The origin and assessment of cotton fibre maturity

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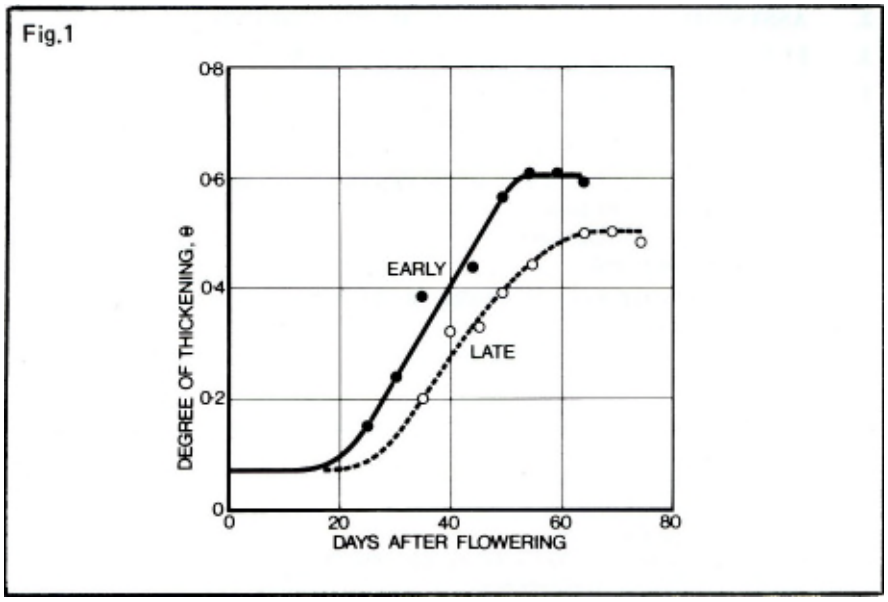
This booklet was published originally in 1975 and revised in 1981 when some additions were made. It has become apparent, however, that recent developments affecting the subject matter needed to be introduced more appropriately and a completely new chapter has been compiled by S. A. Heap of IIC and added as section 8. The original text by E. Lord has been retained as sections 1 to 7 with only minor amendment.

1. The Physical Meaning of Fibre Maturity

The term *fibre maturity* is somewhat vague mainly because of its early origin in the marketing of raw cotton. It is not an estimate of the length of the period of fibre growth, although many years ago it was commonly thought that all 'immature' cotton was caused by early death of the boll or seed. Fibre maturity is now generally accepted as meaning that the fibre wall has developed to an acceptable level of thickening. It is therefore appropriate to consider first the pattern of cotton fibre growth within the cotton boll before reviewing the ways of assessing wall thickening.

Many cotton fibres originate about one or two days before the flower opens, when some of the epidermal cells on the small ovules or young seeds start to develop differently from their neighbours [21]. These budding cells rapidly assume an elongated tubular shape. If opening of the flower is followed by fertilisation, this wave of epidermal cell differentiation spreads over the seed surface and the special cells continue to elongate. These future cotton fibres attain their maximum length usually about 25 to 35 days following flowering. As fibre length growth approaches its completion, the secondary fibre wall starts to be built up on the inner surface of the thin primary wall; it continues for a further 35 to 50 days [23]. The time scale of initial length growth and secondary-wall development depends markedly on variety, growth conditions and the plant season [40]. Generally, the build-up of the secondary wall is initially fairly rapid, but the rate then slackens and growth has usually stopped a few days before the cotton boll splits open. The free moisture inside the boll evaporates into the open air, the fibres dry and collapse to give the typical convoluted ribbon form of 'raw cotton'.



The pattern of cell wall thickening is illustrated, for early season, well-grown Sudan Sakel cotton, by the upper curve of Fig. 1. Flowers opening on the same day on plants in several short rows of the same field were identified by tags. Later, 100 bolls were picked at 5-day intervals from 20 days after flowering to full maturity at 65 days old when normal boll split occurred. The partly-grown bolls of the premature pickings were opened by hand and the contents removed to dry in the sun. Determinations were made of the maturity ratio on each sample to assess the degree of wall thickening discussed in Section 3. For very young fibres with no secondary wall development the degree of thickening characterising the primary wall was estimated from section measurements and by weighing single fibres: both estimates were about 0.07. Examination of the diagram shows that by the time the fibres were 25 days old rapid development of the secondary wall had started. The growth rate slackened after about

45 days and ceased some 10 days before normal boll split occurred. By this time the final degree of thickening of the ripened fibres was about 0.6, an average value much below unity which would correspond to all fibres having a solid circular form.

For flowers produced later in the season, the lower curve of Fig. 1 shows that development of the secondary wall starts somewhat later, then increases at a slower rate, and finally terminates at a lower level a few days before boll split occurs at 75 days after flowering. The fully-grown 75-day old cotton of lower degree of wall thickening is 'immature', but the immaturity was not caused by premature death of the fibres: it is a consequence of the poorer growth conditions including plant senescence.

All fibres do not grow in the same manner. If growth conditions are favourable most of the fibres are mature, i.e. they have relatively thick walls. But even under the best conditions some fibres have poorly-developed, thinner walls. If growth conditions are poor the onset of secondary wall thickening is commonly delayed, then proceeds at a slower rate and finally ceases at a lower level. These fully-grown bolls contain a much higher proportion of poorly-developed immature fibres. Fibres from different localities, different fields, different plants, different bolls on the same plant and even from different places on the same seed differ markedly in the pattern of wall development because of many factors. Thus, in any handful of cotton, even of the highest grade, the individual fibres vary greatly in their wall thickening.

Some illustration of the extent of this fibre-to-fibre variation is given by other test data obtained for the special pickings of the early-flowering Sudan Sakel cotton. Determinations were made of the linear density of the central part of 200 fibres per sample by weighing individual 2-cm cut lengths on a very sensitive microbalance. Table 1 gives the frequency distributions for three samples in the time series.

Table 1

Linear density of single cotton fibres (millitex, or 10^{-8} g/ cm)			
Frequency Group (millitex)	**Frequency**		
	25 days	40 days	65 days
0 -	3		
20 -	108	1	1
40 -	73	11	2
60 -	13	25	8
80 -	3	44	12
100 -		72	35
120 -		37	42
140 -		6	55
160 -		4	29
180 -			9
200 -			5
220 -			0
240 -			2
Totals	**200**	**200**	**200**

Part of the fibre-to-fibre variation in linear density arises from variation in fibre perimeter but this is limited especially because all samples were taken from the same group of plants. The dominant cause of the variation in linear density is the variation in wall development.

Single fibre measurements are only practicable for research investigations; for most purposes changes in fibre maturity must be assessed in terms of changes in average values. Nevertheless, it is always advisable to remember that all qualities of cotton, mature cottons as well as immature cottons, contain many individual fibres and many small tufts of fibres that have very little wall development, like most of those with a fibre weight per centimetre of less than 80 millitex in the well-grown, fully-developed 65-day old sample in Table 1.

2. Assessment of Maturity by Hand and Eye

Fibre maturity affects markedly the appearance and value of raw cotton, its performance in spinning and the quality of the finished textile product. It is therefore not surprising that qualitative assessment of maturity has long been practised. Critical appraisal is achieved after wide experience and various tactile and visual observations are simultaneously taken into account in formulating a judgement. The greatest value is derived when such observations on the raw cotton can be co-ordinated with subsequent experience of processing performance and product acceptability.

Although a good classer notices several features of the cotton simultaneously it is of interest to give them separate consideration.

- The small wall thickness of immature fibres results in a lower fibre rigidity. Thus, a mature sample of cotton has a firm springy handle; an immature sample feels soft and lacks ‘body’.
- The lower rigidity of immature cottons causes a greater tendency for any mechanical treatment to entangle fibres into small knots or neps. Neps are not formed during growth inside the boll. Bad pre-ginning and ginning operations usually give rise to excess nep in both mature and immature cotton, and lower the value of both. However, even good ginning cannot be effected without producing some nep in immature cotton, nep that will be increased even by good mill processing. Thus, nep visible in raw cotton is not only a sign that trouble is already present; it often indicates that more trouble will be seen when the cotton is processed in the mill.
- A third feature commonly observed with immature cotton is the poor lustre, caused by the average shape of fibre cross section being less round than with mature cotton.
- When a cotton boll opens, the fibres move relative to each other as they dry, collapse, and rotate to form convolutions. Immature cotton, even after ginning, is less open and fluffy than mature cotton that has had the same treatment.
- The appearance in raw cotton of small, white, shiny unopened bundles of fibres indicates the presence of bunches of dead fibres of very small wall thickness and few convolutions. When present in quantity it is a sign that the general level of fibre maturity is likely to be low. But even small amounts of such cotton in a bulk of satisfactory average maturity may cause severe neppiness and dyeing problems.
- Whole bolls, or parts of bolls, attacked early by pest or disease may suffer a poor fibre length growth and subsequently a poor secondary wall development. Alternatively, there might be normal length development, but later adverse conditions could give inferior wall development. In both instances the immature cotton would have a low fibre strength and

break more easily in ginning. Excess fibre length irregularity is itself a bad feature; it is often an indication that the cotton is also immature.

- When a small tuft of parallel fibres of a mature cotton is pulled there is a strong resistance to breakage, until eventually rupture occurs with a typical short, sharp 'snap'. A bundle of immature fibres seems to require a lower load before rupture happens, and then there is a typical slow, drawn-out 'softer' break. The process is complex, especially because finger assessment of strength also takes fineness and maturity into account since both these features affect the allowance that needs to be made according to the visual size of the bundles.
- Many cotton plant pests puncture growing bolls and so permit the later entry of micro-organisms, which develop and may cause the death of all or part of the boll contents. Secondary wall development may be poor, or not even start. Cotton from such bolls is often stained a characteristic yellow or reddish-yellow colour, sometimes only spotted if the attack is not general. Stain or spot in raw cotton gives a warning, not only that the cotton is probably immature but also that there may be other adverse aspects such as reduced fibre tenacity or associated staple length irregularity.

The assessment of cotton fibre maturity by hand and eye methods has several disadvantages.

1. The method is subjective: different people attach different degrees of importance to the various adverse features of immature cotton.
2. The various combinations of feel and appearance characteristic of raw cotton immaturity vary in importance according to the variety and type of the cotton. As a simple example, the amount and distribution of observable yellow spot depends upon whether a saw- or roller-gin has been used. A saw-gin tends to dissipate yellow spot. The different action of the roller-gin results in fibres remaining in drawn-out bunches so that even a small amount of spotted cotton may easily be seen.
3. Unbiased assessment of fibre maturity is not feasible because some of the features noted in making a judgement are also related to other fibre characteristics of the sample.
4. The dependence of the assessment on varying combinations of very diverse features of a sample makes it impossible to establish a range of *standard maturity types* analogous to those for grade and staple and used for standardising classing practices.

It is therefore not surprising that the need to express assessments of fibre maturity on a numerical basis, and to obtain reproducibility for all classes of cotton, has long stimulated the development of diverse methods of testing. The fundamental tests are essentially direct but slow, with some technique disadvantages. Quicker tests were later inaugurated but these gave results that were biased because of lesser effects of other fibre features. This encouraged more detailed test techniques to eliminate such bias. The advantages and disadvantages of the several major types of test are discussed in the remaining part of this paper. But, despite the longer treatment that has been given to test methods, it needs to be emphasized that examination of cotton samples by hand and by eye often gives to the breeder, to the grower, to the merchant and to the spinner valuable practical information that cannot be obtained from blind, impersonal, mechanical tests.

3. Fundamental Unbiased Maturity Tests

The average thickness of the cell wall may be measured directly by preparing transverse fibre sections and drawing or photographing an adequate selection of microscope images. Measurements of wall thickness may then be made on these records, preferably not less than 500 per sample. The method is very tedious. Serious errors [23, 31] arise unless steps are taken to avoid unwanted and possibly biased variation associated with distortion of the fibre cross sections, measurement technique, selection and orientation of sections, etc.

An alternative method is to make measurements on longitudinal microscope views of the fibres. However, the refraction and the folding of the fibre wall are sources of additional and appreciable error so that this procedure is suitable only for making crude comparisons and not for absolute measurement.

However, the absolute thickness of the fibre wall does not give an unbiased measure of relative maturity. Intrinsically fine cottons of small perimeter are usually of smaller average wall thickness than appreciably coarser cottons of large perimeter - but they are not necessarily less mature. Relative maturity might perhaps be expressed as the ratio of absolute wall thickness to average ribbon width or average fibre diameter [8]. But this approach also is not satisfactory. Analysis of detailed unpublished data shows that both ribbon width and average diameter vary with fibre wall development because of the associated changing shape of the fibre cross section.

A more satisfactory measure of fibre maturity, one that is independent of fibre perimeter, is the 'degree of thickening', θ defined [23] by

$$\theta = \textit{cross-sectional area of fibre wall} \,/\, \textit{area of equivalent circle}$$

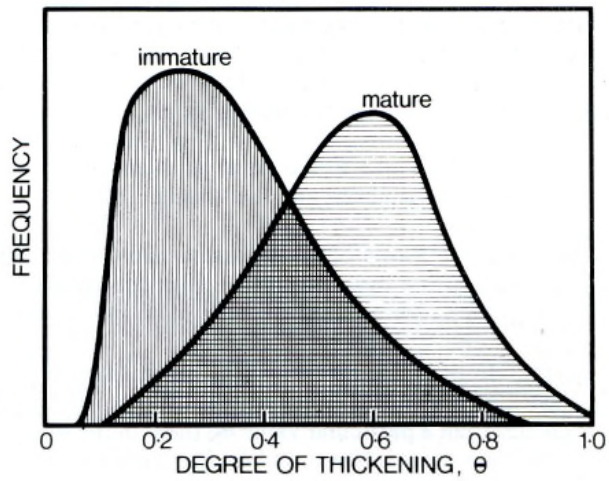
where '*equivalent circle*' is the circle having the same perimeter as that of the fibre cross-section.

Completely circular, solid fibres, irrespective of their perimeter, have a value of θ equal to unity. Typical mature fibres have a kidney-bean shape of section and a moderately high value of θ. Immature fibres with little secondary wall thickening have a small value of θ. The degree of thickening θ may be regarded as a fundamental unbiased measure of fibre maturity, measuring the extent of the fibre wall relative to its maximum potential.

In the foregoing section of this paper attention was directed to the large fibre-to-fibre variation in wall development in any sample of cotton, a feature that may be characterised by the wide spread in the distribution of the value of θ given by single fibres. This is illustrated by the frequency curves given in Fig. 2 for a mature and an immature sample of cotton.

The two distributions overlap considerably in their ranges of θ for individual fibres, but the average value of θ for the mature sample is appreciably higher than that for the immature sample. Characterization of a variable feature such as maturity by an average value does not take into account the pronounced spread on either side of the average. Attention was drawn in the previous section to the danger of a tail of immature cotton in a sample of good average maturity.

Fig.2



The direct determination of θ involves the measurement of both the area of wall thickening and the perimeter for a large number of fibres. The method has given invaluable information for research purposes, but it is extremely slow and errors [23, 31] arising in the preparation, recording and measurement of the fibre cross sections should be minimized by various means. For everyday purposes direct assessment of θ is not practicable. Nevertheless, any test for fibre maturity should give a quantity that varies with θ alone. If there is not a one-to-one correspondence between θ and the measure of maturity given by the indirect test it follows that such a measure depends also upon some other fibre feature or features.

4. Practical Unbiased Maturity Tests

4.1 Standard procedure in the United Kingdom

The most feasible way of obtaining an unbiased estimate of the degree of thickening *0* is the caustic soda swelling test. It has been used for many years and takes two different forms. In the standard method used in the United Kingdom (BS3085) fibres are first swollen in 18% caustic soda solution and then examined under the microscope. The form adopted by a swollen fibre depends upon its degree of thickening and, in this test, fibres are classified into three groups according to their visual appearance.

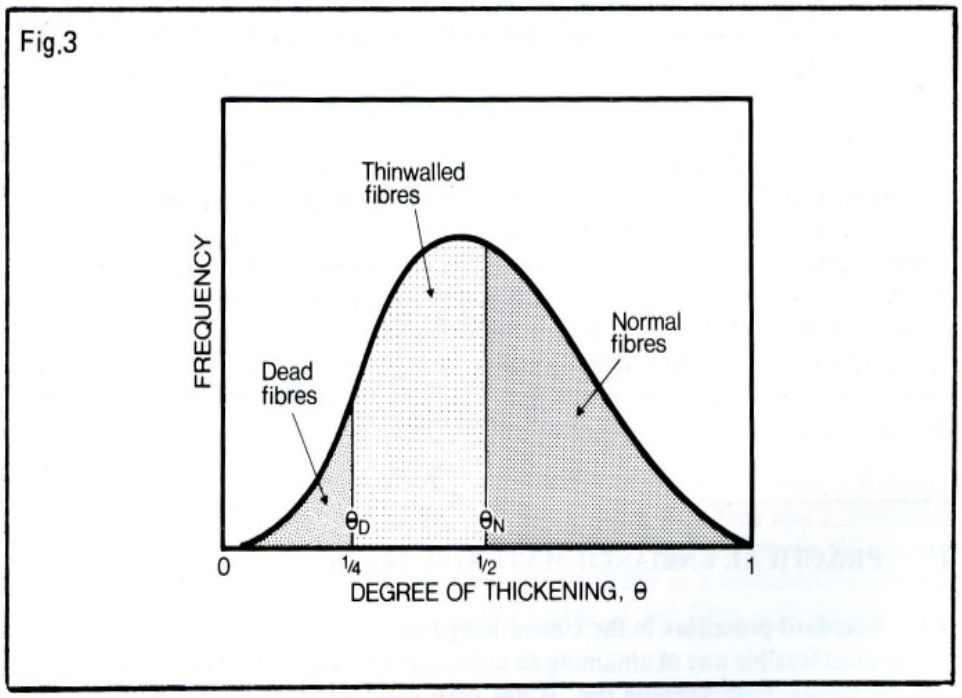
Normal fibres are those which, after swelling, appear as solid rods and show no continuous lumen nor have well-defined convolutions.

Dead fibres after swelling have a continuous lumen and the wall thickness is a fifth or less than the ribbon width (measured at the widest portion of the fibre in the microscope field). They usually show frequent convolutions but those with little or no secondary wall thickening often appear as flat unconvoluted ribbons.

Thin-walled fibres are those which are not classed as Normal or Dead, being of intermediate appearance and thickening.

The test is usually made on a group of five mounted microscope slides, each with about 100 fibres. The results are expressed as the average percentages of Normal (N) and Dead (D) fibres. Replicate tests are made according to the accuracy required.

The physical interpretation of the maturity test percentages is of fundamental importance and is illustrated in Fig.3.



If an untreated raw cotton fibre has a degree of thickening θ that is greater than a particular value θ_N then, in the swollen state, it will be classed under the microscope as a Normal fibre. Similarly, untreated fibres with a degree of thickening less than a fixed value θ_D will be classed as Dead fibres in the swelling test. Thin-walled fibres have a degree of thickening θ between θ_N and θ_D.

Detailed fundamental research has shown that θ_N is closely equal to ½ and θ_D is about ¼. Thus, in the caustic soda swelling test the three determined test percentages correspond to three broad frequency groups of the full distribution of degree of wall thickening θ.

It is highly desirable that any valid unbiased maturity test should give results that may be used to provide an estimate of the degree of wall thickening. The original research [23] effected this transformation by use of the empirical relation

$$\theta^* = 0.577 \, . \, [(N - D) / 200 + 0.70]$$

the symbol θ^* being used to denote the average value of θ corresponding to the maturity test percentages N and D.

For practical convenience and simplicity, the term within the square brackets is known as the *maturity ratio* M, thus

$$M = [(N - D) / 200 + 0.70]$$

The maturity ratio is, of course, directly proportional to the average degree of thickening. It assesses wall thickening relative to a standard maturity level of (N - D) = 60, for example N = 67 and D = 7. This reference level is an optimum level reached only by high grades

and rarely exceeded in raingrown material. The reference level, when M = 1, corresponds to an absolute average degree of thickening of θ equal to 0.577.

Practical everyday interpretation of test values of maturity ratio is simplified by this reference point of M = 1 being within the range of commercial cottons. On the other hand, in even the most mature sample there are few fibres which are completely round and solid, with θ = 1, and the average value θ^* is always much less than 1. Table 2 indicates the interpretation that may be attached to M for two classes of cotton.

Table 2

M	USA Upland	Sudan-Egyptian
1.00 and more	very mature	high grade, mature
1.00 - 0.95	above average	medium grade, average
0.95 - 0.90	mature	low grade, below average
0.90 - 0.85	mature	uncommon
0.85 - 0.80	below average	
0.80 - 0.70	immature	
Less than 0.70	uncommon	

Values of the maturity ratio M and of the estimated average degree of thickening, are independent of the *intrinsic fibre fineness* as described by the fibre perimeter. In this sense these test quantities give unbiased estimates of the true average degree of thickening. They are, of course, subject to some random variation associated with sampling and testing errors, including fluctuations in the test level of operatives who may deviate despite having standard reference samples for check testing. Occasionally there may be additional variation caused by an abnormal change in shape of the distribution curve of θ. However, for most cotton production there is a fairly steady change in the pattern of the distribution curve from very low to very high maturity, so that the three broad frequency groups give good estimates of the average degree of thickening.

4.2 Standard procedure in the U.S.A.

In the United States it is standard practice to use a maturity test (ASTM D1442) in which, after swelling with an 18% solution of caustic soda, the fibres are classified into only two categories. *Mature* fibres examined in the swollen state have a ratio of apparent wall thickness to ribbon width that is greater than a quarter. The remaining fibres are termed *Immature*. Thus, the Mature class of the U.S. test embraces all the Normal and part of the Thin-walled group of the U.K. test. The Immature fibres of the U.S. test includes the remaining lesser-developed Thin-walled fibres and all the Dead fibres of the U.K. test.

The advantages of using the U.S. system include greater simplicity of classification and reduced test time: only 2 instead of 3 counts are required when assessing each single quantity, namely *% Mature Fibres, Pm.*

Test values for percentage Mature fibres (Pm) may be converted into estimates of maturity ratio M from either conversion tables or by use of the empirical relation [19].

$$M = 1.76 - \sqrt{(2.44 - 0.0212Pm)}$$

and, of course, multiplication further by the factor 0.577 gives the corresponding estimate of average degree of thickening θ^*.

The main disadvantage of the U.S. test is that it is less sensitive than the U.K. test for cotton samples that are either

- very immature, when the count of Mature fibres becomes increasingly smaller or, more particularly,
- very mature when differentiation is not critical with very few fibres in the Immature category.

The U.S. test would be expected on statistical grounds to give estimates of average degree of thickening which are less precise than those derived from the U.K. test. In effect the U.S. test is essentially being used to estimate the average of a frequency distribution from the proportions that fall into only two categories. In the U.K. test, more information is available because the average value of θ is estimated from the proportions falling into three frequency groups.

The international standard IS04912 describes both the U.K. and U.S. methods of test and relates one to the other.

5. Strongly Biased Maturity Tests

5.1 Polarized light tests

Various workers [12, 15, 27, 28, 29] have made use of the anisotropic nature of the fibrillar structure of the cotton fibre for assessing fibre wall development. When examined through a microscope illuminated with plane-polarised light, fitted with crossed polariser and analyser and having a first-order red selenite plate to give a magenta background to the field of view, cotton fibres assume different colours according to their wall thickness.

Fibres with very thin walls appear violet or indigo; immature fibres are blue; mature fibres with still thicker walls are yellow.

Counts of fibres based on these effects have been made using several procedures but in general such tests suffer from several disadvantages. These include

- Classing on the basis of colour is partly subjective: partial colour blindness affects the precision of placing fibres in particular categories.
- The random error of classification is increased because the test depends on dividing a continuous distribution of colour grading into finite classes: a temporary change in colour appreciation may alter markedly the fibre ‘maturity count’.
- Like the swelling test, fibre counts in the polarised light test are fairly slow. The test procedure is made still slower by the necessity to align individual fibres, by rotation of the microscope stage, before classification.

Although the method distinguishes to some extent between mature fibres of small perimeter and immature fibres of large perimeter it cannot be accepted that the colour classification grades fibres exactly in accordance with their relative wall thickening, i.e. with θ. The colour appearance depends essentially upon the absolute wall thickness, and hence polarised light tests

for maturity give assessments that vary jointly with degree of wall thickness and intrinsic fibre fineness or perimeter.

The development of the polarised light technique as a basis of the Hungarian *Cotton Grader* eliminated the disadvantages associated with personal classification of individual fibres. It has the additional advantage of surveying specimens composed of a thin layer of fibres instead of single fibres. Nevertheless, it is necessary to prepare and test several such specimen layers to obtain a mean test result of satisfactory precision. The method, however, gives a result that depends not only on the fibre degree of wall thickening but also upon other factors, especially fibre perimeter. Thus, the method works at its best when used for assessing different samples of the same variety, or samples of different varieties that have substantially the same intrinsic fibre fineness. The differences in the instrument readings are then determined more by large differences in fibre maturity than by the smaller effects arising from differences in intrinsic fibre fineness or in optical properties associated with fibre structure.

5.2 Dye tests for maturity

The differential dye test for assessing fibre maturity [10, 26, 41, 42, 43] offers useful scope in several applications. In its usual form, samples dyed in the same bath of red and green dyes are compared visually. Redness is usually accepted as evidence of maturity and a tendency towards greenness denotes a move towards immaturity. The test is clearly useful for detecting a scatter of very immature tufts within a small sample of cotton. But, where it is used to compare different samples, the strong effect of other factors [5] is often overlooked.

The optical appearance of a dyed specimen depends upon the differential absorption and desorption of the two dyes, which in turn depends upon the combination of degree of wall thickening and intrinsic fibre fineness. Conclusions derived from test comparisons and relating to fibre maturity alone are biased according to the fibre fineness. This is illustrated by some experimental data.

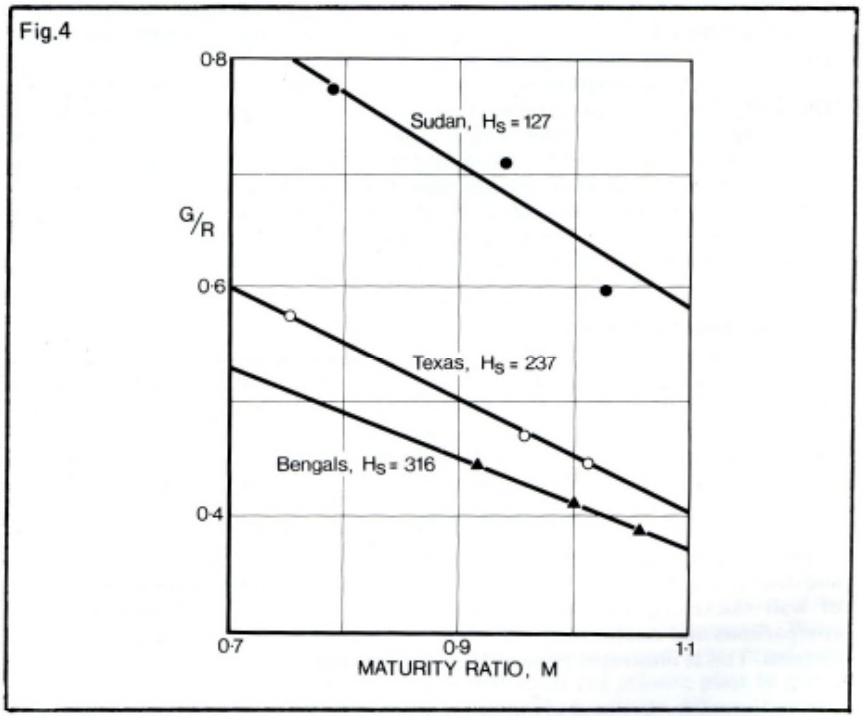
A reflectometer was used to obtain numerical and objective estimates of the colour of test specimens prepared in the form of carefully opened and blended flat pads made from samples dyed in the same bath. Measurements were made of the intensity of light (G) reflected through a green filter and the light (R) reflected through a red filter. The ratio of the two intensities, G/R, is a measure of the colour of the dyed specimen pad; the greener specimens have higher values of G/R than the redder specimens. This procedure eliminates personal errors of assessment and expresses colour on a continuous scale. Several prepared pads were measured for each of a range of cottons differing markedly in maturity and fineness. Routine determinations of average linear density (H) and maturity count (giving the maturity ratio M) were made on the untreated samples of the cottons, usually four independent tests per sample by different operators to obtain accurate average values.

From the full range of cottons a selection was made of three samples of each of three types of cotton. These types differed markedly in intrinsic fibre fineness as assessed by the quantity *standard fibre weight per centimetre*, Hs. This quantity depends only on the square of the fibre perimeter, and is calculated from the fibre test results by the simple relation

$$Hs = H/M \qquad [23].$$

The finest of the three cotton types is Sudan Sakel for which the three samples average Hs = 127 millitex. The samples from the Texas crop were coarser, with an average value of Hs = 237. The Asiatic Bengals *desi* cotton is the coarsest, and gave an average value of Hs = 316.

The three samples of each type differed markedly in fibre maturity. The dependence of the colour ratio G/R on the fibre maturity ratio M is illustrated by the plot of the experimental data in Fig.4.



For each of three types of cotton the greener samples of higher G/R are more immature than the redder samples of lower G/R. However, there is no overall one-to-one correspondence between values of maturity ratio M and values of G/R. Making comparisons at the same value of maturity ratio, the fine Sudan Sakel cotton is higher in the value of G/R than the coarser Texas cotton which, in turn, is greener and higher in value of G/R than the very coarse Bengals cotton. Any acceptance of G/R as a measure of maturity would give estimates that would be biased to an extent depending on the fibre fineness.

It is of further interest to note that the ratio G/R is closely related to surface area per gram of fibre, an estimate that may be calculated from the fibre test figures using the relation

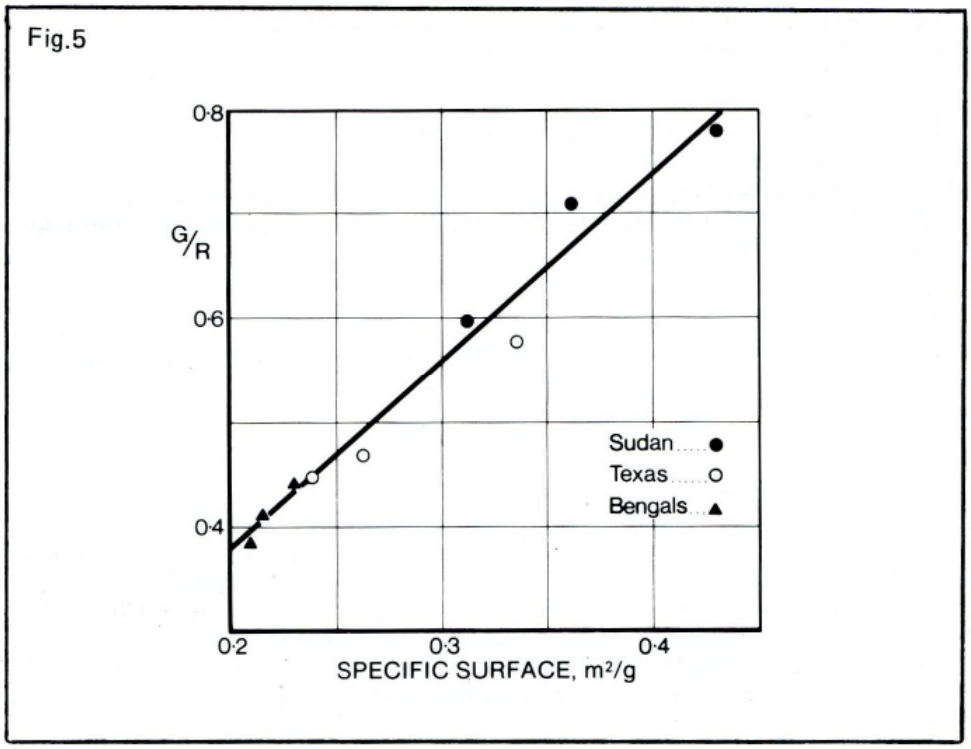
$$S = 3.8 / \sqrt{(MH)} \ m^2.$$

This is illustrated by Fig.5 in which the colour measurements of G/R tend to follow a single relationship with fibre surface area.

It may be added that a plot of values of G/R against calculated values of the absolute wall thickness shows a similar degree of association, but in the opposite direction with G/R decreasing with increasing wall thickness. These results are not unexpected. Dye absorption rates are partly dependent on fibre surface area per unit mass and, after dyeing, the optical appearance is dependent on the fibre thickness.

The full set of test data yielded other information. Some of the cottons gave values for the colour ratio G/R that were not in full accord with the maturity and fineness of the cotton, sometimes being much bluer and sometimes much redder than other samples of

similar fibre test values. This indicates that the dyed shade depends upon not only the size and shape of the fibres but also on other aspects of dyeing affinity such as natural pigmentation, structural features, and minor chemical constituents according to variety and growth.



5.3 Micronaire tests

The Micronaire test is the most widespread instrumental cotton fibre test in use (ISO 2403). Details differ somewhat according to the particular type of instrument used [13, 24], but essentially the quantity determined is an indirect measure of the air permeability of a test specimen of fixed mass contained in a holder of fixed dimensions. Originally it was considered that the air permeability of a specimen was determined by its fibre linear density, its mass per unit length. This approach resulted in the development of the Micronaire scale [30, 32, 37, 38] in units of fibre weight per inch. This interpretation was dropped as fuller understanding of the basic principles of airflow behaviour became more general. However, use of the Micronaire test had then become so widespread that the early curvilinear scale was retained. The numbers marked on the scale are now necessarily regarded as forming an arbitrary scale of air permeability associated with this type of test alone.

Basic theory of fluid flow [9, 11, 16, 33, 34, 35] indicates that the air permeability of a test specimen, measured under the above conditions, should vary inversely as the square of the specific fibre surface So^2 which varies inversely as the product of fineness and maturity, MH. This product MH may be written as M^2Hs (because Hs = H/M) to separate effectively fibre maturity and standard fibre fineness. Hs is the fineness, (H) at unit maturity ratio and normally has units of millitex.

A detailed investigation [17] showed that Micronaire value (X) of a cotton is associated closely with the corresponding fibre maturity and fineness by the relation.

$$MH = M^2Hs = 3.86\,X^2 + 18.16\,X + 13$$

The quadratic equation arises because of the curvilinear relation of the original empirical calibration of the Micronaire scale and also because the use of air at high pressure gives turbulent flow. The equation is tabulated for practical use so that measured Micronaire values may be expressed as estimates of the product M^2Hs. This approach and others enable a more critical interpretation to be made of Micronaire values. Higher fibre maturity (M) and increased coarseness (Hs) may each operate independently to give higher Micronaire values. Lower Micronaire values arise from more immature cotton or from more pronounced intrinsic fibre fineness characterized by a lower value of Hs.

It is fortunate that in most countries a given cotton variety usually varies within only about ±5% in intrinsic fineness Hs. For raingrown crops in particular the maturity ratio may easily range from about 10% above average for good conditions to 20% less than average when it is immature. This variation in maturity has a magnified effect on the Micronaire value which depends on M^2. The Micronaire value may easily range from 20% above its typical value to 40% lower, a range far greater than that caused by differences in intrinsic fibre fineness. For many practical purposes, therefore, differences in Micronaire value are accepted as denoting differences in fibre maturity.

There is the great disadvantage that interpretation of the Micronaire scale of assessment varies according to the type of cotton. The physical significance of a given Micronaire value only becomes apparent if other information is available. This is illustrated by the data in Table 3 which gives fibre test results that were obtained for several samples of cotton that all yielded the same Micronaire value.

Table 3

Values of Maturity Ratio (M) and Intrinsic Fineness (Hs)
for samples with Micronaire test value of 3.5

Country	Type	M	Hs
Egypt	Giza 45	1.00	124
Swaziland	Pima S2	0.96	134
Sudan	GV4S	0.93	144
Sudan	G6B	0.89	160
Brazil	Sao Paulo 6666	0.72	240

The fibre test figures given in the two right-hand columns show the pronounced variation in combinations of maturity and fineness that may occur for samples having the same Micronaire value.

If the Micronaire test is used in breeding some bias is inevitable if additional basic tests are not applied. Continued selection of material of relatively high Micronaire value from breeding lines certainly may give strains of higher than average maturity. But in general, these selected lines will also tend to be coarser than average. This associated pressure towards increased coarseness also tends to give a small reduction in fibre tenacity, because not infrequently there is a loose genetic relation between fibre fineness and fibre tenacity.

6. Reduction of Bias in Fibre Maturity Tests

6.1 Causticaire test

With increasing recognition that the Micronaire value depends upon both fibre maturity and fibre fineness there have been various efforts to develop airflow tests and obtain separate estimates of these two fibre features. One approach was to change the shape of the fibre cross section by mercerizing. A Micronaire test was made first on specimens of the raw cotton followed by further tests after the cotton had been mercerized in caustic soda solution, washed, dried, conditioned, opened and blended. Mercerized cotton fibres have cross-sectional shapes that are rounder than those of raw cotton. Consequently, the air permeability of test specimens varies less with fibre maturity for mercerized cotton than for untreated cotton, so that the relative change in air permeability between the untreated and treated state is smaller for mature cotton than for immature cotton.

The original investigations of the Causticaire method [1, 39] of test showed how estimates of fibre linear density and of fibre maturity may be calculated from the two test figures. Later it was noted [6, 18] that the calculated estimates of fibre maturity were biased somewhat according to fibre fineness. Although this bias might perhaps be removed by a modification in the method of calculation there would still remain some adverse features of practical importance.

- Choice of the specimen density for the treated cotton was limited by the need to obtain readings for as many varied cottons as possible on the restricted flowmeter tube of the Micronaire instrument. The particular specimen densities used in this test do not necessarily give maximum emphasis to the effects of maturity on the relative air permeabilities of raw and treated cotton.
- Features other than fibre maturity and fineness sometimes operate to give appreciable discrepancies between test estimates and directly measured values of maturity and fineness.
- There are serious practical disadvantages associated with specimen preparation.

6.2 Double-compression airflow tests

The Arealometer [14] was the first instrument for estimating both fibre fineness and fibre maturity from airflow observations made on untreated raw cotton. Determinations were made at two different specimen compressions and the scale of the instrument was calibrated in estimates of specific fibre surface. It was noted that estimates of the fibre surface measured at the higher compression are higher than those measured at the initial, lower compression, the difference D between them varying from one sample to another. Trials showed that the value of D increases as the Immaturity Ratio I increases; I being the reciprocal of the degree of thickening θ considered earlier.

Empirical relations were obtained to permit the calculation of separate estimates of fibre immaturity, linear density and also other fibre dimensions, from the two test estimates of specific surface.

Various investigators [7, 20, 22] noted that pronounced differences in Arealometer estimates of maturity and fineness could arise from

- variation in the manner of specimen preparation, even when two operators apparently followed exactly the same test operations;
- repeated handling and re-testing of the material;
- the previous history of the mechanical treatment of the cotton which affected the manner in which it compressed, e.g. blended and opened samples produced from raw cotton, from laps, from slivers, etc. give different test results.

Some of these troubles were considered to arise from the use of a very small test specimen, only 0.152 g, and from the manner of manipulation of the specimen when inserting it into the holder.

The Arealometer is no longer manufactured, and the ASTM standard for its use has been withdrawn.

In general, it was found that sampling and handling errors were reduced by use of a larger test specimen. Some of the earlier investigations [2, 3, 36] concentrated on using the Micronaire instrument for simplicity and because of its widespread availability. A spacer ring was used to make an initial determination, the longer length of the specimen holder giving a reduced specimen density. A second determination was made after removing the ring, which increased the specimen density to the standard value. In several investigations the initial specimen density varied from 2/3 to 4/5 of the second standard density. It was found, that the relative Micronaire instrument readings at low and standard compression were related to measures of fibre maturity obtained from other forms of test. However, the relationships were not satisfactorily high, and sometimes the flow estimates of fibre maturity were biased according to fibre fineness. Disadvantages of using a standard Micronaire instrument in a double-compression airflow test include those associated with

1. The turbulent nature of the airflow through the instrument, the non-linearity of the scale of measurement and the uncertainty of maintaining a constant pressure differential along a compressed specimen. (Note: the check on air pressure is made at zero flow, not under running conditions).
2. The small difference between the initial and final specimen densities that must necessarily be used if tests are to be made on a standard instrument for even a moderate range of cotton types.

Greater possibilities for improving the accuracy of test estimates may be explored by use of equipment specially designed to permit measurement of a wider range of air permeability. The double-compression airflow instrument developed at Rouen by CRITER [4, 25] uses test specimens of mass 5g. The pressure drop along the specimen packed into a holder is measured at a constant rate of flow, first with the specimen compressed to give a density of 0.243 g/ ml and then again after further compression to a density of 0.588 g/ ml and a lower constant rate of flow. From empirical calibration of the instrument the test results may be converted into estimates of specific fibre surface. As with the Arealometer, these two estimates differ, and the difference between them varies fairly closely with fibre maturity.

The CRITER instrument gives results that are more accurate, and more sensitive to the effects of fibre maturity, than those obtained using the Micronaire instrument at two compressions. However, the method of calibration is not wholly satisfactory because groups of material that differ in fibre fineness do not give the same relation between the difference in estimates of specific surface and the corresponding fibre maturity. Thus, although the method has effected an improvement in test accuracy the results are still somewhat biased.

After further development, an improved version of the CRITER instrument was manufactured and marketed, under the name Maturimetre, which enjoyed a limited commercial success. However, the instrument is no longer available.

A project was sponsored by the International Institute for Cotton at the Shirley Institute to develop a trial instrument that would:

1 depend on assessing air permeability of a specimen measured under two different test conditions;

2 use conditions that would lead to optimum test accuracy consistent with ease of test operation;

3 give unbiased estimates of both fibre maturity and fineness.

Experimental apparatus was first developed for measuring air permeability along the axis of compression of a test specimen and also perpendicularly to the axis. The ratio of these two permeabilities was found to vary appreciably with fibre maturity; the magnitude of the differential effect was dependent on the specimen packing density.

However, the greatest accuracy of differentiation between samples of different maturity was furnished by air permeability measured along the compression axis at two specimen densities.

Reproducibility of test figures was found to be improved, as with the CRITER instrument, by measuring pressure difference at constant flow rather than by noting flow at constant pressure difference. This is because manometers can be calibrated over a wide range more accurately than flowmeters.

The choice of the two values of specimen density presented a complex problem. With very low densities (about 0.06 to 0.10 g/ ml) the air permeability was verified to vary closely with the product M^2Hs, a result in accordance with classical theory of fluid flow. In experiments with the specimen density increasing over the range 0.06 - 0.60 g/ ml, the effect of fibre maturity on the air permeability was found to become more pronounced than indicated by simple flow theory. However, the effect was not simple and as the specimen density was increased there was found to be a small but significant increasing effect of intrinsic fibre fineness Hs. This secondary effect also needed to be taken into account to avoid bias. An empirical and better description of the flow phenomenon was obtained by relating air permeability not to M^2Hs but to an expression of the form

$$M^{n1} \cdot Hs^{n2}$$

where n1 and n2 are parameters that increase steadily above the values 2 and 1 as the specimen density is increased.

An unfortunate consequence of this joint varying effect of maturity and fineness is that it is necessary to use complicated equations for estimating these two quantities from test results obtained at two specimen compressions. If for a given test specimen, the pressure difference P1 is noted at a constant flow for the initial lower specimen density, and P2 is the pressure difference measured after compression to the higher specimen density and using different but constant flow, the estimate of maturity ratio M is obtained from an expression of the form

$$M = constant \cdot (P_1 / P_2)^{k1} \cdot P_2^{k2}$$

For convenience a second similar relation (with different values for the parameters) is used to obtain an estimate of the average linear density H of the sample. The standard fibre weight per centimetre Hs is obtained from the usual relation Hs = H/M.

Use of a very low and a very high specimen density gave the maximum differential effect of maturity on the relationship between the two measured air permeabilities. However, values of P1 and P2 measured at very low and very high specimen densities do not give the most accurate estimates of fibre maturity ratio M. Test variation is often pronounced with specimens of very low density because of irregular fibre arrangement. Indeed at 0.06 g/ ml the specimens may easily be permanently deformed by a sudden surge of air. At very high densities test variation also became pronounced, because irregularities from place to place became increasingly important in determining the air permeability. The use of intermediate densities was studied in detail. The standard error of the difference between airflow estimates of M and direct swelling test values was found to fall to a minimum in the region 0.2 g/ ml for the initial compression and 0.4 g/ ml for the high compression.

For a group of 100 cottons examined on a prototype machine the correlation coefficient between predicted airflow values and direct test values was computed to be r = 0.934 for maturity ratio M, r = 0.994 for average linear density H, and r = 0.934 for standard fibre weight per centimetre Hs. The corresponding coefficients of variation of the differences between predicted and direct test values were 3.8%, 3.5% and 5.5%.

These values for the coefficients of variation, although not unduly large, are all somewhat larger than might be expected by chance from random sampling and test variations in the quantities involved. The effect of increased variation was not uniform. A small minority of the samples gave aberrant results that persisted even when repeat tests were made using different test specimens. Other cotton samples, in contrast, always gave results in accordance with expectation from the direct fibre test results. The conclusion is clearly that, although maturity and fineness largely determine the air permeability of specimens at different compressions, there are small disturbing effects that arise from limited variation in other fibre features, of the type instanced in 6.1(b).

For this and indeed for any dual-condition test it is essential to prepare samples in a standard manner, to blend the fibres and make them uniformly open. The airflow estimates of the fibre characters are based on differential and not major effects, and consequently high test reproducibility is far more essential than in a single-condition airflow test. Mechanical opening must be carried out, for example by using a miniature card or fibre blender to give a fleece or web of substantially random orientation. Raw cotton of variable degree of openness gives test estimates of maturity and fineness of low precision.

7. Summary and Discussion

1. The nature and origin of fibre maturity is outlined, and emphasis is given to the large fibre-to-fibre variation in fibre wall development which is masked when maturity is characterized by a measure of the average level only.

2. Assessment of maturity by hand and eye is subjective and has the additional disadvantage of being partly dependent upon other features of a sample. However, it is rapid and a classer should note always the various adverse aspects that indicate probable low maturity when examining samples for other characteristics such as grade and staple. An expert examination of a sample can reveal information that otherwise could only be obtained laboriously by use of several types of test.

3. A small amount of excessively immature, dead cotton in a sample of average maturity may cause pronounced neppiness or dyeing trouble. An experienced classer can often detect such cotton: fibre tests measuring average maturity fail to discover this potential serious trouble.

4. Fibre maturity is best described in terms of the *degree of wall thickening*, which is defined as the ratio of the area of actual wall thickening to the area of potential wall thickening if all fibres were of round, solid transverse cross section. This quantity is a measure of shape: it is independent of intrinsic fineness as assessed by the fibre perimeter.

5. Estimates of degree of wall thickening are made most accurately by use of the long-established tests based on classing fibres according to their shape under the microscope after swelling in caustic soda solution. The *maturity ratio* may be calculated from the test results. It is a quantity that serves for everyday practical purposes and is directly proportional to the degree of wall thickening.

6. The disadvantages of the caustic soda swelling test are mainly slow speed of testing and the need for skill and attention of the test operator. The advantages are that the method provides the most direct and most unbiased measure of maturity available for critical quality assessment.

7. The need for rapid estimation has stimulated the development of tests that assess maturity from determinations of the values of other fibre features which themselves are affected by maturity. These test methods are still less direct, and include assessment of fibre appearance in polarised light, appraisal of the shade of dyed specimens, and measurements of the air permeability as in the Micronaire test. The results of these tests are also affected by the fibre fineness of the cotton samples and consequently the assessments of relative maturity are biased. But, in applications when large fineness effects are unlikely, the simplicity and the speed of the Micronaire test have made the method widespread in commerce and industry.

8. Many efforts have been made to separate the effects of maturity and fineness, especially by making an airflow test at two different specimen densities. The salient features of these methods are outlined.

9. The disadvantage of any double airflow technique is that the differential effects of maturity and fineness are much smaller than the major effects. Therefore, it is essential that the accuracy of measurement in such a test shall be greater than the accuracy of measurement in a one-quantity test. Furthermore, because the method depends upon small differences in the flow pattern at different specimen compressions, it is essential to avoid unwanted specimen differences by preparing uniformly opened samples by use of a miniature card or fibre blender.

10. In double-compression airflow test methods the estimates of fibre maturity are biased somewhat according to the fibre fineness unless a complicated mathematical treatment is adopted to describe the intricate flow phenomena. This paper shows how the bias may be avoided and how separate estimates may be made of fibre maturity and fibre fineness measures.

11. In both single- and double-compression airflow methods the air permeability under a given set of conditions is not determined solely by the fibre maturity and fineness. Cotton is a biological material and inevitably there is some limited variation in other dimensional features or shape characteristics, for example in overall fibre density and

in distribution of fibre shapes for a given average fibre maturity. Therefore, any assessment of fibre maturity or of fibre fineness is of lower reliability than one made by a more direct method of test which is less dependent on these secondary variants.

12. Unfortunately, there is no single bulk feature of a cotton sample that is determined entirely by fibre maturity alone. There is no combination of two bulk features determined only by different functions of maturity and fineness. Other fibre features, or features of the fibre arrangement in a test specimen, also operate. Hence any test measurement of a bulk feature or features can provide only *estimates* and not *measures* of maturity or fineness.

13. The various tests discussed all have useful application in different circumstances, especially for assessing cotton quality in cotton breeding, marketing and textile manufacture. Rapid tests are essential when dealing with large numbers of samples in cotton marketing and therefore only simple tests are feasible. But the cost of making a simple rapid test is an inevitable loss of relevant information and precision. This cost is often acceptable in commerce when the only alternative to a simple test is no test at all.

In cotton breeding the objective is to eliminate unsuitable material at the source, to choose acceptable cotton for growing on a wide scale. Under these circumstances the cost of making more elaborate tests to obtain a larger amount of more critical information, more accurate information, but more expensive information, should be regarded as acceptable because it forms but a small part of the costs of breeding.

Between the commercial field where cheapness and speed of testing is essential, and in the final stages of cotton breeding where accuracy and full information is essential, there are many fields in which somewhat slower tests of but moderate precision can be employed usefully, especially where there is the need to apply a ‘selection pressure’ designed to advance fibre maturity. Appraisal of the final evaluations by more critical tests can always be made at a later stage using the more accurate fundamental tests.

8. Recent Developments in The Measurement of Maturity

Since this booklet was first published, in 1975, there have been several significant developments and some important publications in the field of cotton fibre maturity testing.

Rather than intrude upon Mr. Lord's original text, which has received only minor editorial changes, this additional section has been prepared in an effort to summarise the most important developments since 1975.

It covers three main subject areas:

1. Basic research work using microscopical and image analysis techniques.
2. New testing methods based on the Digital Fibrograph and on near infra-red reflectance instruments.
3. Further development and evaluation of the new double-compression airflow instrument mentioned in section 6.2, which was originally developed by Mr. Lord and subsequently launched commercially as the IIC-Shirley Fineness & Maturity Tester.

The review is not intended to be exhaustive, either in scope or in treatment, but it is hoped that the most important developments have been covered.

8.1 Microscopical studies

Despite the tricky and arduous nature of direct microscopical measurements of fibre dimensions, several workers have ventured into the field recently.

Stephens [101], after discussing the difficulties in preparing proper cross-sections, contends that it is possible to measure the wall thickness of small lengths of unsectioned fibres accurately if the cut fibre segments are mounted in a medium which allows them to be flattened in one plane and which makes the boundary of the lumen clearly visible. Several mounting media were tested and, although none was totally satisfactory, the best results were found with a mixture of 3 parts 85% lactic acid to 1 part isopropyl alcohol. Measurements of maximum ribbon width, Fl, and (flattened) lumen width, L1, were made on about 200 segments taken from each of the ten International Calibration Cotton Standards and the results were compared with the corresponding Arealometer readings. Excellent correlations were obtained between the two basic Arealometer readings, A, (A+D), and the expressions:

$$F / (F^2 - L^2) \qquad \textit{and} \qquad F1 / (F^2 - L^2)$$

where F and L are the diameter and the lumen width, respectively, of the equivalent uncollapsed circular fibre and are calculated from the measured ribbon width, F1 and lumen width, L1 via the expressions:

$$L1 = \pi . L/2 \qquad \textit{and} \qquad (F1 - L1) = (F - L)$$

The derived values for F and L were also used to calculate the relative wall thickness, as

$$RWT = 100(1 - L/F) \ \textit{per cent}$$

and their values were compared with estimates of fibre diameter and relative wall thickness derived from Hertel and Craven's I and P formulae for the Arealometer [102]. Very poor agreement was found and Stephens suggests that the Arealometer formulae for I and P may be significantly in error.

In a later paper [103] he presents a table for converting Arealometer A and D values to estimates of fibre width and relative wall thickness using the regression equations found between A and (A + D) and the expressions given above, which are given the symbols S and R, respectively. Thus:

$$F1 / (F^2 - L^2) = R = [110.358 (A+D) + 24719] . 10^{-6},$$

and

$$F / (F^2 - L^2) = S = [94.166A + 22055] . 10^{-6}$$

Fibre width and relative wall thickness are then estimated from:

$$F = S / \{S^2 - [1.7519(R-S)]^2\} \ \textit{micro metres}$$

and

$$RWT = 100 - [175.19(R-S) / S] \ \textit{percent}$$

Stephens appreciates that these relationships are only valid when the fundamental assumption holds true, namely that the mean projected width of a convoluted, collapsed fibre is the same as the diameter of the equivalent circle. Whenever there is pronounced incurving of the cross-section, as can happen with immature fibres, then this relationship breaks down.

In a further paper [104] the R and S parameters derived from the microscopical measurements are shown to be very closely related to the P_L and P_H values obtained from the IIC-Shirley Fineness & Maturity Tester.

It should be noted that Stephens' definition of relative wall thickness, RWT corresponds to that of Du Bois [105], and is not the same as Pierce and Lord's degree of thickening, θ. RWT is the ratio of wall thickness to diameter of the equivalent circle whereas θ is the ratio of wall cross-sectional area to the area of the equivalent circle.

Workers at the De Meulemeester laboratories [106, 107, 108] have also preferred to use relative wall thickness. In a very important paper [107] they present the results of large numbers of microscopical measurements made on cross-sections of unswollen fibres, as well as on longitudinal views of fibres swollen in an 18 percent solution of sodium hydroxide for a range of 31 cottons. Frequency distributions and cumulative frequency curves of RWT are presented for a few of the cottons and it is claimed that for nine different varieties, the cumulative frequency curves all have the same shape which is given by an equation of the form:

$$f = 1 - exp\ [-a(RWT)^b]$$

For measurements on unswollen fibres, correlation coefficients were generally around 0.99 and always better than about 0.95. For the measurements on swollen fibres there was more scatter but still the correlation coefficients were always better than about 0.91. The values for the coefficients, a, and the exponents, b, were different for the different cottons, but both seemed to be functions of the mean relative wall thickness.

Furthermore, it was found that, across the 31 cottons, the relationship between the means of the relative wall thicknesses and the proportion of mature fibres followed very closely a similar type of curve.

The relationships given were:

for unswollen fibre cross-sections;

$$m = 1 - exp\ (-2.425X^{2.9} \cdot [1 - X]^{-1.0})$$

where,

- m is the proportion of fibres having a relative wall thickness (2e/D) equal or greater than 0.5
- e is the measured wall thickness
- D is the diameter of the equivalent circle, calculated from the measured perimeter
- X is the mean relative wall thickness

and, for swollen fibre longitudinal views,

$$M = 1 - exp\ (-2.245X^{2.093} \cdot [1 - X]^{-0.324})$$

where,

- M is the proportion of fibres having a relative wall thickness (2E/W) equal or greater than 0.5
- E is the measured wall thickness
- W is the measured ribbon width
- X is the mean relative wall thickness

The definition of M is, of course, the same as that used in the Standard ASTM method for determination of the percentage of mature fibres, Pm.

The coefficients and the exponents for these two equations are not too dissimilar and, in fact, if one extracts the mean values for m and M given in ref [108] and plots them on the same graph against the means of 2e/D or 2E/W, respectively, for the 31 cottons, then it is easy to imagine that both the swollen and the unswollen data follow the same basic curve.

Whether or not the mean RWT values for swollen and for unswollen fibres follow the same curve, these results lead to the hypothesis that, at least for these cottons, the entire distribution of maturity can be generated from a knowledge of one parameter, the percentage of mature fibres. The hypothesis is actually quite consistent with the findings of Pierce and Lord [120] concerning the joint variation of perimeter and degree of thickening, but in order to confirm it, it would be necessary to establish:

a) that the relationship between the percentage of mature fibres and the mean of 2e/D is in fact a unique one for the whole range of commercial cottons;
b) that the coefficients and exponents of the cumulative frequency distributions for individual cottons are indeed a close function of, and only of, the mean of 2e/D;
c) that, furthermore, these coefficients could be predicted from a knowledge of some basic fibre property such as the mean perimeter, or Standard Fineness.

In order to make practical use of the relationships one would then have to develop sound estimates for the various coefficients and exponents. This would be an extremely arduous work but, once it had been accomplished a rather powerful tool would be made available for predicting such interesting properties as the percentage of immature fibres, nepping potential, fibre breakage potential, maximum bundle and yarn strengths, rate of dyeing and colour yield to name only the most obvious.

At about the same time, workers in India were also making microscopical measurements on fibre cross-sections of cottons having a range of maturity and fineness [109]. They have presented frequency distributions of perimeter, cell wall thickness and cell wall area, and have concluded that these conform approximately to a normal distribution. In a later paper [110] the changes in these parameters at weekly intervals during growth were reported for four varieties (one from each major species) and again approximate conformity to the normal distribution at each stage was shown.

Recently, Peeters *et al*, have made cross-sections of never-dried fibres [111] as well as dried and reswollen fibres [112]. They conclude that the original shape of the never- dried fibres is indeed truly circular and that measurements of relative wall thickness are most reliably made on the never-dried materials. Although this is not practical for characterising the fibres of commerce, it could be a useful tool for cotton breeders who have access to the ripe but unopened bolls.

A further conclusion of importance was that measurement made on longitudinal views of dried fibres reswollen in caustic soda were inherently unreliable due to optical effects and to the often folded and incurved nature of some cross-sections.

Taken as a whole, the few detailed microscopical studies which have been undertaken in the last decade have significantly deepened our understanding of the meaning and the measurement of maturity. It is a pity that such studies are so difficult and arduous as to be infrequent and one can only hope that further progress in the development of electronic automated procedures will increase their number over the next decade.

8.2 Image analysis

One of the most important and difficult problems in developing or evaluating new techniques or instruments for measuring maturity is the question of calibration.

As mentioned in Section 3, the only acceptable fundamental unbiased estimate is obtained by direct measurements of wall thickness or area and of perimeter of large numbers of very carefully prepared fibre cross-sections.

Since the direct method is so difficult and time consuming it is very seldom used. Even Pierce and Lord's original derivation of the relationship between θ and the proportions of normal, immature, and dead fibres was based mainly on only 15 cotton samples, and has since been challenged [113]. Therefore, calibration exercises are normally carried out using indirect methods such as the counting of swollen fibres or the use of double-compression airflow instruments. It is not unusual to see new techniques being evaluated by indirect, and possibly biased instruments which themselves were originally calibrated by an indirect method.

Thus, any attempts to make the direct method of measurement simpler and faster, without loss of accuracy, are to be welcomed and several workers have taken steps in this direction by the use of automated or semi-automated electronic measurement systems.

Herbert et al [114] used a graphic digitiser to trace the outlines of individual fibres on photomicrographs of sections prepared with a Hardy hand microtome. The digitised data were processed by a computer programme which determined both perimeter and cross-sectional area. On each photomicrograph 25 fibres could be measured in 15 minutes.

Worley et al [115] used a πMC particle measurement computer system to measure the cross-sectional areas of individual fibres prepared by embedding yarns in an epoxy resin before slicing sections with a glass-bladed ultra microtome. The sections were stained with crystal violet to increase contrast. Individual fibres were outlined with a light pen and the computer calculated the area of cross-section. Comparisons were made with Micronaire, Arealometer, Causticaire, and Fibrograph Maturity for two separate groups of cottons.

Gilhaus and Lünenschloss [116] describe briefly both semi-automatic and fully automatic image analysis systems for measuring cross-sectional areas of fibres. They measured the cross-sectional area, A, and the perimeter, P, of the ten International Cotton Calibration Standards, and transformed these into a form factor, defined as:

$$FOR = 4\,\pi\,A/P^2$$

which is equivalent to Pierce & Lord's degree of thickening, θ, being the ratio of the area of cross-section to the area of the circle having the same perimeter.

Frequency distributions of FOR were given for three of the cottons and the average of FOR was compared to the Maturity Ratio, MAT, measured with the IIC-Shirley FMT. From Pierce & Lord's original definition of Maturity Ratio, the slope of MAT against FOR should be 0.577. Gilhaus does not give his regression coefficients but a few measurements made on Figure 8 of his paper reveal an average slope of about 0.65 if the regression line is made to pass through the origin. This result is actually quite consistent with Neelakantan's reinterpretation [113] of Pierce and Lord's original data [120].

Gilhaus states that the measurement of 100 fibre cross-sections takes about 20 minutes with this technique.

Berlin et al [117] have reported further investigations with the πMC particle measurement computer and have addressed the problem of obtaining adequate sections as well as the reproducibility of their technique. They conclude that a hand microtome is not satisfactory and recommend a rotary microtome with a glass knife. Provided that the instrument is properly calibrated, an ultra-low viscosity medium is used for embedding, the sections are about 1.5μm thick, and they are stained with crystal violet, then the area measurements can be reproduced to within ±2.3%. About 500 fibres must be examined. Unfortunately, the perimeter could not be measured.

Barker and Lyons [118] also used the πMC instrument to measure the maximum and minimum ribbon widths from longitudinal views of fibres to derive an index of cross-sectional circularity, and a similar approach was taken up by Thibodeaux and Evans [119] who used a more sophisticated image analyser, from Cambridge Instruments, giving much higher resolution. Pierce and Lord [120] had already pointed out that the mean projected diameter of a convoluted fibre is equal to Perimeter/π, the same as for a circle, provided that the cross-sectional shape is not excessively incurved (as is often the case for immature fibres) when the mean ribbon width will be significantly less than P/π.

Thibodeaux and Evans measured the mean projected diameter as well as the maximum and minimum ribbon widths on a range of five cottons whose Arealometer values were well known. They found excellent correlation between the Arealometer-derived perimeter, Pa, and mean projected diameter, Fm. Averaged over the five cottons, the ratio π.Fm/Pa was 0.93 showing a discrepancy of about 7% in estimating fibre diameter,

This "error" is at least as likely to arise from the Arealometer estimate of perimeter as from the direct measurements, [101] but could presumably also contain a component due to incurved fibre cross-sections.

The ratio of maximum to minimum ribbon width was taken as an index of maturity, Im, and was compared with the Arealometer Immaturity Index, Ia, An excellent, correlation was found, but an equally good correlation obtained between Ia and Fm, showing that the joint variation in maturity and Standard Fineness had been too narrow in the five cottons for a proper evaluation of the usefulness of Im to be made.

Thus, although individual workers have made valuable contributions to the field of image analysis there is still a great deal to be done before the direct method of calibration is brought within the reach of the majority.

8.3 Optical Density - the Fibrograph Method

In 1972 Gridi-Papp and Sabino [121] proposed that, when the optical density of a beard of cotton fibres is estimated on a Digital Fibrograph instrument, then the ratio A^2/W should be proportional to the average maturity of the fibres in the beard. A is the "amount" reading from the Fibrograph and W is the weight of the beard. For a series of 59 cottons the optical density measurements were compared with the percentage of mature fibres, Pm, measured by counting caustic-soda swollen fibres under the microscope. The best-fit expression for maturity was given by a relationship of the form:

$$log\ (Pm) = a - b\ (A^2/\ W)$$

At about the same time Krowicki and Duckett were looking for a rapid method to estimate the mass of a bundle of fibres and they studied the relationship between mass and optical density, also using a Digital Fibrograph [122]. For a range of nine cottons, these authors found that, in

order to predict the mass accurately from Fibrograph amount readings, it was necessary to introduce a correction for the square root of the average cross-sectional area of the fibres (estimated from Arealometer measurements).

Gridi-Papp and Sabino's work was followed up by Gutknecht [123] who found that it was necessary to hold the amount value, A, to within strict limits by limiting the size of the beard and he standardised his results on an amount value of A = 750. In order to do this, it is necessary to apply a correction to the average value of A^2/W obtained for a given cotton. For a range of 98 different cottons Gutknecht deduced the relationship:

$$IOM = A^2/100W - 0.0918(A - 750)$$

where IOM is an Optical Index of Maturity and the A, and the A^2/W are the averages of a series of (preferably ten) measurements on a given cotton, IOM was found to have a good correlation with maturity, as measured by the Maturimetre instrument but the best-fit regressions were found to be different for *Hirsutum* and *Barbadense* types. Correlation coefficients were of the order of 0.87 for *Hirsutum,* to 0.90 for *Barbadense.* For the *Hirsutum* series it was found that the correlation could be slightly improved by taking account of the 2.5% Span Length, a finding which was later supported by Worley *et al* [115].

As a result of the early studies, a test method was proposed by the Spinlab company [124] and an ASTM Standard was issued, in the latest version of which the following formula is given [125]

$$FM = 176 - (1.23A^2/W + 1.3L)$$

where

FM is the Fibrograph maturity index

A is the amount reading times 100

W is the beard weight in mg times 100

L is the 2.5% span length.

For this test, only specimens whose amount reading falls between 7.25 and 7.75 volts are to be accepted, thus obviating the need for Gutknecht's correction to be made.

Meanwhile Krowicki and Duckett had continued their work on the relationship between the optical density and the mass of a bundle of cotton fibres using a fine (1.5 or 0.8mm) laser beam to scan across a fibre bundle [126]. They defined total absorption of light as the area under the Intensity / distance curve and found an exponential relationship of the form,

$$Area = a + b.\ exp(km)$$

where m is the specimen mass and the constants a, b, k are specific to the particular optical system, and are influenced by the geometry of the bundle and of the fibres.

A similar relationship was found by Gutknecht [127] in an important paper which seems to have thoroughly elucidated the practical meaning of the Fibrograph measurements. For a wide range of cottons Gutknecht studied the relationship between the amount value, A, and the weight of the fibre beard, W. He found that a simple exponential function:

$$W = a\ .\ exp(bA)$$

gave the best form for this relationship, but that the values for the coefficients a, b varied widely according to the cotton type. Gutknecht then attempted to relate the Fibrograph

measurements to Micronaire, Maturity, and Standard Fineness as estimated using the IIC-Shirley Fineness & Maturity Tester. He found that, among his 87 different cottons, having a wide range of both Standard Fineness and Maturity, the best explanation for the variation in Fibrograph readings was the Micronaire value, in combination with the 2.5% span length. Correlations involving the Maturity were very poor unless the Standard Fineness was also included or unless consideration was restricted to groups of cottons having similar levels of Standard Fineness.

This conclusion, arrived at by extensive experimental work, is supported by the earlier theoretical considerations and more limited measurements of Krowicki and Duckett [128] who had by this time extended their analysis to deal with uniform hollow cylinders and confirmed that the relationship between mass and optical density should include a term for the square root of the cross-sectional area or the average specific surface area of the fibres. In a later paper [129] their basic theoretical expressions were reduced to the form:

$$A = Co\ (M/V)^2 \qquad \textit{and} \qquad P/A = C1\ .\ (V/M)$$

where

A is the average cross-sectional area of the fibres

M is the mass of the Fibrograph beard

P is the average fibre perimeter

V is an expression which approximates the total light absorption of the beard

Co and Cl are constants

On a series of 18 cottons, estimates of cross-sectional area and specific surface were compared with corresponding values obtained from Arealometer measurements. Correlation coefficients of up to about 0.98 were obtained, depending on the expression used to arrive at the optical density function, V. Correlations with Maturity Index from Causticaire or Arealometer measurements were also quite good.

Nayar et al, by assuming that the Fibrograph 100% amount reading is proportional to the product of the total number of fibres and the average fibre diameter, have proposed an Optical Fineness Coefficient (130) defined as:

$$OFC = (W/AL)^2$$

where

W is the weight of the beard

A is the Fibrograph amount reading

L is the 2.5% span length.

For a range of 44 cottons, including all four commercial species, the OFC was found to be closely correlated with gravimetric fineness, with r = 0.97, thus supporting Krowicki and Duckett's suggestion that Fibrograph measurements can be related to the average cross-sectional area of the fibres.

Finally, Gilhaus and Lünenschloss have shown that the Fibrograph Maturity is very highly correlated (r = 0.99) with the specific surface area measured directly on fibre cross-sections with an image analyser system [116].

Thus, it seems that the Fibrograph Maturity Index, as presently defined, is actually an indication of the average fibre cross-sectional area, or of the average fibre specific surface. It is therefore an estimate which is biased in the same way as the Micronaire. Provided that its use is confined to cottons having similar Standard Fineness (similar perimeter) as is often the practical situation, then the Fibrograph could return useful indications of differences in maturity. For an unbiased estimate, other methods must be used.

8.4 Near infra-red reflectance

The use of near infra-red (NIR) reflectance measurements was proposed by Ramey [131] for estimating the cross-sectional area and the specific surface of cotton fibres.

According to Weyer [132] the absorption bands in the NIR region are due to overtones and combinations of the fundamental mid IR molecular vibration bands. The NIR region is therefore rather complex, with many band assignments unresolved. The most prominent overtone bands are those related to O-H, C-H and N-H groups. The first two are common in cellulose, the third in protein which will be present in raw cotton. NIR reflectance instruments are designed to gather reflected light very efficiently, during a scan over a range of wavelengths, and are combined with a computer which analyses the collected spectral data. The reflectance readings are first converted to a form which is approximately linear as a function of concentration, namely the logarithm of their reciprocals. Calibration is effected by obtaining sets of log (1/R) readings for a series of samples having known values for the required property and an empirical calibration equation is derived automatically by the computer. It is of the form:

$$Y = Ko + K1 \log (1/Ra) + K2 \log (1/Rb) \ldots\ldots$$

where

Y	is the property to be estimated
Ko, K1, K2 …	are the regression coefficients
Ra, Rb …	are the reflectance values at the chosen wavelengths

With some instruments a more complicated treatment of the reflectance data is possible in order to allow for variations in sample presentation and in particle size, both of which can affect the results. Thus, the initial calibration series is of vital importance. An excellent discussion of calibration techniques has been given by Montalvo et al [136, 136a].

Ramey [131] using a Neotec model 41 Grain Quality Analyser, found good correlations between NIR measurements and Micronaire, Causticaire Maturity Index, Arealometer readings, and cross-sectional area measured with an image analyser. Correlation coefficients were all greater than 0.90. It was estimated that the technique is rapid enough that about 150 specimens per day could be handled.

This work was followed up by Ghosh [133, 134] who used a Technicon model 400TX instrument calibrated against the Causticaire values of a range of US upland cottons. Good correlations were shown between NIR readings and Causticaire Maturity Index, IIC-Shirley FMT maturity, and colour yield after dyeing. Correlation coefficients were in the range 0.92 to 0.96.

Price and Smith [135] have compared the results from two such Technicon instruments, calibrated according to Ghosh [134], with measurements of Micronaire, IIC-Shirley FMT

maturity, Causticaire Maturity Index, and percent mature fibres by the US standard caustic soda swelling and counting method using 36 cottons with maturity ratios from 0.6 to 1.01 and standard fineness from 134 to 236. They conclude that for these cottons NIR measurements are most closely correlated with the Micronaire value rather than the maturity. They suppose that reflectance is governed by the total surface area and that absorption is governed by the fibre wall thickness. Since neither of these in itself is a direct indicator of maturity it is perhaps not surprising that Micronaire value yields the best correlations.

More recently, Montalvo et al have reported [136] on a series of measurements made with an NIR instrument from the Neotech Division of Pacific Scientific Co. on two series of cottons. One series represented different stages of growth, 3½ weeks, 5 weeks, and 8 weeks, for two varieties, where it was found that there was no change in the shape of the NIR spectrum with maturity but that the main absorption peaks were intensified. The second series comprised three varieties (American, Egyptian and Greek) each at three levels of maturity. For these samples data were available from the IIC-Shirley FMT and also from image analyser measurements. It was concluded that the NIR results were correlated with the absolute wall thickness.

Extraction of the raw fibres with solvents, to remove non-cellulosic matter, intensified the peaks but destruction of the fibre shape by ball-milling completely removed any correlation with fibre properties.

Enough work has been done with NIR instruments to show that the technique has potential for measuring some aspect of maturity and that it can be extremely rapid. However, the definitive study remains to be done. In particular it will be necessary to establish a proper calibration with a range of cottons having the necessary amount of variation in *both* maturity and Standard fineness. A range of cottons, moreover, which has been adequately characterised by a more direct measurement of maturity than the Causticaire method or even the IIC-Shirley Fineness & Maturity Tester.

8.5 IIC-Shirley Fineness & Maturity Tester

In section 6.2, the development to prototype stage of a new double-compression airflow instrument was described. Since that time the instrument has been commercially released under the name IIC-Shirley Fineness & Maturity Tester, which is often abbreviated to FMT, and a standard test method has been issued by ASTM; method D3818-1979.

The specimen has a weight of 4.000 ±0.005g. At the lower level of compression, its density is 0.1911 g per cubic cm and the airflow is set to 4.0 litre/ min. At the higher level of compression, the sample density is 0.3821 g per cubic cm and the airflow is 1.0 litre/ min. The pressure drops, P_L and P_H are recorded at each condition in turn and are converted into Micronaire equivalent (MEQ), Maturity Ratio (MAT), and Fineness (FIN) using the following empirical expressions:

$$MEQ = 0.6 + 850 / (P_L + 40)$$

$$MAT = 0.247 \, . \, P_L^{0.125} \, . \, (P_L / P_H)^2$$

$$FIN = 60000 / P_L \, . \, (P_H / P_L)^{1.75}$$

The Maturity Ratio, MAT, refers to the British standard caustic soda classification procedure BS 3085, as described in section 4.1, but a conversion is available to give the percentage of mature fibres by the American method, ASTM D1442, using the equation given in section 4.2.

In the original version of the instrument, P_L and P_H values were converted to MEQ, MAT, and FIN by use of tables or a programmable calculator but the modern versions have a small computer attached which automates the whole data logging and analysis procedure.

Since the appearance of the instrument, several studies have been carried out which either investigated the effect of specimen preparation variables or compared the results from a FMT with those obtained by other methods. Unfortunately, many of the comparisons were made with methods which themselves are biased, such as Causticaire, or were carried out on a range of cottons which was not wide enough (in terms of both maturity and Standard Fineness) to yield a proper evaluation of the usefulness of the FMT. An inadequate range of cottons is betrayed by high correlations between Fineness and Maturity, or between Micronaire and Fineness, or between Micronaire and Maturity. Indeed, Gutknecht has argued [137] that the best estimates for maturity given by an airflow instrument are those which yield the lowest correlations with Micronaire (when an adequate range of cottons is investigated).

It has been confirmed [138, 139, 140, 141] that sample preparation is extremely important. The specimen must be clean, well opened and thoroughly randomised.

All comparisons of MEQ with Micronaire have shown extremely close agreement [138, 139, 141, 142, 143, 144] and it is clear that the FMT delivers a true Micronaire equivalent. This is not surprising since the test conditions at the low level of compression on the FMT are very similar to those on the Micronaire instruments.

Comparisons of FMT MAT results with the Causticaire [138, 141, 142], or Arealometer [139, 104, 142, 143, 108] Maturity Indexes tend to give mediocre correlations but Stephens [104] and Raes [108] have demonstrated a very close correlation between the basic Arealometer readings A and (A+D) with P_L and P_H.

Comparisons of FIN with gravimetric fineness, H, and of MAT with Maturity Ratio, M, (British method) or the Percentage of Mature fibres, Pm, (American method) have shown fair to excellent correlations [108, 116, 141, 142, 144, 145, 146]. In general, it is found that FIN and H are better correlated than MAT and M or Pm. However, as both Mitchell [145] and Raes [108] have pointed out, it is not altogether clear whether the lower correlations with maturity are due to deficiencies in the FMT or to uncertainties in the standard caustic soda classification or microscopical techniques. The most comprehensive evaluations have been carried out by Mitchell [145], by Raes [108], and by Gutknecht [137].

Mitchell has used 30 cottons having a wide range of both maturity and Standard Fineness whose values for Maturity ratio, M, and Gravimetric Fineness, H, were well established because of multiple tests carried out over many years on the same samples. When these cottons were tested with the FMT, very close agreement was found. Between MAT and M, the correlation coefficient was $r = 0.986$; between FIN and H it was $r = 0.990$.

Mitchell also recalls the relationship between Micronaire value and the product MH, which was described in section 6.3, and he compares the measured products with those calculated from Micronaire measurements on the same range of cottons. Again, very close agreement was found, $r = 0.971$.

Heap [144] has also studied this relationship for the FMT instrument. The ten International Calibration Cotton Standards were measured on 15 different FMT instruments and, in addition, a series of 22 experimental cottons, having a good range of standard fineness were measured on two different FMT instruments. The standard Micronaire, Maturity Ratio, and Gravimetric Fineness measurements were also available.

It was found that, for the relationship between the standard values, Lord's original, equation, namely

$$MH = 3.86\,X^2 + 18.16\,X + 13.0$$

where X is the Micronaire value, was a good fit for the data. However, for the results of FMT testing, slightly different coefficients gave a better description. After averaging over all fifteen FMT instruments, the following equation emerged.

$$MAT.FIN = 2.07\,MEQ^2 + 32.09\,MEQ - 12.68$$

this equation yielded a correlation coefficient between measured and calculated values for MAT.FIN of r = 0.999.

Raes and Verschraege [108] have used a group of 30 cottons to compare the results from a FMT instrument with those obtained from direct measurements of relative wall thickness, RWT, made on unswollen cross-sections as well as on longitudinal views of fibres swollen in 18% caustic soda solution. Measurements were also made with an Arealometer instrument. The agreement between the percentage of mature fibres, Pm, derived from the microscopical measurements and that derived from MAT was said to be good but it was claimed that better agreement could be found by using alternative relations such as:

$$Pm = 112.375\,.\,(P_L^{1.21} / P_H^{1.36}) \qquad \text{with } r = 0.91$$

which avoids the necessity for converting MAT to Pm via Lord's original regression formula (see section 4.2).

Of course, with any given series of cottons there is always a high probability of finding alternative regression coefficients which satisfy the experimental data for that series better than the basic calibration equation of the instrument. Furthermore, it has to be recognised that there have been criticisms [113, 145] of Pierce and Lord's original relationship between θ and M and of the practice of using measurements on swollen fibres to calibrate an instrument which actually operates on dry, unswollen ones [101, 108]. In addition, it is true that the basic equations for the FMT, as given at the beginning of this section have never been thoroughly and independently tested using an unequivocal calibration procedure and an adequate range of cottons.

However, it should also be noted that the original calibration equations for the FMT are based on multiple (at least four) measurements of Maturity Ratio and Gravimetric Fineness on a set of 100 cottons, representing all the major botanical species and having a range of:

Maturity Ratio, M,	from 0.63 to 1.095
Gravimetric Fineness, H,	from 99 to 395 mtex
Standard Fineness, Hs,	from 113 to 361 mtex

More importantly, the correlation between Maturity Ratio and Fineness in the calibration series was only r = 0.5 and that between Maturity Ratio and Standard Fineness was only r = 0.2.

The importance of an adequate range of Standard Fineness has been thoroughly discussed by Gutknecht [137] who made FMT measurements on a range of 204 cottons ranging in Maturity from about 57 to 90 in percent mature fibres and from about 160 to 300 in Standard Fineness. He has compared several of the alternative calibration equations which have been proposed for the FMT and concludes that Lord's original calibration is the best.

The International Committee on Cotton Testing Methods has carried out controlled inter-laboratory evaluations of the FMT and has recommended that this method should be adopted as the standard technique for rapid measurement of cotton maturity [144].

Finally, it may be noted that the FMT can also be used to obtain an estimate of the relative wall thickness, RWT, since Ramey has pointed out that, based on simple geometrical considerations, the following relationships apply [149]

$$D^2 = 4.FIN/\pi\rho\theta \qquad d^2 = (4.FIN/\pi\rho) \,.\, (1/\theta - 1) \qquad RWT = (D - d)/D$$

where:

D is the diameter of the equivalent circle

d is the diameter of the equivalent lumen

ρ is the density of the cell-wall material

θ is the degree of thickening = 0.577.MAT

Assuming a cell-wall density of 1.52, these expressions reduce to

$$D^2 = 1.452.\ FIN/MAT \qquad d^2 = 1.452.\ FIN/MAT - 0.838.FIN$$

9. LITERATURE (Sections 1 to 7)

1 S.T. Burley and E.S. Barness. *The Causticaire Method for Determining Cotton Fiber Maturity and Fineness*. Preliminary Report, USDA, Washington, 1952.

2 W.E. Chapman. Text. Res. J., 1961, **31**, 429.

3 W.E. Chapman and G. Staten. Text. Res. J., 1957, **27**, 991.

4 C.R.I.T.E.R. *Appreciation de la maturite du coton au moyen d'appareils Air-Flow*. Bull. I.T.F. 1961, **93**, 43.

5 D.De Meulemeister, A.D.J. Meeuse, G. Raes and Armand-M. Van den Abeele. Textielwezen, 1950, **6**, 21.

6 D.De Meulemeister, G. Raes and T. Fransen. Ann. Sci. Text. Belges., 1954, No.2, 49.

7 D.De Meulemeister, G. Raes and T. Fransen. Ann. Sci. Text. Belges, 1955, No.3, 260.

8 W.F. Du Bois and G.H.J. Ten Cate. Melliand Textilberichte International, 1970, **51**, 1118.

9 J.L. Fowler and K.L. Hertel. J. Applied Physics, 1940, **11**, 496.

10 C.F. Goldthwaite, H.O. Smith and M.P. Barnett. Textile World, 1947, **97**, No.7, 105 *et seq.*

11 Mary A. Grimes. Textile Research, 1942, **13**, 12.

12 Mary A. Grimes. Textile World, 1945, **95**, No.2, 196.

13 F. Hadwich, Textil -Praxis, 1960, March, 221.

14 K.L. Hertel and C.J. Craven. Text. Res. J., 1951, **21**, 765.

15 P.A. Koch and B. Wulfhorst. Textil-Industrie, 1971, **73**, No.4, 196.

16 E. Lord. J. Text. Inst., 1955, **46**, T 191.

17 E. Lord. J. Text. Inst., 1956, **47**, T 16.

18 E. Lord. J. Text. Inst., 1956, **47**, T 635.

19 E. Lord. J. Text. Inst., 1956, **47**, T 209.

20 W.E. Morton and S. Radhakrishnan. J. Text. Inst., 1954, **45**, T 774.

21 J. McD. Stewart, Amer. J. Bot. 1975, **62**, 723-730.

22 N.L. Pearson. Text. Res. J., 1955, **25**, 124.

23 F.T. Pierce and E. Lord. J. Text. Inst., 1939, **30**, T 173.

24 A.T.C. Robinson, Texture, 1955, 1.

25 J. Roch. Coton et Fibres Tropicales. 1967, **22**, 473.

26 O. Roehrich. Bull I.T.F. 1950, No.17, 9.

27 E.R. Schwarz and G.H. Hotte. Textile Research, 1935, **5**, 370.

28 E.R. Schwarz and G.H. Hotte. Textilberichte, 1936, **17**, 549.

29 E.R. Schwarze and L. Shapiro, Rayon Textil Monthly, 1938, **19**, 370 *et seq.*

30 Sheffield Corporation (U.S.A.) Textile World, 1948, No. 1., 150

31 E.L. Skau. Text. Res. J., 1951, **21**, 14.

32 W.S. Smith. Textile Industries, 1947, **111**, Nov., 86.

33 R.R. Sullivan. J. Applied Physics, 1941, **12**, 503.

34 R.R. Sullivan. J. Applied Physics, 1942, **13**, 725.

35 R.R. Sullivan and K.L. Hertel. Textile Research, 1940, **11**, 30.

36 V. Sundaram and R.L.M. Iyengar. Text. Res. J., 1958, **28**, 1045.

37 USDA. *Revised Micronaire Fiber-Fineness Scale for Use in Testing American Upland Cottons*. Washington, October, 1950.

38 USDA. *Micronaire Fiber-Fineness Scale for Use in Testing American-Egyptian Cotton*. Washington, 1952.

39 R.W. Webb and S.T. Burley, Market Research Report No.57, Washington, 1953.

40 H.H. Ramey Jr., *The Meaning and Assessment of Cotton Fibre Fineness*. 1982. The International Institute for Cotton.
41 L. Rebenfeld and Hong Wu; Textile Res. J., 1961, **31**, 886.
42 E. Schollmeyer and U. Denter; Textile Praxis Int. 1983, **38**, 1235.
43 ASTM method D1464

(Section 8)

101. S.G. Stephens. Textile Research J., 1976, **46**, 835.
102. K.L. Hertel and C.J. Craven, Textile Research J., 1951, **21**, 765.
103. S.G. Stephens, Textile Research J., 1977, **47**, 1.
104. S.G. Stephens, Textile Research J., 1977, **47**, 526.
105. W.F. Du Bois and G.H.J. Ten Cate. Melliand Textilberichte International, 1970, **51**, 1118.
106. G. Raes & L. Verschraege Textil Betrieb. 1980, **98**, No.6, 32.
107. L. Verschraege & T. Fransen, Coton et Fibres Tropicales, 1980, **35**, 335.
108. G. Raes & L. Verschraege, Journal of the Textile Institute, 1981, **72**, 191.
109. R.S. Chauhan, N.M. Shah, & N.E. Dweltz, Textile Research J., 1981, **51**, 399.
110. B.M. Petkar, D.V. Mhadgut, P.G. Oka, Textile Research J., 1986 **56**, 642.
111. M-C. Peeters, J. Wijsmans, I. De Langhe, E. De Langhe. Textile Research Journal, 1986, **56**, 529.
112. Idem Ibid, 1986, **56**, 621.
113. P. Neelakantan, Journal of the Textile Institute, 1975, **66**, 332.
114. J.J. Herbert, E.K. Boylston, J.I. Wadsworth, Textile Research J., 1979, **49**, 540.
115. S. Worley Jr., E.O. White, M. Preysch, J.L. Cormany, Melliand Textilberichte, 1988 **69**, 785.
116. K. Gilhaus & J. Liinenschloss, International Textile Bulletin (Spinning), 1980, 117.
117. J.D. Berlin, S. Worley Jr., H.H. Ramey Jr., S.S. Linkous, Textile Research J., **51**, 109.
118. R.L. Barker, D.W. Lyons, J. Engineering Industries, 1979, **101**, 59.
119. D.P. Thibodeaux & J.P. Evans, Textile Research J., 1986, **56**, 130.
120. F.T. Pierce & E. Lord, J. Text. Inst., 1939, **30**, T173.
121. I.L. Gridi-Papp & N.B. Sabino, Revista brasilieira de tecnologia, 1972, **3**, 99.
122. R.S. Krowicki & K.E. Duckett; Journal of the Textile Institute, 1972, **63**, 650.
123. J. Gutknecht, Coton et Fibres Tropicales, 1976, **31**, 267.
124. Spinlab Information Bulletin, Nos 107, 108 & 108A, 1976/77.
125. ASTM method D3817-1979.
126. K.E. Duckett & R.S. Krowicki, Journal of the Textile Institute, 1976, **67**, 334.
127. J. Gutknecht & J. Fournier, Coton et Fibres Tropicales, 1982, **37**, p. 249.
128. R.S. Krowicki & K.E. Duckett, Journal of the Textile Institute, 1979, **70**, 78.
129. Idem. Textile Research J., 1980, **50**, 354.
130. S.G. Nayar, V.G. Munshi, V. Sundaram, Textile Research J., 1979, **49**, 513.
131. H.H. Ramey Jr., Textile Research J., 1982, **52**, 20.
132. Weyer, Applied Spectroscopy Reviews, 1985, **21**, 1 (Marcel Dekker Inc).
133. S. Ghosh, Textile World, 1985, **135**. 145.
134. S. Ghosh, Textil Praxis International, 1986
135. J.B. Price & H.R. Smith, *Annual Progress Report*, Textile Research Center, Texas Tech University, 1985/86, 317.

136. J.G. Montalvo, D.P. Thibodeaux, S. Faught, S.M. Buco, Proceedings Beltwide Cotton Production Conferences, 1987, (National Cotton Council), 155.

136a. J.G. Montalvo, S.E. Faught, S.M. Buco, A.M. Saxton, Applied Spectroscopy, 1987, **41**, 645.

137. J. Gutknecht, Textil Praxis International, 1982, **37**, 1032.

138. F. Hadwich, Melliand Textilberichte, 1975, **56**, 862.

139. De V. Aldrich, SAWTRI Technical Report No. 274, 1975.

140. S, Smuts & L. Hunter, SAWTRI Bulletin, 1980, **14**, 22.

141. J. Liinenschloss, K. Gilhaus, K. Hofmann, Melliand Textilberichte, 1980, **61**, 5.

142. R. Lawson & H.H. Ramey Jr., Journal Testing & Evaluation, 1978, **6**, 248.

143. L. Verschraege & T. Fransen, De Tex-Textilis 1978, No.11, 9.

144. S.A. Heap, Proceedings International Committee on Cotton Testing Methods, 1986, I.T.M.F., Zurich.

145. J.T. Mitchell, Melliand Textilberichte, 1977, **59**, 955.

146. I.K.P. Iyer, V.G. Munshi, Journal Textile Association (India), 1985, 4.

147. P. Neelakantan & N.M. Shah, Textile Research Journal, 1977, **47**, 407.

148. G.S. Rajaraman, Textile Research Journal, 1980, **50**, 747.

149. H.H. Ramey, Jr., Private Communication, May 1987.