Introduction and the role of testing

Introduction

The pleasure derived from imparting colour to clothing has existed since the time of the earliest civilisations; a world of fashion without colour is impossible to imagine. Coloration processes produce the most visible results of all the finishing operations carried out during the preparation of textile goods. As such they reveal both the care taken with the coloration and the degree of control exercised during other stages of the manufacturing process.

Dye uptake is dependent on many factors. The result of unplanned variations in the conditions of operation during any step of the manufacturing chain may not become apparent until the fabric is dyed. Variations in temperature during the texturising of synthetic fibres, or irregular changes of tension during weaving, can lead to the development of either darker or lighter stripes after dyeing, wherever the affected yarns are incorporated into the fabric. Freedom from manufacturing faults and adequate resistance to the various treatments likely to be received by the goods during use are important to a consumer. Intensive efforts are therefore made to ensure that the coloration of fabric and yarn conforms to well-defined specifications. Commercial success in the modern coloration industry depends upon technical efficiency at all levels of activity, and this requires an appreciation of the properties of dyes and fibres, the way in which both behave during use and the objective manner in which the results of coloration processes are represented.

Origins of textile dyeing



The origins of dyeing are uncertain, but it is believed that coloured fabrics found in the ancient tombs of Egypt were in existence before 2500 BC. It is likely that the ancient art of dyeing originally spread westwards from India, and it may well have been accidental staining from berries and fruit juices that initially stimulated its development.



The use of colouring materials from plants (using roots, stems, leaves, flowers, fruit, seeds and lichens) and from the animal kingdom (using insects and shellfish) continued until the latter

part of the nineteenth century. During the Middle Ages relatively few dyes were available, and European dyers imported their dyes from the Middle and Far East. During the fifteenth and sixteenth centuries their sources extended to the Americas with the discovery there of previously unknown natural dyes. It was during this period, when Spain and Portugal became prominent in the art of dyeing, that the Portuguese discovered a tree in South America from which a very desirable red dye could be extracted. In fact the tree was also known to the inhabitants of the Anderman Islands in the thirteenth century as 'sappan', but in its South American location it was called 'brazil'. For this reason the Portuguese called the area in which it was found Terra de Brazil, and thus the name of the country was derived from the dye rather than the other way around.

The colour range of the natural dyes could be expanded to a limited extent by treating the fabric or yarn with metal salts (mordanting). Some of the results obtained on wool are illustrated in Plate 1 [1]. Even so the only blue dye of the time was indigo, extracted either from the indigo plant (*Indigofera* sp.) or from woad (a different species of plant which gave a colour of less satisfactory performance), and greatly valued because of its excellent fastness to light.

Some form of quality appraisal existed even in ancient times. For example, the use of the dye Tyrian purple, with its high resistance to daylight, was restricted to the garments of those in authority. Tyrian purple was extracted from shellfish in the eastern Mediterranean, and was the most highly prized and expensive dye of classical times, being used for the robes of the kings of Medea and the royal houses of Persia, Babylon and Syria, as well as for the togas of Roman emperors. It ranked alongside indigo as one of the few natural dyes with resistance to fading. The high cost of this dye in ancient times is indicated by the quantity of sea snails required to provide sufficient extract for the purposes of dyeing. The efforts of Friedlander may be quoted as an example. During his elucidation of the chemical structure of the colour, he needed 12 000 of the creatures to obtain just 1.4 g of the dye!

Nowadays, when care of the environment is a major issue, it is tempting to assume that the use of natural colours is an environmentally friendly alternative to present-day practice. Unfortunately such assumptions change to doubts after the imagination has been stimulated by the details in Figure 1.1, which shows a description of the application of the once widely used Turkey red dye, obtained from the roots of the madder plant.

Indigo was the only natural dye to yield blue shades; its fastness to light was outstanding when compared with other natural dyes. As a result it achieved particular importance. Even so, the



bacterial fermentation process used for its extraction from either *Indigofera* or woad plants was highly unpleasant and prompted Queen Elizabeth I to order the curtailment of the production of woad.

Compared with the extensive lists of dyes in today's *Colour Index*, the colorants in use up to the nineteenth century were few in number. The more important ones are listed in Table 1.1. The last natural dye to be used commercially was logwood, which remained in use until the 1940s.

Mordanting

Treat the cloth with sour olive oil, pearl ash, sheep's dung and water. Dry in warm air. Repeat seven or eight times.

Tanning

Treat in a decoction of nut-galls. Dry. Treat with neutralised alum. Age for three to four days. Treat with warm water containing ground chalk.

D**yeing** 7reat with extract of madder root.

Cleaning Treat two or three times with salt solution.

Figure 1.1 Recipe for the application of Turkey red

Synthetic dyes

The most significant event in the recent history of coloration was the discovery by W H Perkin in 1856 of the first dye to be obtained by synthesis rather than by tedious extraction from natural products, which he named mauveine. Thousands of dyes have since been made by synthesis, and dye manufacture has become a significant part of the chemical industry. Natural dyes have now been superseded, along with the need for extensive pretreatments with mordants. Colorants with chemical and physical properties better suited to contemporary demands have been synthesised, sometimes aiding the development and extension of the use of particular products. For example, the development of synthetic fibres such as polyester and cellulose triacetate would have been severely hindered without the design and synthesis of dyes with







appropriate properties. These fibres do not absorb water to any marked extent and therefore the water-soluble dyes available at the time of their discovery could not be used. Dyes and pigments can now be synthesised for almost any purpose, and consumers can be assured of satisfaction with the

properties of coloured fabric during use. The initial sign of confidence came from Sir James Morton, the first member of the textile trade to offer a replacement of the fabric if its resistance to fading in light and ordinary washing was not sustained during the product's lifetime.

The present-day consumer can now assume that purchased goods will be fit for their purpose because the industry is able to provide assurances of product quality by working to voluntarily agreed standards. It has taken time for the concept of providing consumer reassurance to develop. The mechanisms now adopted started to evolve during the industrial revolution through cooperation between producers of basic products and the manufacturing industry. Although it was some time later that they were adopted and rigorously applied in the textile industry, the efforts made at that time were the forerunners of current quality assurance schemes.

Development of standards

The advantages of working to standards first became established during the development of the railways. Easier transportation of goods from one place to another removed the need to obtain components locally, but small variations in the dimensions and quality of goods made in different locations caused difficulties in that the buyer could not know exactly what to expect. This was an inconvenient source of the mismatch of components in construction work. For instance, variations in the cross-sectional shape of steel girders and in the screw threads of nuts and bolts highlighted the advantages of standardisation – the reason why the familiar Whitworth screw thread was adopted as a standard during this period.

The need to manufacture goods in different parts of the country during the two world wars intensified efforts to bring uniformity in production through the use of voluntary standards and specifications. The British Standards Institution (BSI) took up this responsibility and an illuminating account of the origin, development and introduction of standards has been published [2].

During World War 2 various standard specifications were produced for textile goods, in particular for blackout material. It was at this time that the BSI began to formulate standards relevant to consumer goods. The best use had to be made of very scarce resources, and by working to specified standards manufacturers supported the 'Utility' scheme operated at the time by providing clothing and household textiles of predictable quality. The domestic consumer derived further help



from the specifications for the sizes of a wide range of children's clothing; nowadays, however, articles of clothing are no longer labelled with garment measurements but according to a system based on body size.

After the war the Utility scheme was eventually abandoned in favour of informative labelling. This was due in part to a survey carried out by the Consumer Advisory Council, which revealed a strong desire among the public to receive more safety information about textile goods. Children's clothing, blankets, furnishing fabrics and carpets featured high on their list of priorities. Some time later various tragic accidents involving burning textiles directed attention to the development of standards for flame-resistant fabrics and the fillings of mattresses and pillows.

At the time of World War 2 the natural fibres cotton, linen, silk and wool and the synthetic fibres viscose, secondary acetate and cuprammonium rayon were the only ones available. Subsequently, with the intensive development of synthetic fibres and production methods, modern fabrics have become so multifarious that neither the sales person nor the purchaser can be expected to predict the performance of textile products using only personal experience. The mixing of natural with synthetic fibres to give blends with some of the characteristics of each component, and the development of finishes that impart to the synthetics characteristics previously associated with natural fibres, have contributed to the complexity of the situation. Consequently the use of informative labels has become a most appropriate way of providing a measure of quality assurance. The use of hallmarks on gold and silver jewellery by craftsmen to assure their customers of the quality of their goods can be traced back to the Middle Ages, but the origin of labelling schemes for textile goods is a concept of modern times.

There was considerable consumer dissatisfaction with synthetic dyes produced during the first 50 years of their existence [3]. Their convenience of application was counteracted by poor resistance to both daylight and washing. This stimulated the continuing search for dyes with improved resistance to a variety of destructive agencies, and as progress was made manufacturers began to approve various labels with colour fastness implications. The Sundour label of Sir James Morton was probably the first example; many other manufacturers' brand-name labels, however, did not indicate the quality of the dyes used. Some dye manufacturers attempted to help the consumer by approving labels for fabric dyed with particular dyes selected for their high standard of fastness. Similarly fibre manufacturers imposed the requirement for the fibre content of the fabric to appear on the label, whilst other labels have been promoted by dyers and printers, manufacturers of





detergents or appliances, retail organisations and consumer-oriented magazines. At the present time an overview of the International Care Labelling Scheme, discussed in Chapter 2, is maintained by the Home Laundering Consultative Committee.

The familiar Kitemark now attached to a wide range of goods to indicate quality assurance was first introduced in the 1950s. Its presence on an article is a visible means of assurance, denoting conformance to relevant standards developed through the BSI.

The initial standards for textiles were concerned mainly with constructional details. But the continuing development of domestic appliances and of new materials has led to the provision of labels containing more detailed information relevant to the performance of manufactured textile goods during use. For the consumer, quality assurance is now indicated through labels that show both fibre content and information on aftercare. In addition the manufacturers benefit from the efforts of their suppliers in ensuring that the raw materials and the products of intermediate stages of manufacture will also match agreed standards.

The fastness of dyes to various agencies is of particular relevance to the subject matter of this book, and voluntary coordinated activity in this sphere began in 1927 with the Fastness Tests Committee of the Society of Dyers and Colourists. Since that time the committee has been responsible for the development and continuous review of a wide range of appropriate fastness tests for dyed materials, of which full accounts are to be found in other publications [4,5].

The International Organisation for Standardisation (ISO) was formed in 1947 and a technical committee, the Colour Fastness Subcommittee, became responsible for bringing uniformity to the various systems adopted in different parts of the world. The internationally agreed standard test procedures recommended by that committee are now automatically adopted as British Standards.

The concept of quality assurance is now well documented and embraces all facets of production, services and management, with British Standards having been developed for every aspect of related activity, most of which are covered in appropriate publications [6].

Requirements for the adoption of standard quality procedures appropriate to both large and small organisations are detailed in a far-reaching British Standard, BS 5750. It covers the organisation, management and documentation of quality control procedures from the initial purchase of raw materials to the appraisal and delivery of the goods or service, including matters relevant to process design and staff training. Like many other industrialists, textile manufacturers are increasingly following the principles laid down in BS 5750.



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Significance of quality in relation to coloured goods

Quality as defined by the BSI is 'the totality of features or characteristics of a product or service that bear on its ability to satisfy a given need' [6]. From the point of view of the retailer this also involves the *value* of a product as represented by the relationship: value = quality/price [7].

The implication of this relationship to a company concerned with providing quality and value is that price and quality are balanced. Clearly there is no point in providing perfection of quality if the added cost is so high that the value of the goods falls to a level too low for the purpose which the customer has in mind.

Perception of quality varies from one person to another, depending upon his or her immediate needs. Thus to a commercial buyer price, delivery date, colour and style may be more important than technical quality. To an interior decorator the fastness to light of curtains for the stage of a cinema or theatre is less important than their aesthetic properties. On the other hand, the same person would regard fastness to light as of prime importance if asked to provide curtains for a south-facing window of a seaside hotel. Similarly a wedding dress does not require the same fastness specifications as a child's anorak. Many highly fashionable and expensive goods bearing brand names have a low quality in objective terms, and the simple relationship cannot always be valid when choice is made entirely on subjective grounds. The final judgement of the consumer with regard to quality and value is influenced by his or her perception of both subjective and objective factors. This book, however, deals with only the objective factors involved with coloration. Accordingly the first consideration will be the principles of some of the underlying testing procedures used by manufacturers in establishing consistency and quality during the preparation and use of their products.

Standards related to coloration



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The development of standards has been of great value to the colourist in both the provision of objective assessments and the transmission of relevant information between dye manufacturer and dye users. The general groupings of British Standards cover a wide range, including the following:

(a) glossaries, which provide agreed definitions of terminology in specialist fields of activity

- (b) dimensional standards, which provide for interchangeability of manufactured components from different sources
- (c) performance standards for the specification of expected performance

- (d) standard methods of test with precise and detailed specification of operation that are of particular use in setting up quality assurance schemes
- (e) codes of practice for the design, installation, maintenance and servicing of equipment or services.

These are not the only source of standard definitions, however, and readers may also find other references to be of equal value when becoming acquainted with terms used in the technology of the coloration of textiles [8,9]. The subject matter of this area of study falls into the fourth category of British Standards, i.e. standard methods of test.

Results from the standard methods of test are widely used for the objective appraisal of the behaviour of dyes under an extensive range of circumstances [10]. Once the commercial decisions about an appropriate level of fastness have been taken, a performance specification may be formulated and dyes selected for the purpose in hand.

Principles of colour fastness testing

Appraisal of the performance of a dye begins at the time of its synthesis and ends with tests designed to indicate the level of performance during its use. Obviously, consumer goods must have satisfactory resistance to domestic cleaning treatments and a reasonable resistance to fading under the action of daylight, but many other factors also need to be considered if the requirements of the textile finisher and dyer are to be met. In some cases a very high level of fastness is provided for the consumer because the dyes used are expected to withstand processing conditions that are far more severe than any likely to be encountered during normal use. In other cases special efforts are made to find new dyes that will withstand particularly intensive conditions associated with the use of a new product.

The wide-ranging end uses associated with coloured textiles are accommodated by the development of realistic test methods for the fastness to wet treatments. These often involve the formulation of appropriate variations in the severity of the testing conditions. The objective assessment of the effects obtained are usually made on the basis of visual comparison of the intensity of any change in the appearance of the sample with calibrated standard *grey scales*. The fastness of the coloured textile is then rated numerically according to the contrast step in the scale that matches the intensity of the observed change. The test is twofold: changes in both the depth of the



dyeing and the staining of previously undyed fabric in the presence of the test pattern are examined, although different scales are used for the assessment. These are shown in Figure 1.2.

After the dyed material has been subjected to the test conditions, the extent of staining is assessed by placing a sample of the unstained material alongside the stained material. A judgement of the degree of contrast between the two is then made by comparison with the relevant steps in the grey scale under the recommended conditions of illumination. The scale used for staining (BS 1006:A03:1978) covers five full steps arranged in geometrical progression.

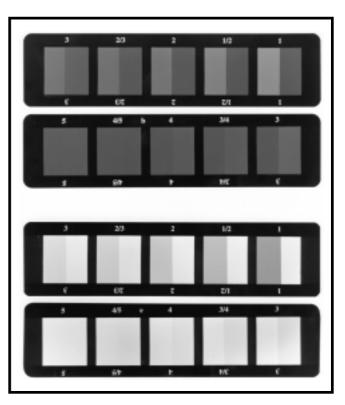


Figure 1.2 Grey scales: (top) the scale used for the assessment of colour change in the sample, (bottom) the scale used to assess staining

When no staining occurs a rating of 5 is appropriate but the numerical rating decreases as the staining worsens. Half-steps in the grey scale are provided to increase the precision of the assessment.

The scale for assessing the change in colour is used in a similar manner to compare the treated and the untreated dyed fabrics, but in this case the rating may be supplemented by letters to indicate an accompanying change of hue or brightness [11]. For example, the change may be indicated by a number alone to indicate a loss in depth only, but the number followed by a letter indicates other changes; for example 3 W, Bl, D signifies that a loss in depth (weaker) corresponding to grade 3 of the grey scale is accompanied by a change in hue towards blue and that the pattern has become duller. According to convention the qualifiers are always placed in order of magnitude. The terms used for this purpose are shown in Table 1.2 overleaf, and are discussed further in Chapter 7 (page 140).



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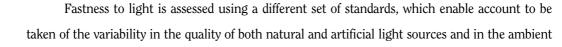


Table 1.2 Terms used for the qualitative description	n
of colour changes	

Redder (R) Yellower (Y) Bluer (Bl) Greener (G)	Weaker (W) Stronger (Str)	Duller (D) Brighter (Br)
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conditions of weathering. The standards recommended by ISO for this purpose consist of eight blue dyeings on wool, of which No. 1 provides the lowest rating and No. 8 the highest. Each successive standard requires twice the exposure time of the one below in the series to cause the same

degree of fading (Plate 2). As with other standard tests, the conditions of exposure are rigorously specified and include exposure to standardised sources of artificial light as well as to daylight.

Standard depths of shade

In addition to the inherent resistance of a particular dye–fibre combination to the agency in question, the assessment is also dependent upon the depth of the colour. Obviously a deeper dyeing of poor fastness to wet treatments will release more dye into the water than will a paler dyeing, and thus both the degree of staining and the visual effect on the coloured pattern itself will be affected. The results from individual fastness tests are therefore characterised by reference to one of a series of standard depths of shades. The ISO has recommended the use of 20 reference colours, for which a series of six standard depths are displayed. These are known as 2/1, 1/1, 1/3, 1/6, 1/12 and 1/25 standard depths respectively [12]. They are produced on a matt wool gabardine cloth and on a lustrous bright viscose fabric; whenever tests are carried out the standard depth of the shade closest to the pattern under test is quoted.

Resistance of coloured fabric to harmful agencies

The visual effects observed as a result of fastness testing may be caused either by a chemical breakdown of the dye or by removal of dye from the fabric, or by both. Whether or not the adjacent uncoloured material will be stained by dye removed from the pattern will depend on the attraction of the textile for that particular dye under the testing conditions. The dye may also decompose for any of a variety of reasons (such as chemical instability to the action of light or to the oxidising reagents contained in detergent powders, or even the presence of foreign substances in the textile), and the colour of the products of degradation of the dye may be different from that of the dye itself. Dyebath additives necessary for the dyeing of polyester fibres may influence the fastness to light of dyes on





the fibre if residues are left after the washing process. Crease-resist finishes or dye-fixing agents applied to some cotton dyeings also adversely affect the fastness to light of some dyes.

In the production of man-made fibres, titanium dioxide pigment is often incorporated into the polymer mass to render the resulting fibres opaque (dull fibres) and this can reduce their fastness to light as compared with the corresponding unpigmented material (bright fibres). The significant influence of moisture content on the fastness to light is also well recognised.

Fastness assessments are also affected by the fineness of the fibres, simply because a given amount of dye on a fine fibre is spread over a larger surface area than is the same amount on the same mass of a coarser fibre. The compactness of the fibre structure can also have a bearing on how easily the dye fades or can be removed during washing treatments. The nature of the fibre itself can also markedly affect fastness to light: the fastness to light of basic dyes, for example, is very poor on cotton and wool, but high on acrylic fibres.

Further influences originate from the state of the dye in the fibre. Insoluble pigments trapped mechanically inside the fibre, or dyes that have formed a strong chemical linkage with the fibre, will obviously be more resistant to removal by wet treatments than dyes that are more loosely attached. Yet another factor is the yellowing of fibres with age, which is impossible to prevent. Also fluorescent brightening agents in household detergents may be left on the fibre after washing, and these superimpose an additional effect on the change in the appearance of the colour.

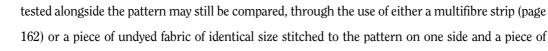
Washing fastness tests (ISO C01-C06)

Domestic and commercial washing conditions are covered by six ISO wash tests, which are directed towards simulation of the conditions likely to be encountered in normal use (Table 1.3). If possible, colour changes should be assessed using the grey scales. If these are not available the staining of different white fabrics

ISO	Composition	Temperature	Duration
test	of wash liquor	(°C)	(hours)
1	Soap 5 g/l	40	0.5
2	Soap 5 g/l	50	0.5
2 3	Soap 5 g/l + soda 2 g/l	60	0.5
4	Soap 5 g/l + soda 2 g/l	95	0.5
5	Soap 5 g/l + soda 2 g/l	95	4.0



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Specimen	First adjacent	Second adjacent
fabric	fabric	fabric
Cotton	Cotton	Wool
Wool	Wool	Cotton
Silk	Silk	Cotton
Linen	Linen	Wool
Viscose	Viscose	Wool
Acetate/triacetate	Acetate/triacetate	Viscose
Polyamide (nylon)	Polyamide (nylon)	Wool or cotton
Polyester	Polyester	Wool or cotton
Acrylic	Acrylic	Wool or cotton

specified but different undyed fabric on the other. The standard 'adjacent fabrics' are listed in Table 1.4.

The composite specimen is agitated in a solution with a defined concentration of soap and, where appropriate, other prescribed additives. Controlled agitation is maintained at the temperature required by the test conditions and the ratio of liquid volume

to mass of fabric is also defined. At the end of the testing the sample is removed and rinsed, and the components separated and allowed to dry. A visual assessment is then made.

The nature of the detergent to be used is stipulated in the standard, together with the concentration of sodium carbonate (soda ash) needed to make the solution alkaline in reaction. Tests 4 and 5 are of greater severity than the others. The mechanical action is intensified by the inclusion of ten non-corrodable steel balls together with the composite sample in the container, while the detergent has been modified so as to allow for the effects of the various components of the different brands of washing powder used in commercial and domestic laundering. The detergent composition is based on a mixture of synthetic detergents and natural soap, a phosphate, a silicate, an inorganic salt (Glauber's salt) and a compound that inhibits soil redeposition when used for cleaning off particulate matter. The specially formulated detergent is used in the additional test proposed for colour fastness to domestic and commercial laundering, which provides for a greater rubbing action through the use of up to 100 steel balls.

Determination of light fastness

The influence of light on the fading of dyes is a complex phenomenon influenced by many variables, and accordingly predictive tests for fastness to light are amongst the most difficult to establish. The depth of colour, the presence of unwanted additives, humidity, air temperature, the surface temperature of the sample, the presence of atmospheric impurities and the spectral quality and intensity of the light source all have a bearing on the end result.



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When using daylight the samples are exposed behind glass alongside the blue wool standards,

taking care to place the samples at an angle equal to the latitude of the location of the testing station, facing due south in the northern hemisphere and due north in the southern hemisphere. Adequate ventilation is essential. The rate at which the samples fade is determined by partially covering both standard and pattern with an opaque card and inspecting both fading standards and sample periodically. The specimen is faded to the equivalent effect on the pattern of a grade 3 contrast on the grey scales. At this stage the degree of contrast between the exposed and unexposed pattern is compared with that for the standards, and the rating given is the number of the standard that exhibits the same degree of contrast.

The time taken for daylight testing can be inconveniently long, however; furthermore, the conditions are variable from one geographical location to another. Consequently it would be useful to be able to carry out the tests using artificial light sources. For many years various light sources were used to imitate the spectral distribution of daylight, all of which have been subjected to close scrutiny. Although the light of a xenon arc is currently favoured as the best substitute for daylight, the matter is constantly under review.

There are other factors to be taken into account when designing the equipment for carrying out fading tests. A means has to be found for maintaining the humidity at the surface of the patterns to prevent them drying out. The variations that occur between the results from different light sources and from operating the same light source under different conditions are mainly due to differences between the spectral composition of the lights, their intensity and the effective humidity at the surface of the pattern. The effective humidity represents a combination of air temperature, the temperature of the surface of the pattern and relative humidity, which governs the moisture content of the fabric. The conditions of operation are therefore specified very carefully in the standard tests, and control of the effective humidity is recommended through the use of a humidity test control in the form of a fabric coloured with a particular red pigment. The relationship between the fastness to light of this fabric and the operating humidity is known with reasonable precision. Preliminary tests are then carried out to check if the humidity test control fades to the correct blue standard, and adjustments in the operating conditions made if required. Once the correct conditions have been established the testing is carried out in the normal manner.





Photochromism

Sometimes the colour of a dyeing changes on exposure to light, but reverts to its original state after

the sample is kept in the dark. In such a case an additional test is carried out to indicate the extent of the change (as well as the fastness rating test). The extent of this *photochromism* is expressed as a grey scale assessment given alongside the rating for fastness to light.

Other fastness properties

Tests have been designed to establish the resistance to a wide range of aqueous agencies, including distilled water, sea-water, chlorinated water and perspiration, and to spotting in cold and hot water, acid and alkaline solutions, and various other agencies relevant to different end uses of the goods.

The resistance to bleaches of different types, atmospheric contaminants and a variety of other agencies are all covered by tests described with full details in the appropriate manual [10]. The situation with regard to the development of tests is never static; they are perpetually under review as conditions and products change. Very often intensive effort is required to ensure reproducible results by establishing conditions that simulate those encountered by the material. There is also a dominant need for speed with accuracy in executing the tests because they may form part of an independent certification scheme that must be completed before further steps in manufacture, marketing or purchasing can proceed. A comprehensive bibliography concerning the development of the various testing methods has been compiled [4], and their application to coloration practice is described in various sections of this book.

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