, volume of fibre *plus* volume of air spaces, either free or filled with sizing and loading materials). A blotting paper depends for its absorbency on its bulk—that is, in the absence of loading, on its large proportion of air space. Comparative bulking properties in papers may be determined as follows :—A piece of the paper of known area is weighed, and its thickness determined with the micrometer. The product of the thickness and the area gives the volume of the paper—*i.e.*, volume of fibre *plus* air space. The paper is now reduced to ash on platinum foil, and the ash is weighed. The figure thus obtained is deducted from the original weight of the paper, when the difference, of course, represents weight of fibre. The weight of fibre

divided by 1·5 (average specific gravity of cellulose) gives the volume of fibre present, according to the formula $\frac{W}{s.g.} = V$. Dividing the figure representing volume of fibre by that of volume of the original paper, we get the volume of fibre as compared with unit volume of paper—*i.e.*, the Bulk.

Handle.—This term expresses the impression conveyed to the observer on handling the sheet. It takes cognisance of the feel of the paper, the substance, and the "rattle"—*i.e.*, the sound heard when the sheet is shaken sharply and rapidly between the hands. For instance, an all-linen paper, as a bank note, is expected to impart a peculiar soapy feel, and to give a very distinctive "rattle." Considerable practice is necessary to acquire the ability to estimate the weight of a paper merely by handling, as well as to give facility in recognising the characteristic "handle" of special papers.

Cleanliness, etc.—The ream should be looked through carefully for specks, adventitious substances, such as particles of metal, india-rubber, and grit, which frequently get into paper, soiled sheets, finger marking, fibre lumps, pinholes, rosin spots, patches of raw pigment, etc. The presence of the above indicate that the ream has not been properly "retreed." Cockling of the paper is sometimes

found, and thin patches, especially down the edges, may occasionally be seen.

Ink-bearing Properties.—The degree of sizing varies, of course, with the purpose for which the paper is intended. A duplicating paper should, as a rule, be "soft" sized—*i.e.*, while ink marks should not spread as they would on blotting paper, still the paper should be sufficiently pervious to allow the ink to be absorbed, and thus dry quickly. On the other hand, a fine ledger paper should be extremely well sized, and ink, allowed to dry naturally on the paper, should not come through to the under side. A rough idea of the ink-bearing quality of a paper may be obtained by pressing the tongue against a sheet for a few seconds. If the paper is poorly sized, the outline of the tongue will be plainly visible on looking through the sheet at the light, and that portion of the sheet which has been moistened will be seen to be flabby, saturated, and transparent. A good estimation of the degree of sizing of a paper may be obtained by writing thickly upon it with ink, preferably using a quill pen to avoid injuring the surface of the paper, and observing the time taken for the ink to make its appearance on the under surface. When the ink has dried, the paper may be torn across the pen marks, and the depth to which the ink has penetrated noted. Some examiners test sizing by ruling lines on one side of the paper by means of a

pen dipped in a solution of perchloride of iron, removing excess with blotting paper, reversing the paper, and ruling similar lines at right angles to the first with a solution of tannic acid, removing excess as before. The points of intersection of the lines are now watched, and the time noted for the appearance of the black marks caused by the interaction of the iron and tannin. Another means of comparison is afforded by simultaneously dropping on the papers a solution of aniline blue in alcohol. The paper through which the dye appears soonest is least sized.

Strength.—The determination of the strength of a paper—*i.e.*, its resistance to a breaking or rupturing stress—is rightly considered to be one of the best means we possess of gauging its quality, since those fibres whose presence in a paper is most esteemed for other reasons are found to possess to a proportionately high degree the power of imparting strength to the finished product. Thus cotton and linen whose fibres possess great "felting" or interlacing properties both impart a considerable degree of strength to paper, and at the same time bestow the lasting and resistant characteristics of rag papers generally. On the other hand, papers made from the weakest fibres, as "mechanical" wood, are the least desirable from the point of view of permanency. At the same time, too great stress should not be laid on mere strength, for sulphite

wood pulp under-bleached and well sized is capable of producing a very strong and tough paper, which yet, by reason of the chemical nature of the material itself, is not desirable for documents of permanent value. In many instances, of course, strength is the *principal* requirement in a paper, as in wrappings, and in these cases quality of the ingredients is negligible, except in so far as it contributes to such strength.

Much ingenuity has been expended on designing machines for testing the strength of paper. The best of these is the Schopper (Leunig) illustrated below, which is the recognised standard machine of the German Paper Testing Institute at Charlottenburg. This machine registers the absolute tensile strength of a strip of definite dimensions.

In using the Schopper machine four strips of paper, two from each direction of the sheet, are cut by means of the guillotine provided. The guillotine cuts the paper of a fixed and definite width of 15 mm. (five-eighths of an inch), and when arranged for use the distance between the clamps E_1 and E_2 is also a fixed and definite amount—viz., 180 mm. (7 inches). The arm A with its weight is placed vertically, so that the pointer indicates zero on the scale B, and one of the strips of paper is firmly secured between the clamps E_1 and E_2, great care being taken that the strip lies evenly. The handle is now evenly and slowly turned, when

the clamp E_2 is drawn slowly downwards, exerting an increasing strain on the strip, and at the same time the arm A with its weight travels gradually

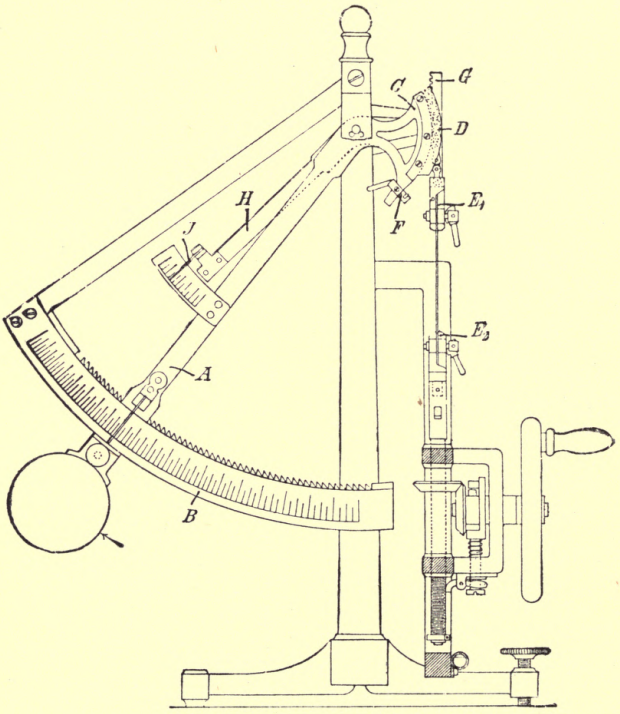


Fig. 2.

outwards along the scale. At the limit of its strength the strip fractures sharply, and the arm A is automatically arrested and retained in position. The breaking weight can now be read off on the scale.

At the same time another pointer J, attached to the smaller arm H, registers automatically on a smaller scale the "stretch" of the strip—*i.e.*, its elongation before breaking. This stretch is read off as a per cent. of the length of the strip. Both breaking strain and stretch vary with the direction in the sheet from which the strips are cut, and it is necessary to make at least four determinations, and to calculate the mean from the figures obtained. Stretch should be proportionate to breaking strain —*i.e.*, a paper giving a high average breaking figure is expected to show a proportionately high degree of stretch. Apart from machine direction, such factors as the humidity of the atmosphere and the rate of revolution of the apparatus have a practical effect on the figures obtained.

For all ordinary *comparative* determinations of strength the Mullen bursting machine is quite satisfactory. The paper is clamped over one end of a cylinder filled with glycerine, and having a rubber diaphragm between the glycerine and the paper. On actuating the handle the column of fluid is pressed strongly against the rubber, forcing it in turn against the paper. At the limit of resistance of the paper the latter bursts, and the strain is read off on the dial of a pressure gauge in lbs. per square inch. It is necessary to actuate this machine at a constant rate to get an accurate comparison. A high rate of revolution will force the observed

figure up much above the true value, and *vice versâ*. The figure given on the Mullen is about $2\frac{1}{2}$ times that of the Leunig.

The only other machine that will be mentioned here is the Carrington, which is most suitable for

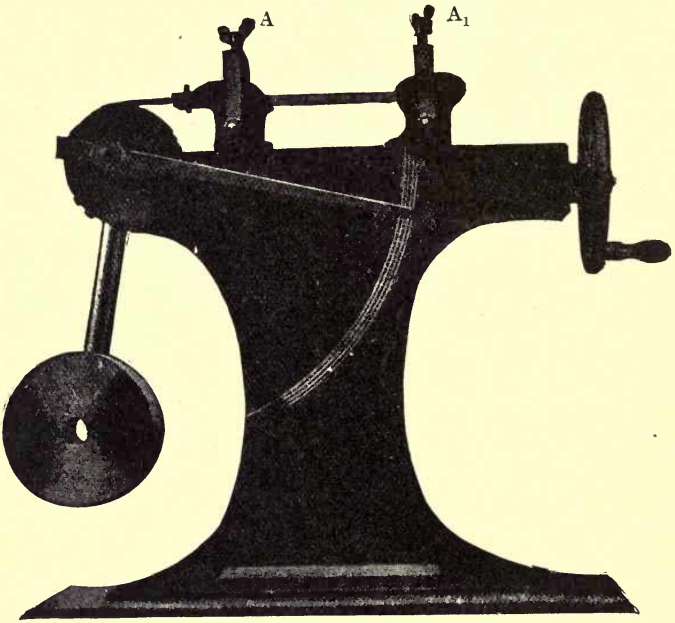


Fig. 3.

testing heavy papers, such as browns. In the illustration A and A_1 represent the clips between which the paper is fixed. On revolving the handle the clip A_1 is forced along the bed of the machine in the direction of the handle, and the strain on

the strip causes the weight to rise, carrying with it the pointer. When the strip breaks the weight and pointer remain in position, and the breaking strain in lbs. can be read off on the scale. In the Government Stationery Office the strips used on this machine are cut to the dimensions of a brass template $7'' \times 2''$.

Breaking length is of purely theoretical interest, and will not be considered here.

Rough tests of the resistance of a paper to wear and tear may be made by observing the resistance to tearing in each direction of the sheet ; by crumpling a small piece of paper into a ball, opening out, crumpling again, and repeating the operation until a hole appears, observing the number of operations necessary to produce the hole ; and by repeatedly folding pieces of the paper in the same place, and noting the number of times the folding can be performed before the paper easily pulls apart at the crease.

CHAPTER III.

PAPER: ITS MICROSCOPICAL EXAMINATION.

THE object of the microscopical examination of paper is the determination and estimation of its fibre constituents. The following are the fibres most commonly occurring in the composition of paper, arranged in groups according to their colour reactions with staining materials :—

(1) Cotton, Linen, Hemp, Manila.
(2) Wood (chemically treated), Straw, Esparto.
(3) " Mechanical " Wood, Jute.

The following staining reagents are all that is necessary to an ordinary examination. On the whole, the writer recommends No. 2 for all-round work :—

(1) Potassium iodide, . . 2 grms.
Iodine, ... 1·5 grms.
Glycerine, . . . 2 c.c.
Water, ... 20 c.c.

(2) Zinc chloride, . . 20 grms.
Potassium iodide, . . 2·1 grms.
Iodine, ... ·1 grm.
Water, ... 5 c.c.

The following table shows the colours imparted by the above reagents :—

Fibres.	*Iodine Solution.*	*Zinc-Chlor-Iodine.*
Linen, Cotton, Hemp.	Brown.	Faint to strong wine-red.
Manila.	Brown; some yellowish.	Wine red; some yellowish.
Straw, Esparto.	Greyish-brown.	Blue to violet.
Wood (chemical).	Colourless.	,, ,,
Wood (mechanical), Jute.	Yellow.	Yellow.

Specimens for microscopical examination are prepared as follows :—

Unsized Papers.—A small portion is torn up into pieces and boiled in a beaker with a little water containing a trace of ammonia.

Sized Papers.—Are boiled in a *weak* aqueous solution of caustic soda to dissolve out the size.

The sample is then washed with water and reduced to a pulp by shaking vigorously in a test tube with water, holding the thumb over the mouth of the tube.

The use of beads or shot in this operation is unnecessary.

A small sample of the pulp is then taken out and placed on a glass slide, a drop or so of the staining reagent is placed on the pulp by means of a glass rod, and the fibres teased out with a couple of teasing needles (long pins will do quite well).

The cover is now put over the specimen, any

liquid which may have escaped from under the edges is removed with blotting paper, and the slide is transferred to the microscope stage, and examined under the $\frac{1}{4}$-inch objective. A micrometer eyepiece is useful for estimating the comparative lengths of fibres.

The following is a description of the microscopical characteristics of paper-making fibres.

Cotton.—The fibres are long, opaque, flattened tubes characteristically twisted upon themselves, the central canal is large, and the side walls correspondingly thin. The ends are naturally blunt, but may be found beaten out into tufts. The cell walls are often characteristically marked in a curious lattice-like way. Plate I., No. 2.

Linen (flax).—The fibres are long and tubular, but the side walls being thick and the central canal very small, the fibre preserves its stiffened rounded appearance. The walls exhibit characteristic bamboo-like knots at intervals. The ends are naturally pointed, but in beaten pulp may be considerably frayed out. Plate I., No. 1, and Plate II., No. 3.

Hemp.—These fibres are exceedingly difficult of distinction from linen. We may say that, as a rule, hemp exhibits bundles of fibres, showing striæ parallel to the length of the fibre, and fissures, also adherent hairs. The ends are large and flattened (the latter not often seen), and the central

canal, as a rule, almost obliterated. Plate II., No. 4.

Manila has a larger diameter than hemp. The central canal is large and easily seen. Flat cuticular cells, rather like those of straw, are to be seen, and the fibres exhibit nodes. Cellular structures of a curious " brick-wall " appearance are sometimes seen, and when found serve to identify the fibre. Plate III., No. 5.

Jute strongly resembles manila. One striking characteristic is the extreme irregularity and variable nature of the cell wall, causing the central canal to be broad in places, and exceedingly narrow in others. The fibres which, like hemp, are often found in bundles, are in many cases covered with encrusting material. Their yellow colour serves to distinguish them from hemp. Plate VI., No. 11.

Chemical Wood shows transparent ribbons often twisted like cotton, and frequently exhibiting latticed striping. The fibres often show nodes or knots like linen. *Pine wood* may be distinguished by the dotted vessels to be seen in the fibre : thus ⊙. Plate III., No. 6.

Poplar cells always present a characteristic honeycomb appearance, well shown in the plate. Plate IV., No. 7.

Esparto fibres are short, smooth, and tubular, with finely-pointed ends, and a small central canal. The serrated cuticular cells with saw-like edges,

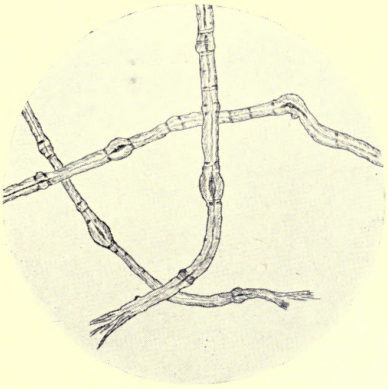
and the characteristic hairs, looking like little teeth, serve to distinguish this fibre. These hairs are *never* seen in straw. Plate V., No. 9.

Straw.—The fibres are shorter and smoother than those of esparto, and have more finely-pointed ends. They present a jointed appearance at intervals. Serrated cells are present, as in esparto, together with large characteristic oval cells, which are usually found in pairs, and are never seen with esparto. Ring cells and spirals are occasionally found. Plate IV., No. 8.

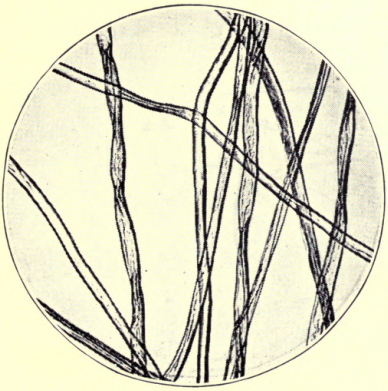
Mechanical wood fibres present opaque structurless bundles of tangled ribbons, showing pine cells and medullary rays—*i.e.*, groups of cells running perpendicularly to the wood cells. Incrusting matters are plentiful. Plate V., No. 10.

Wool fibres are used exclusively for grey filter papers. Their microscopical appearance is given in Plate VI., No. 12, and is unmistakable.

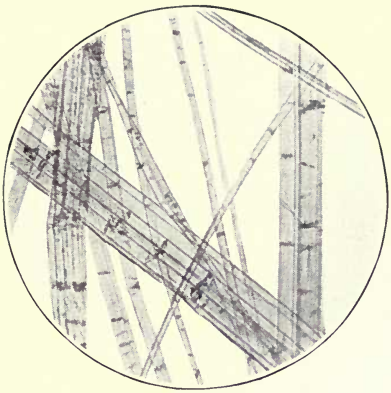
Percentage Estimation of Fibres.—This is a difficult matter without very considerable practice. A good way is to count the fibres in a field of the microscope, using the $\frac{1}{4}$-inch objective, and starting from one corner of the slide, and to repeat the process in several other portions. The percentages of the particular fibres under estimation can be calculated in each field, and an average taken of the results.



No. 1.—Linen.

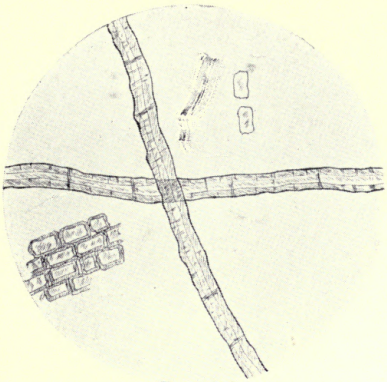


No. 2.—Cotton.

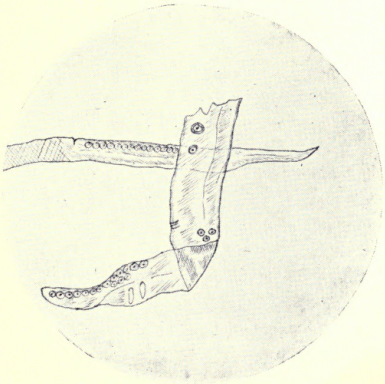


No. 3.—Raw Flax.

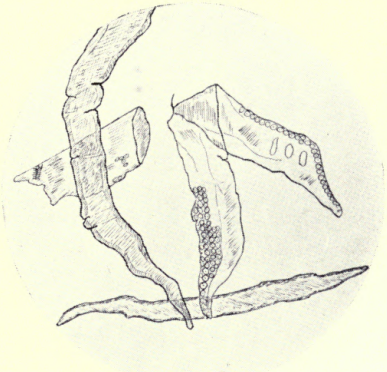
No. 4.—Hemp.



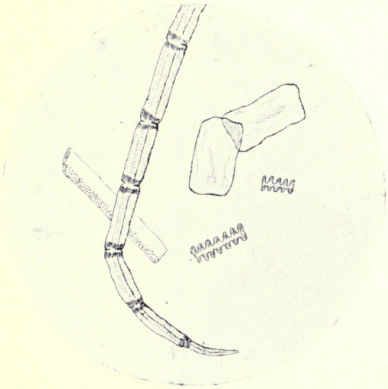
No. 5.—Manila.



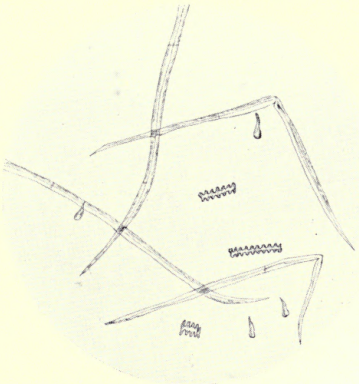
No. 6.—Pine Wood.



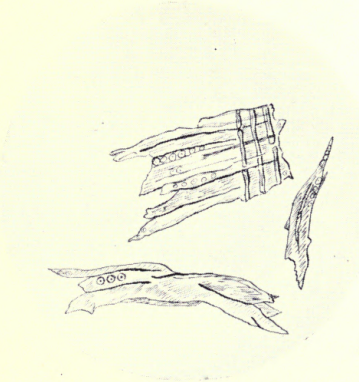
No. 7.—Poplar Wood.
Showing Vessels.



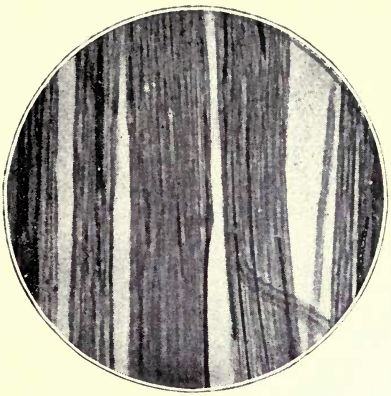
No. 8.—Straw (highly magnified).
Showing Oval and Serrated Cells.



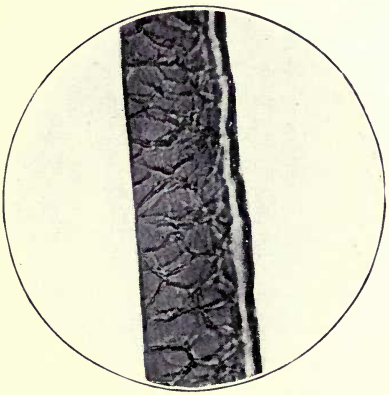
No. 9.—Esparto
Showing Hairs and Serrated Cells.



No. 10.—Mechanical Wood.
Showing Pores and Medullary Rays.



No. 11.—Jute.
Showing Bundles.



No. 12.—Wool (highly magnified).

Table of Approximate Lengths of Paper-making Fibres in Millimetres.

Linen,	25-30
Cotton,	20-40
Hemp,	20-25
Manila,	4-6
Jute,	2
Esparto,	5
Straw,	0·2

CHAPTER IV.

PAPER: ITS CHEMICAL EXAMINATION.

THE chemical examination of paper consists in the determination of the nature and amount of the mineral matter added in the process of manufacture for the purpose of weighting (loading) or surfacing the paper; of the nature of the sizing materials used and the quantity present; of the nature of any colouring matter that may be present; together with tests for the presence of those fibres which give specific chemical reactions, and for the presence (or absence) of certain undesirable constituents.

Loading.

For all practical purposes the estimation of the loading present in paper may be considered to mean the estimation of the *ash*. The presence of an amount of ash equal to from 2 to 2·5 per cent. is inevitable from the nature of the materials used in the manufacture and sizing. Anything above this figure may, perhaps, be taken as indicating the deliberate addition of loading materials.

The estimation of the ash is conducted by burning a weighed quantity of paper on a piece of platinum foil and transferring the ash, which in uncoloured

papers should be nearly white, to a tared watch-glass, and weighing. From the weight of ash found the percentage in the original weight of paper is easily calculated. Where a hasty examination is necessary and the paper is well loaded, the operation of burning may be conducted by fixing the paper in small pieces on the ends of a twisted platinum wire, as one holds toast on a fork. The ash will usually hang together in this way very well.

Blottings, copyings, and tissues should be practically free from ash. In coloured papers, more especially blues and buffs, the colouring matter itself frequently contributes to the weight of ash, and such colouring matter must be considered as loading in itself.

Since loading undoubtedly lessens the resistance of paper to wear and tear, the maximum amount permissible in any *uncoated* paper may be taken as 10 per cent.

The following mineral substances are those most frequently added to paper :—

1. *China clay* (hydrated silicate of aluminium) is light, absorbent, and largely added to "news" and cheap printings generally. Imitation art papers are usually heavily loaded with this substance.

2. *Pearl hardening* (sulphate of calcium) is added to good quality writings to give brilliancy.

3. *Barytes—blanc fixe* (sulphate of barium).—Much used as a surface in coated papers.

4. *Agalite* (magnesium silicate).—Uncommon. Imparts a peculiar soapy feel to a paper.

5. *Satin White* (alumina with precipitated calcium sulphate).—Used for surfacing.

The identification of the added mineral matter is conducted as follows :—

1. Reduce to ash a good quantity of paper on platinum foil. Place a small portion in a test tube and add fairly dilute hydrochloric acid. If the ash is entirely soluble (or practically so) on warming, probably the loading material consists of calcium sulphate only. In this case the addition of a solution of barium chloride will give a white precipitate ; and the analysis need not be conducted further. If none, *or only a portion*, of the ash is soluble in the acid, proceed as follows :—

2. Mix the ash with three or four times its weight of " fusion mixture " (sodium and potassium carbonates) in a crucible, and fuse the mixture for half an hour at a good red heat, finally employing the blowpipe for several minutes. Cool the fused mass and extract with hot water. Filter.

The filtrate may now contain	The residue may comprise
Sodium and potassium sulphates and silicates.	Calcium, barium, magnesium, and aluminium carbonates and hydroxides.

3. (*a*) *The Filtrate* is acidified with hydrochloric

acid (dilute) until any effervescence subsides. Barium chloride solution is then added, when a heavy white precipitate indicates the presence in the original ash of a *sulphate*.

(*b*) *The Filtrate*.—A further portion is acidified with hydrochloric acid and evaporated to dryness. Hot water and more acid are added in a test tube, when a gelatinous flakey precipitate indicates the presence in the original ash of a *silicate*.

4. *The Residue*.—Dissolve in dilute hydrochloric acid, filter, and evaporate the filtrate to a small bulk.

(*a*) To this filtrate add ammonium chloride solution and strong ammonia in *slight* excess, when a white gelatinous precipitate indicates the presence in the original ash of *aluminium*. A mere cloud will be due to the alum in the *sizing*, and may be disregarded.

(*b*) Filter the solution from (*a*), discard the precipitate, and to the filtrate add a solution of potassium chromate, when a lemon-yellow precipitate indicates the presence in the original ash of *barium*.

(*c*) Filter the solution from (*b*), discard the precipitate, and to the filtrate add a solution of ammonium carbonate, when a white precipitate indicates the presence in the original ash of *calcium*.

(*d*) Filter the solution from (*c*), discard the precipitate, and to the filtrate add more ammonia solution (unless it still smells strongly), followed by a solution of sodium phosphate. Shake vigorously, when a crystalline white precipitate re-

dissolved on addition of an acid indicates the presence in the original ash of *magnesium*.

Note.—If both calcium sulphate and alumina are found, *satin white* has been used.

Examination of the Sizing.

The usual sizing agents employed in paper-making are :—

1. *Gelatine* (glue) : in hand-made papers, good writings and drawings, and as an adhesive in coated papers.

2. *Rosin.*—Alone in printings and cheap writings, and as an auxiliary to gelatine.

3. *Casein.*—Mostly as an adhesive in coated papers.

4. *Starch.*—And as a dressing.

Gelatine is detected as follows :—

A strip of paper is torn into shreds and boiled with water in a beaker. The liquid extract is transferred to a test tube, cooled, and a few drops of a weak solution of tannic acid added. A yellowish flocculent precipitate (often not appearing immediately) indicates gelatine. If the contents of the test tube be now boiled the precipitate should coagulate into a mass and fall to the bottom.

Note.—Starch is also precipitated by this method. The appearance of the precipitate (whitish with starch, yellowish with gelatine) usually serves to discriminate between the two, but where any doubt exists a weak solution of iodine should be applied

to the paper. If a decided blue colouration is given a further quantity of paper must be boiled, the extract transferred to a test tube, allowed to cool, and a little ammonium chloride followed by iodine solution added. The blue liquid is now filtered and the filtrate boiled and allowed to cool, when tannic acid solution is added as before. A flocculent precipitate now indicates gelatine only.

For the detection of small quantities of gelatine in the presence of starch, the writer employs the following method, which depends on the fact that starch forms an insoluble compound with barium :—

The paper is first boiled in *distilled* water, and to the aqueous extract is added baryta water. The white precipitate formed is then filtered off. A solution of sodium sulphate is added to the filtrate to precipitate the excess of barium, the liquid is boiled and again filtered, when the addition of tannic acid indicates the presence (or absence) of gelatine.

For the *quantitative* estimation of gelatine, reference should be made to the larger text-books. The estimation is seldom or never required, inspection of the tannic acid precipitate being a sufficient guide for practical purposes to the quantity present.

Rosin.—A strip of the paper under examination is torn into shreds and placed in a test tube with sufficient spirits of wine * to cover the paper. The whole is now heated *in a beaker of hot water* until

* Methylated spirits must *not* be used.

the spirit has boiled for some time, fresh spirit being added as evaporation goes on. A further test tube is filled with cold water, and the spiritous extraction, previously acidified by the addition of a drop of acetic acid, is poured into it. In the presence of rosin a more or less distinct white cloud is formed, which on inversion of the test tube becomes distributed throughout the bulk of the water. The cloud is most distinctly seen against a dark background and in a strong light.

Note.—Hand-made papers in which no rosin has been used will occasionally give this reaction. This is due to the fact that in these papers *soap* is frequently added in course of manufacture, to give "feel" to the paper. In this case, however, the presence of a fair degree of gelatine sizing in a paper serves to indicate that the reaction is not due to rosin. In any case of doubt it may be necessary to apply the test for rosin given on page 58.

A simple test for rosin consists in letting a drop or two of ether fall on the paper. On spontaneous evaporation of the ether the rosin separates out as a brownish ring.

Probably all the methods for the *quantitative* estimation of rosin are more or less untrustworthy. A rough and ready approximation may be made by the repeated extraction of a known weight of paper with absolute alcohol as described above, and a determination of the loss of weight thereby.

Casein.—In uncoated papers in the *absence* of a precipitate with tannic acid (absence of gelatine), casein may be detected by dipping the paper in Millon's reagent, and warming, when a pink colouration indicates the presence of this sizing agent.

Millon's reagent is prepared by treating a little mercury with its own weight of fuming nitric acid of specific gravity 1·4, and gently heating till the metal is dissolved. The solution is diluted with twice its volume of water, allowed to stand until a deposit forms, and the supernatant liquid poured off.

In the case of coated papers the paper must be boiled with a solution of carbonate of soda, the solution filtered, and the casein re-precipitated from the filtrate with dilute acetic acid. The precipitate, after again filtering, is warmed with a mixture of glacial acetic acid and strong sulphuric acid in the proportion of two volumes to one, when a reddish violet colour shows the presence of casein.

Starch.—Although it is convenient to consider starch under the heading of "sizing," it is really to be considered in the nature of an *adulterant.* Starch, on account of its liability to decomposition, should be absent from all papers which are required for the purpose of permanent record. It is admissible in *small quantities* in paper otherwise soft-sized or unsized, as duplicatings or blottings.

Starch is detected by the well-known blue colouration produced with iodine. A *weak* solution of

iodine in potassium iodide is dropped on the paper, when the intensity of the colour produced is a guide to the quantity of starch present, 5 per cent. producing an intense blue-black.

Starch may be estimated quantitatively by boiling a known weight of paper for a few minutes with absolute alcohol acidulated slightly with hydrochloric acid. This removes rosin. The paper is then washed with spirit, dried, and weighed again. It is next boiled with a mixture of equal volumes of alcohol and water also acidulated, until the paper no longer gives the blue colour with iodine solution, washed again, dried, and weighed. The difference between the two last weighings gives the amount of starch present in the weight of paper taken.

Identification of the Colouring Matter.

Blue Papers.—The pigments used may be Ultramarine, Prussian blue, or Smalts. The cheapest blue papers, as blue wrappings, are usually dyed.

Place a drop of hydrochloric acid and a drop of caustic soda, about an inch apart, on the sheet of paper under examination.

(*a*) If the paper is decolourised by the *acid*, the colour is probably ultramarine. Confirm by warming a little of the sample in a test tube with hydrochloric acid and introducing a lead acetate test paper into the mouth of the tube, when the paper will blacken.

(*b*) If the paper is decolourised by the *alkali*, Prussian blue is probably present. Confirm by boiling more paper with weak caustic soda solution until decolourised, and acidifying the whole with hydrochloric acid, when the blue colour will be partly reformed.

(*c*) If the colour is modified by *both* the acid and alkali, it is probably a mixture of ultramarine and Prussian blue.

(*d*) If the colour is *changed* entirely by either acid or alkali, it is a dye ; but note that dyes in general are not *necessarily* unstable to these reagents. They are best tested for by extraction of the paper with alcohol, as described under yellow papers (*infra*).

(*e*) If the colour is unaltered by acid and alkali, and at the same time the ash of the paper is blue, it is probably due to smalts. Smalts is only used in the most expensive papers.

Yellow Papers may be coloured by lead chromate (chrome yellow), or by Auramine, Metanil yellow, and other coal-tar dyes.

(1) Place a strip of paper in spirits of wine with the addition of a drop of ammonia, and warm over the water bath. If the colour is a dye, the spirit will extract it, and itself become coloured.

(2) Reduce some of the paper to ash, when, if the latter is coloured yellow, it suggests chrome yellow. Confirm by heating some of the paper in

a test tube with a little strong hydrochloric acid, and a drop or two of alcohol, when the liquid will turn green in colour from the reduction of the lead chromate.

Buff Papers.—The colour is usually due to oxide of iron, but chrome yellow may be present.

Dissolve the coloured ash in dilute hydrochloric acid with one drop of nitric acid added. Add to the solution a little water in a test tube, and finally a few drops of a solution of yellow prussiate of potash, when a deep blue colouration reveals the presence of iron. Chrome yellow has already been considered.

Brown and Red Papers.—Brown and red writing papers are usually dyed ; but brown wrappings may contain iron, or if dark brown, manganese. To test for the latter, fuse the ash on platinum foil with a little solid caustic potash and chlorate of potash, when a green mass soluble in water yielding a green solution turning red on boiling indicates manganese. If the ash is red, test for iron as above.

Green Papers.—These are usually dyed. A coloured ash may be due to a yellow pigment mixed with a blue one, such as Chrome yellow with Prussian blue.* Confirm the latter mixture by treating the paper with a drop of hydrochloric acid, when the yellow will be decolourised, the blue left.

Black Papers.—These may be coloured with iron

* If Prussian blue has been used the ash will be yellowish-brown.

and tannin, in which case the colour will be discharged and a reddish solution contained on warming some of the paper with hydrochloric acid.

The Chemical Reactions of Certain Fibres.

Straw and Esparto.—The presence of either or both of these fibres is detected by warming the paper in a basin with a 10 per cent. solution of aniline sulphate, which imparts a rose-pink or red colour to the paper, according to the amount of the fibre present. Esparto gives a considerably more pronounced colour than straw.

Mechanical Wood is detected by moistening the torn edge of a strip of the paper with a solution of phloroglucinol made by dissolving 5 grms. of the salt in 125 c.c. of hydrochloric acid diluted with an equal volume of water. In the presence of this reagent papers containing mechanical wood fibre are coloured a more or less intense red, according to the amount of fibre present. A small amount of colouration may be due to the inclusion, accidentally or otherwise, of "broken" paper. It should be remembered when performing this test that certain dyes (notably Metanil yellow) are also coloured red by hydrochloric acid *alone*. Where any doubt exists, the paper should be first tested with a drop of the acid. Mechanical wood is inadmissible in any papers but those used for the most temporary purposes, such as News, etc. An ad-

mixture (up to 15 per cent.) may be allowed in coloured printings and buff papers only.

Note.—Isolated fibres are frequently coloured by phloroglucinol in papers free from mechanical wood. Such fibres are merely under-bleached.

Detection of Certain Impurities in Paper.

Free acid in paper is very destructive to the fibre. It is tested for by extracting a large quantity of the paper with hot distilled water for some time, evaporating the filtered extraction to the smallest possible bulk in a white basin, cooling, and adding a drop or two of methyl orange. In the presence of free acid the yellow colour of the methyl orange acquires a reddish tinge.

Chlorides.—The presence of chlorides in any appreciable quantity in finished paper suggests incomplete removal of the products of the bleaching process, and is objectionable. Extract the paper with *distilled* water, adding one drop of nitric acid and then a few drops of silver nitrate solution. A white precipitate indicates the presence of chlorides. A mere cloud may, as a rule, be disregarded.

CHAPTER V.

THE CHARACTERISTICS AND REQUIREMENTS OF SPECIAL PAPERS.

"Hand" or "Mould"-made Papers.

Distinction of "hand"-made from "machine"-made.—1. A "hand"-made paper tears in an almost equally straight line in each direction. A "machine"-made paper tears much more irregularly in its cross direction than in the direction of its length.

2. Machine-made papers, as a rule, show a considerable difference between their breaking weights in each direction of the sheet, while "hand"-made show little.

Distinction of "hand"-made from "mould"-made.—This is a difficult matter. Mould-made papers are said to have a wider and thinner "deckle" edge in their web direction than in their cross direction. There is also usually more difference in their strength in each direction than is the case with "hand"-made papers.

General Specification.—As the papers of this class are very high-priced, a high degree of cleanliness and perfection is to be expected. They should show an all rag (linen and cotton only) composition, and be "tub" (gelatine) sized.

Drawings should be free from objectionable smell.

Loans should be of great strength, and bear ink after erasure.

Blue papers should owe their colour to smalts.

Best Writings.

General Specification.—The reams should be well "retreed." Paper for permanent documents should be all rag in composition, and give not more than 3 per cent. ash. Gelatine alone should be used for sizing.

Ledger papers should show a high average breaking strain (15 to 20 lbs.), and a corresponding "stretch" (3 to 3·5 per cent.). They must stand ink after erasure, and should be air-dried and well rolled. Ultramarine is permissible for blue papers.

Ordinary Writings.

Should be well rolled and tub-sized. Not more than 2 per cent. rosin sizing nor more than 50 per cent. wood cellulose should be present. Mechanical wood pulp should not be used, and the loading should not exceed 10 per cent. Starch should be absent.

Blottings.

The best blottings may be expected to be all rag, cotton largely predominating. They should be free

from loading. Coloured and poor white blottings frequently contain wood cellulose, and starch is often present in all grades. A certain compactness and smoothness of surface is to be looked for. Hairiness is objectionable.

Blottings are examined as follows :—

1. By the " Mounting test."

2. For behaviour with ink, and for the character of the zones left by blots.

Mounting Test.—Two strips of about 3 by $\frac{3}{4}$ inches are cut, one from each direction of the sheet. A pencil line is drawn across each strip close to one end, and the papers are suspended in a glass of water with the lines on a level with the surface of the liquid for exactly one minute. The strips are then quickly removed, and the height to which the water has risen in each case marked with further pencil lines. The distance between the lines on each strip is now measured with a millimetre rule, and the mean of the two readings calculated. A good white blotting should average 28 to 30 mm. Coloured specimens may show considerably below this figure. If the absorbency is poor, the paper should be examined under the microscope for the presence of wood fibre.

Behaviour with Ink.—The following tests are comparative only, and a definite standard should first be set up from a thin blotting of known good quality :—

1. A small burette graduated in tenths of a c.c. is filled with Stevens' ink. A piece of the paper under examination is supported across the open top of a large glass beaker, and placed under the tap of the burette. Drops of ink, each corresponding to $\frac{1}{10}$ c.c., are now dropped on to the paper at intervals of half a minute (more or less, according to the rate of absorption), until the ink commences to run through and appear on the under side of the paper. The amount of ink used is noted, the area of the blot calculated, and the figures obtained compared with those given by the standard paper. The rate at which the drops are taken up is also a useful guide, being, of course, directly proportional to the quality of the paper.

2. The blot produced by the above method is allowed to dry. On examination it will be found to consist of two parts, an inner darker portion of more or less irregularly defined circular shape, and an outer zone of lighter shade, due to the iron, phenol, etc., of the ink. The area of each of the two portions is to be determined, and the proportion of each to the whole blot calculated. Of the two areas, the outer is quite unabsorbent, while the inner may or may not have retained some bibulous properties. Manufacturers making a speciality of blotting papers endeavour to reduce the proportionate area of the outer zone to a minimum. More drops from the burette are allowed to fall on this

inner area after drying, and the behaviour of the paper noted as before. A good blotting paper will absorb quite a respectable amount of ink in its inner zone.

Art Papers.

The surface coating must be sufficiently soft to allow of proper absorption of printing ink, and yet hard enough to withstand any tendency to come away from the paper itself. Press the moistened thumb on to the surface, and observe how much coating comes away. Gum on a piece of paper, and allow to dry. Then attempt to separate the two, and observe the behaviour of the coating as to its tendency to "lift." Enamelled papers should be rosin-sized, but not hard-sized. The use of china clay for the coating goes with cheapness.

Wrapping Papers.

Brown wrappings are either (1) "Air" dried or (2) "Cylinder" (machine) dried. The former are required for papers subjected to excessively severe wear and tear. To distinguish "air"-dried from "cylinder"-dried papers, rub two portions of the sheet together for a short time. The cylinder-dried paper will soon perforate, while the air-dried paper should rub down to a rough leathery consistency long before holes appear.

Old hemp rope and waste are largely used in the manufacture of brown papers, and lend a high

degree of strength to the finished product. Undoubtedly the strongest browns are the "Kraft" papers. These are made entirely from sulphate wood pulp, and, until recently, were only to be obtained from abroad. Almost any rubbish may be found in the commonest papers.

The following table gives the breaking strain and stretch of a fairly representative selection of good class wrapping papers. The results shown may reasonably be expected from any wrappings of the same price and class :—

Class of Paper.	Size.	Weight per Ream of 480 Sheets.	Price per Lb.	Mean Breaking Strain Calculated on Strip, 7″ × 2″.	Mean Stretch. Per cent.
Golden Kraft,	Double Imperial	120 lbs.	1¾d.	80 lbs.	3·5
Glazed Kraft,	,,	120 ,,	2d.	85 ,,	3·5
Casing (air-dried),	,,	180 ,,	2¾d.	50 ,,	3
Casing (air-dried),	,,	140 ,,	2¾d.	35 ,,	3
Casing (white M.G.,	,,	100 ,,	2d.	55 ,,	..
Brown (cylinder-dried),	,,	120 ,,	2d.	45 ,,	..
Glazed brown,	,,	120 ,,	2d.	50 ,,	..
Glazed white rope,	,,	100 ,,	2d.	40 ,,	..
Drab sealing,	Double Demy.	34 ,,	2d.	40 ,,	..

Miscellaneous Papers.

(1) **Carbon Papers** are smeared with a mixture of lamp-black and lard. Wax is used in the "greaseless" variety. Carbons are tested by repeated rubbing with a pencil, after laying them between

two sheets of paper. When the colour is nearly removed from the rubbed patch, the latter is still expected to give a fair copy of written characters made in the usual way.

(2) **Cardboards.**—These may be "pasteboards" (sheets of white paper pasted on to thick middles of varying quality) or "pulp boards," made in sections on the machine without paste. Pasteboards may be distinguished by placing in warm water for a few minutes, after which it should be possible to remove the outside sheets with the fingers. The paste used may be recognised by its greasy feel, and by the blue reaction with iodine solution. Another simple test for a pasteboard consists in setting light to the edge of a strip of card and, after a second or so, blowing out the flame, when the charred sections will curl away from each other.

(3) **Copying Papers** should be strong for their weight, and able to give at least three good impressions with copying ink. Rapid penetration of the ink downwards, but *not* laterally, is required.

(4) **Cutlery Paper** must be entirely free from acid (see p. 36) and sulphides, which would injuriously affect metal. Test for sulphides by warming the paper with hydrochloric acid in a test tube, and holding a strip of filter paper dipped in lead acetate solution across the mouth of the tube. If the test paper is blackened sulphides are present. This

paper should also be damp-proof (see parchment paper).

(5) **Litho Papers** are best tested by taking several impressions on a sheet from the stone, and examining the result for defects in register. They are preferably soft-sized.

(6) **Parchment Papers.**—Real parchment paper is always made from cotton fibre only. Imitation parchment paper will generally contain sulphite wood pulp.

These papers should be water-proof. The writer tests this by making a mark on one side with a copying-ink pencil, reversing the paper, and placing a large drop of water on the surface opposite the mark. Any tendency for moisture to penetrate the paper is seen by its effect on the mark.

(7) **Tracing Papers** should not be stained with the oil used. The surface should stand writing without the ink spreading. The best tracing papers are made from flax, tow, and straw.

(8) **Waxed Stencils.**—Look for roughness of surface, thin patches in the coating, lumps of wax. Make an impression in the usual way, and see how many copies can be obtained (1) before cracks appear, and (2) before the stencil is perforated. Examine the behaviour of the coating towards the letter " e " on the typewriter.

CHAPTER VI.

PARCHMENTS, VELLUMS, AND LEATHER.

Parchments and Vellums are only examined for their physical qualities. The skins themselves are subject to certain inherent natural defects, and their suitability for use is also dependent upon the care with which they have been prepared in the course of manufacture of the finished article. A good writing parchment is expected to show the following characteristics :—

1. It should be of a uniformly white colour, without stains or "fog" spots. Skins bought in the winter are frequently anything but satisfactory as regards colour.

2. Scab marks and scars, although they usually bear ink pretty well, are unsightly, and should not be present, at all events in any number.

3. The right (writing) side should be of a uniform smoothness, and the wrong side should be free from harshness on rubbing gently with the palm of the hand.

4. The surface should be free from lime or whitewash (the latter is often used to disguise discolourations, etc.).

5. The skin should be uniformly stout without thin patches. About six thousandths of an inch is a fair thickness, but parchments are stouter in winter than in summer. Worm holes are to be looked for.

6. Coarseness or a hard boardy feel should be absent. A good skin is flexible and gives a soft velvety impression to the touch.

Most of the above remarks apply equally to vellums, although the latter are, of course, much stouter and stiffer than parchments, and, as a rule, of a harder surface.

Leather.—An important consideration in leather, from the stationer's point of view, is its neutrality. Free mineral acid is frequently present, although often only in minute quantity, and is most destructive in time to the leather. The crumbling of bindings so often seen is due to this acidity. Extract a piece of the leather with hot water for some hours. Evaporate the extract to the smallest possible quantity. Dip a glass rod into a weak solution of Congo red and then into the extract in a porcelain basin. If free acid is present the red colour will turn blue.

The following determinations are usually made in the chemical examination of leather,—viz., moisture, extractives, and ash :—

Moisture.—Heat a weighed piece of the leather in the oven until it ceases to lose weight. The

amount of moisture expelled is thus readily determined. It should vary only between the limits of 12 to 15 per cent.

Extractives.—These are water-soluble substances, such as tannin and sugar. Soak a weighed piece of the leather in distilled water for at least twenty-four hours, and finally evaporate the aqueous extract to dryness over the water bath. The limit of extractable matter obtained in this way may be taken as 10 per cent. on the weight of the leather.

Ash.—Take a small quantity of the leather and incinerate in a crucible with the addition of some ammonium nitrate to ensure that combustion is complete. Weigh the white residue, which represents lime together with any mineral substances which may have been added for the purpose of weighting the leather. These latter include zinc sulphate, and chlorides of lime and barium. The ash of a good class tanned leather may vary between ·8 and 1 per cent.

CHAPTER VII.

WRITING INKS.

Black writing ink should be composed of a finely-divided precipitate of gallo-tannate of iron suspended in water with the aid of a little gum. A small quantity of carbolic acid is usually added as a preservative. The best ink is made by the interaction of sulphate of iron and an aqueous extract of nut galls. Such an ink is found to be superior to any other from the point of view of permanence and penetration. To assist the colouring powers of the ink Aniline black is frequently added. In cheaper inks part or all of the tannin is derived from logwood instead of galls, and in such inks potassium chromate is a common ingredient. Logwood inks are subject to decomposition on exposure to air, the colour tending to deposit in black flakes. So-called "Alizarin" inks are manufactured from iron, tannin (either from galls or logwood), and indigo carmine. Another class of ink contains tannin and ammonium vanadate.

The cheapest black inks are made from Aniline black alone. These latter are fugitive to light, and quite unsuitable for the purposes of permanent record.

Blue-black ink should be made from the same materials as black ink—viz., galls and iron—with the addition of a blue colouring matter. This colouring matter may be soluble Prussian blue, or a dye. Indigo is frequently used.

THE CHEMICAL EXAMINATION OF INK.

1. Black Ink.

1. Put a little of the ink in a test tube, dilute it with water, and add some strong hydrochloric acid.

(*a*) If the solution changes to a clear green, Aniline black is probably the sole ingredient.

(*b*) If the solution becomes of a clear reddish colour, the ink is probably made from iron. Confirm by adding excess of caustic soda solution, when a reddish precipitate should be thrown down.

(*c*) If the ink is made from ammonium vanadate the colour will change to blue.

Note.—Aniline black may be used *in conjunction with* iron. Where this is suspected, the ink should be diluted with water and strong ammonia added. On filtering the reddish solution formed the filtrate may be tested for Aniline black with hydrochloric acid as above.

2. *Tests for Logwood.*—(*a*) On the addition of *strong* hydrochloric acid the ink is turned yellow to reddish when derived from logwood.

(*b*) On boiling the ink with *dilute* hydrochloric acid a cherry red is produced, turning deep violet on addition of excess of ammonia.

3. *Test for a Chrome Ink.*—Evaporate some of the ink to dryness. Fuse the residue in a crucible with a mixture of soda carbonate and nitre. Dissolve out the fused mass with hot water and filter. Acidulate the filtrate slightly with acetic acid, and add a solution of lead acetate, when a yellow precipitate indicates the presence of chromium.

2. Blue-black Ink.

1. Put a little of the ink in a test tube as before, dilute it with water, and add hydrochloric acid. The black portion of the ink should dissolve and leave on addition of more water a clear blue solution. Note, however, that the presence of aniline black causes this blue colour to take a decided violet shade.

(*a*) To test for iron, dissolve a crystal of yellow prussiate of potash in water in a white basin, and to the solution thus obtained add one drop of the clear blue liquid from (1), when, if the ink is made from iron, a deep blue precipitate of *insoluble* Prussian blue, easily seen in the blue solution, will be produced.

(*b*) Take a further quantity of the solution from (1), and warm with a little caustic soda solution. If the blue colour of the ink is due to soluble Prussian

blue, the further addition of hydrochloric acid will cause its reappearance.

2. *Test for Indigo.*—The addition of hydrochloric acid to the ink will yield in the presence of indigo a clear blue colour instantly discharged on adding bleaching powder solution. Indigo is also decolourised on boiling its solution with dilute nitric acid.

3. *Test for Indigo and Logwood together.*—On addition of concentrated sulphuric acid the ink is green if both the above colouring matters are present. On diluting the green fluid with water and filtering several times the filtrate gradually turns yellow from retention of the indigo on the filter (*Allen*).

Coloured Inks.

In the great majority of instances these are simply solutions of organic dyestuffs.* Very few coloured inks are permanent.

Red Inks may be solutions of Brazil Wood, Cochineal, Magenta, or Eosine. The following simple tests will serve to distinguish the above colours :—

Brazil Wood.—A drop of hydrochloric acid changes the colour to orange.

* For the detection of aniline colours in general the following test may be applied :—Take 4 c.c. of alcohol in a test tube, add one or two drops of water, and dissolve as much caustic potash as the solution will take up. Now add a drop or so of the ink and two drops of chloroform, and warm the whole to boiling, when, if an aniline colour is present, a characteristic and highly offensive odour will be developed (isonitril). A drop of sulphuric acid will bring out the effect in a more pronounced manner.

Cochineal is turned violet-red with ammonia solution.

Magenta is turned yellowish-brown by hydrochloric acid.

Eosine.—A drop or two of hydrochloric acid will precipitate orange flakes, which are rendered soluble on shaking with a little ether. At the same time the clear colour of the ink is restored.

Blue Inks may be soluble Prussian blue, indigo, or an aniline blue. The identification of these has already been considered.

Violet Ink is usually an aniline colour. The addition of a little hydrochloric acid will probably turn it first green and then yellow.

Green Ink may be made from verdigris, or bichromate of potash, or an aniline colour. To test for verdigris, evaporate some of the ink to dryness in a basin. Dip the end of a piece of clean platinum wire in hydrochloric acid, and then in the dry residue from the ink, finally holding the platinum wire in the Bunsen flame, when the latter will be coloured a livid green if verdigris has been used. To test for bichromate, proceed as described under "Chrome Ink."

General Examination of a Black Permanent Record Ink.—An ink for permanent record purposes must be, above all things, durable—*i.e.*, must preserve its colour unchanged from the action of

light and air for many years. It is also desirable that it should produce its permanent colour quickly and strongly, should be of such consistency as to flow freely from the pen, should not have a markedly corrosive action on pens, and should possess good penetrating powers on the fibres of paper.

Examine the Ink as follows:—1. Take the specific gravity with the hydrometer after allowing any froth on the surface to subside. A good ink should give a reading near 1·036 (taking water as 1).

2. Evaporate a weighed quantity of the ink in a basin over the water bath. Dry the residue and weigh. The total amount of solid matter should be not less than 6 per cent. on the weight of ink taken.

3. Take a weighed quantity of the ink in a beaker and add *just sufficient* hydrochloric acid drop by drop to form a clear solution. Now add excess of ammonium sulphide, and filter the resulting black precipitate. Wash this precipitate on the filter paper with ammonium sulphide and then hot distilled water. Transfer the whole to a basin and redissolve in hydrochloric acid. Boil well, add a crystal of potassium chlorate, allow to cool, and then add excess of strong ammonia to precipitate the iron. Boil the whole and filter off the precipitate. Wash the latter with boiling distilled water, dry, transfer to a weighed crucible, and ignite. The residue represents the iron in the ink in the form of oxide.

Weigh the cooled crucible and deduct its original weight. On multiplying the figure obtained by ·7 the result will give the weight of iron present in the amount of ink taken. This weight should represent not less than ·6 per cent. of the ink.

Example.

Weight of ink taken = 20 grms.
,, solid matter = 1·15 grms. = 5·75%
,, iron oxide = ·151 grm.

∴ Weight of iron = ·151 × ·7 = ·105 grm.

$$i.e., \frac{\cdot 105 \times 100}{20} = \cdot 52 \text{ per cent.}$$

4. Observe the action the of ink on steel pens immersed therein for some days. The less the amount of corrosion the more care has been taken in the manufacture of the ink to keep it as neutral as possible.

5. Taking Stephens' ink as a standard, compare the time taken for the black colour of the ink under examination to develop completely. See that the ink flows freely and without stickiness from the pen.

6. Expose characters written with the ink on rag paper to strong sunlight for a week, and look for any fading or discolouration.

7. Let the ink stand for a fortnight in a tall glass jar covered with paper. Note whether any mould forms or sediment deposits on the sides and bottom of the jar.

CHAPTER VIII.

SEALING WAXES, GUM, AND OFFICE PASTE.

Sealing Waxes are of several qualities—viz., extra superfine, superfine, fine, and common. In the first qualities the best pale shellac is the principal ingredient, together with Venice turpentine (to neutralise the brittleness of the other ingredients), a little rosin, and any colouring matter necessary in the form of good quality pigments. In common waxes rosin is used largely, or wholly, to replace shellac, and the very cheapest materials are employed for colouring. The best red waxes are coloured with vermilion (frequently mixed with gypsum or chalk), cheaper waxes have red lead as their pigment, and for the very commonest waxes some form or other of oxide of iron is employed. Black waxes are usually coloured with lamp-black or soot. Brown wax will probably contain iron ochres.

All these colouring matters, in addition to the earthy matter of the wax itself, tend, of course, to reduce its adhesive powers.

Physical Examination.

A good wax is of a glossy smooth surface, and presents, when broken, an even fracture without

striæ or holes. It should not be too brittle, extreme brittleness suggesting considerable adulteration with rosin. It should not run into thin drops when melted in the flame of a candle. It should soften on heating, without dropping, and not harden too quickly after cooling. Impressions taken with it should be sharp and clear.

Chemical Examination.

The chemical examination of sealing wax is usually confined to the determination of the per cent. of ash or mineral matter present, and to the detection and estimation of colophony—*i.e.*, rosin. Tests may be made, if necessary, to determine the nature of the colouring matter added.

The Ash is estimated by burning about 5 grms. of the wax in a large porcelain crucible previously weighed, and continuing to heat strongly until the organic constituents are entirely removed. The crucible is then cooled, and its weight again taken, when the difference gives the weight of the ash. The figure found is expressed as a per cent. of the weight of wax taken, by a simple calculation.

Sealing wax should not show more than 20 per cent. of ash in the case of superfine, and 35 per cent. in second quality, while 50 per cent. in common waxes should be the maximum permissible.

Rosin (Colophony) is commonly added to even the

most expensive sealing waxes, on account of the comparatively high price of shellac, but shellac itself is quite frequently adulterated with rosin (up to 10 or 12 per cent.); in fact, it is somewhat uncommon to find a sample which does *not* contain colophony. The following process may be employed in testing for colophony in sealing wax :—

Take a minute quantity of the wax and dissolve as completely as possible in a little anhydrous acetic acid. Pour off the acid into a narrow test tube, cool under the tap, and let a large drop of sulphuric acid of specific gravity 1·53 run down the side of the tube *very gently* on to the acetic solution. In the presence of colophony a reddish-violet colour changing to red-brown is immediately produced at the line of juncture of the two acids. The colour is best seen against a white background (*Storch* and *Morawski*). Where a large percentage of rosin is present, the colour will probably be developed throughout the bulk of the solution.

It is to be regretted that there is no simple, and at the same time accurate, process for the *quantitative* estimation of colophony in sealing wax. The following method used by the author depends on the varying degree of solubility of rosin and shellac in ether. Both these substances are soluble, rosin entirely so, shellac to the extent of about 5 per cent. only.* Since, however, the admixture of rosin in

* Rather more than less.

sealing wax is not of great importance, unless present in a considerable proportion of the weight of the wax, the method gives, within certain limits, a fair means of estimating the value of the wax. Proceed as follows :—

Step 1.—Weigh out accurately about 5 grms. of the finely-powdered wax, and transfer to a weighed stoppered flask. Half-fill the flask with ether of specific gravity ·720, shake well, and allow to stand for some time, shaking at intervals. Pour or syphon off as much as possible of the ether, fill up again with fresh spirit, and proceed as before.

After again decanting, draw off the last few drops of ether with a pipette, being careful not to disturb the residue. Drive off any remaining spirit *over the water bath*, dry, and weigh the flask. The loss in weight represents the rosin in the wax, together with about 5 per cent. of shellac.

Step 2.—Fill up the flask, this time with absolute alcohol, and warm over the water bath, stirring the mixture until the spirit shows signs of boiling. Allow to settle, pour off the extract, put in more spirit, warm again, and remove as before.

Drive off any remaining alcohol, dry and weigh the flask with the residue, which, of course, is the inorganic and colouring matter of the wax. The further loss of weight represents the greater part (about 95 per cent.) of the shellac present. Calculate as follows :—

Suppose —Weight of wax originally taken = 5·78 grms.
„ rosin, etc., from Step 1 = 1·50 grms.
„ shellac from Step 2 = 2·28 grms.

Then weight of total resinous matter dissolved = 3·78 grms.

And total weight of shellac present $= \frac{2{\cdot}28 \times 100}{95} = 2{\cdot}4$ grms.

∴ total amount of rosin present = 3·78 − 2·4 = 1·38 grms.

i.e., $\frac{1{\cdot}38 \times 100}{5{\cdot}78} = 23$ per cent.

Note.—The " Venice " turpentine added to sealing wax is now almost invariably a factitious article prepared by dissolving rosin in oil of turpentine. In any case it is entirely soluble in ether, and must be included for the purposes of the above estimation with rosin as an adulterant of the wax.

Examination of the Colour.

The colouring matter of *red* wax only will be considered here. Test for :—

1. **Vermilion.**—Dissolve a little wax in spirits of wine, collect the red insoluble residue, dry and dissolve in strong hydrochloric acid to which a crystal of chlorate of potash has been added. Now add a little caustic soda solution, when a yellow precipitate will be thrown down if vermilion has been used.

2. **Red Lead.**—Dissolve the wax as before in alcohol. Warm the dry residue with dilute hydrochloric acid, when the colour will be changed from red to buff if red lead is present.

Gum and Office Paste.

The liquid commonly known as "gum" consists of one of the following substances—viz., Gum Arabic or Dextrin.

Gum Arabic is employed as an adhesive in solution in water, with the possible addition of a little alum. The dry powdered article is frequently adulterated with flour, starch, and inferior gums, such as cherry-tree gum and gum tragacanth. Starch may be detected by giving a blue colouration on dissolving some of the gum in boiling water, cooling, and adding a little dilute solution of iodine in potassium iodide. If cherry-tree gum or tragacanth are present the gum is only partly soluble in *cold* water, while the resulting paste is partly coloured and more or less interspersed with gelatinous clots (*Cooley*). Solid dextrin is sometimes substituted for gum arabic, in which case the solution in water will give a *brown* colouration with solution of iodine. Pure gum arabic gives a precipitate with a solution of basic lead acetate. The solid matter left on evaporation of the gum solution should give not more than 4 per cent. of ash, after burning in a weighed crucible.

Dextrin (British gum) in its commercial form consists of a number of allied substances, among which erythrodextrin largely predominates. This latter body should cause a brown colouration on addition of iodine solution to dextrin, but soluble

starch may be present to such an extent as to obscure the colour by its own reaction. To prevent this use extremely dilute iodine solution. The starch present should not exceed 15 per cent. Dextrin is insoluble in both alcohol and ether, and its solution in water is not rendered turbid by the addition of oxalic acid, as is that of gum arabic.

Office Paste is usually a mixture of soluble starch and glue, with the addition of a little alum, with oil of cloves added as a preservative. Its keeping properties may be tested by exposing it to the air for a week, in a saucer.

Note.—Acidity in both gum and paste will affect the colour of dyed papers, and is destructive to the paper itself. Its presence should be tested for with Congo red, as described on page 46.

CHAPTER IX.

MISCELLANEOUS ARTICLES OF STATIONERY.

String and Cord.—The fibres used in the manufacture of these articles are hemp, jute, and (occasionally) flax. Hemp alone is desirable for ordinary purposes, where strength is the chief consideration. Jute is largely used as an adulterant.

For the examination of the fibres a small piece of the string may be unravelled and boiled in water with a drop or so of caustic soda. The fibres should then be well rubbed up in a mortar, and finally teased out on a glass slide. The stain used may be a 1 per cent. solution (1 grm. in 100 c.c. water) of potassium iodide, afterwards saturated with iodine. This reagent is applied to the fibres on the slide, allowed to remain for a few minutes, and then removed with blotting paper. Finally, the fibres are touched with a drop of a mixture made by carefully diluting three volumes of strong sulphuric acid with one volume of water, and adding two volumes of glycerine, and the cover glass is then fixed in position. The colours produced in the fibres by the above reagent are as follows :—

Flax, . . .	Blue.
Hemp, . .	Yellow or greenish.
Jute, . . .	Brownish-yellow.

The microscopical characteristics of these three fibres have already been discussed in Chapter III.

The following chemical reactions are helpful for distinguishing the fibres without the use of the microscope :—

1. The fibres are soaked in a strong solution of caustic soda. Flax is coloured yellow by this reagent, hemp yellow-brown, and jute red-brown.

2. The application of nitric acid to the fibres should not produce any colouration in the case of pure flax. Hemp is slowly coloured yellow-brown and jute an immediate deep brown.

3. On prolonged immersion in chlorine water flax is bleached white, hemp is partly bleached and partly coloured yellow, and jute becomes strongly yellow. On washing the fibres and immersing in a solution of sulphite of soda hemp turns pinkish and jute a brilliant magenta. Instead of the sodium sulphite, ammonia solution may be used, when jute is coloured violet.

The presence of jute may be confirmed by the following reactions :—

1. The fibre is coloured blue by a dilute solution of chromic acid, to which has been added a little hydrochloric acid (*Herzfeld*).

2. The application of the phloroglucine reagent on page 35 causes a *brilliant* red colouration in jute fibre.

3. A solution of 1 grm. of aniline sulphate in 10 c.c. of water with the addition of one drop of sulphuric acid colours jute fibre a brilliant yellow.

String and cord are tested for tensile strength by means of an apparatus resembling the Schopper machine used for paper testing and illustrated on page 65. The machine has two clips, 2 feet apart, between which the string, etc., is fixed. On operating the handle, the weight travels along the quadrant, until the increasing strain causes the fibres to rupture. The breaking strain in pounds can then be read off on the dial at the top of the machine. The next table shows breaking strains that may be expected from cord and twine of certain definite qualities :—

Description.	Length per Lb. Weight.	Breaking Strain in Lbs.
Hemp cord of 3 strands, about $\frac{5}{8}$ inch in circumference, .	15-20 yds.	450
Twine of 3 threads. . .	170-200 ,,	100
,, 2 ,, . .	550-600 ,,	25

Apart from the above considerations, string and cord are required to be free from fluffiness, to show a high degree of flexibility, to be of a good colour, and reasonably free from greasy matter.

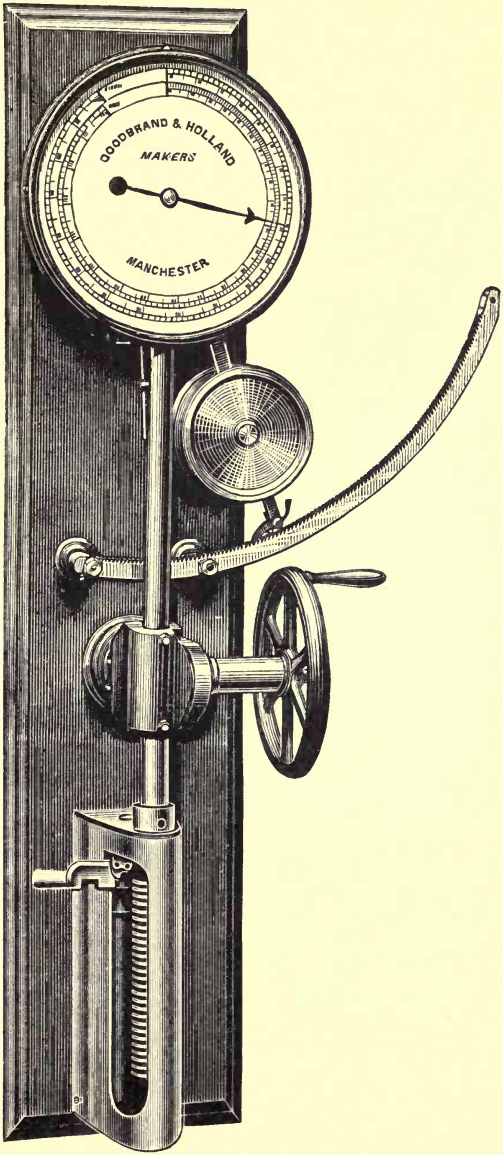


Fig. 4 —Goodbrand's Yarn Testing Machine.

Lead Pencils contain a mixture of graphite and clay pressed hydraulicly into a solid mass. The proportion of clay present determines the "hardness" of the finished product. In the best class pencils great care is taken to free the clay as much as possible from gritty matter; but the latter may be conspicuously present in cheaper varieties.

The "lead" must not be so brittle that it breaks off short in the pencil during the operation of sharpening or during use, nor must it be so excessively soft as to be wasteful or smudge the paper. A fair test of the hardness of a pencil may be made by seeing how many carbon copies of a writing can be obtained at one operation.

Estimation of the Mineral Matter.

Owing to the great difficulty of satisfactorily burning the graphite without special appliances, the writer prefers the following estimation of silica and alumina (the main constituents of the inorganic matter present in the lead of a pencil) :—

Weigh out about 1 grm. of the finely-powdered lead. Fuse strongly in a crucible about 5 grms. of a mixture of sodium carbonate and potassium carbonate, intimately mixed with the lead. When the mixture is well fused add some nitre and continue heating strongly until the black carbonaceous matter has quite disappeared, adding more nitre as required. Cool the crucible, dissolve its contents

in distilled water by placing the crucible bodily in a beaker and boiling, and filter. The filtrate will contain the silica and alumina combined with sodium and potassium.

1. Acidulate the solution strongly with hydrochloric acid, transfer to a basin, and evaporate down to dryness over a beaker of boiling water. Heat the residue in the basin in an oven for a few minutes, then moisten it with more hydrochloric acid, add distilled water, and boil up again.

2. The *silica* will now be left as an insoluble residue, and is to be filtered off, washed with distilled water on the filter paper, dried, and finally transferred bodily with the paper to a weighed crucible, and ignited. On cooling and weighing again, the weight, after deduction of that of the crucible itself, represents the *silica*.

3. The *alumina* is left in solution after operation No. 1 (above), and is to be precipitated by adding to the filtrate from operation No. 2 just enough strong ammonia to neutralise the acid and make the solution smell faintly. The alumina, together with a small amount of iron, is precipitated as a reddish gelatinous substance, and must be filtered off, washed, dried, and ignited precisely as was done in the case of the silica. On deducting the weight of the crucible we get the weight of the alumina and iron together.

4. From the above results the percentage weight

of mineral matter in the sample is calculated as follows :—

$$\frac{(\text{Weight of Silica} + \text{weight of Alumina}) \times 100}{\text{Weight of "lead" taken}}.$$

A good H.B. pencil lead, suitable for all-round use, should show approximately 35 per cent. of dehydrated mineral matter present, corresponding to about 40 per cent. of added clay.

Example.

Weight of "lead" taken = ·312 grm.
Weight of silica determined = ·054 grm.
Weight of alumina and iron determined = ·046 grm.

∴ Per cent. weight of mineral matter (dehydrated)

$$= \frac{(\cdot 054 + \cdot 046) \times 100}{\cdot 312} = 32 \text{ per cent.}$$

Typewriter Ribbons.—Their comparative merits may be tested as follows :—

Disconnect the ribbon-propelling mechanism of the typewriter so that the same portion of the ribbon receives continuous impression from the keys. Continue striking the letter "e" firmly and evenly and at regular intervals until the impression becomes very faint, and note the number of taps required before this happens. Note whether the ribbon is perforated, and the amount of blurring resulting from the first few impressions. Expose a sheet of typewritten matter to strong sunlight for

a week, and examine to see how much fading has occurred.

Draughtsmen's Rubber should be flexible and not break on bending sharply. The rubber should not be oily, or give a greasy appearance to the paper on erasure, nor must it crumble or wear off into flakes too easily.

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