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Fiber and yarn identification

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1.1 Introduction

The chemical testing of textile fibers has continued to receive special attention from producers, manufacturers, governmental agencies, domestic and industrial consumers. Of particular interest is the number of publications on recent methods of testing and analysis of textiles in general but with specific focus on physical testing, analysis and quality control. This chapter will present selective discussions of the chemical characteristics of major fiber types since an understanding of the fiber chemistry and morphology will aid the chemical analysis of these fibers/yarns. The chapter will also introduce to the reader a selection of chemical tests that are useful in a textile laboratory and document some of the more common chemical methods of analyzing single textile fibers and yarns. Summaries of chemical testing using modern instrumental/analytical tools such as scanning electron microscopy, SEM, transmission electron microscopy, TEM and Fourier transform infrared spectroscopy, FTIR, will be presented.

In practice, all chemical testing must be performed in accordance with specific standards, preferably internationally approved standards. Sometimes, the actual standard may be a mutually agreed and nationally accepted standard method of testing, or one that is based on the end-use of the product and other customer preferences.

1.2 Natural fibers

1.2.1 Chemical composition of cotton

Cotton is the purest form of natural cellulose. Like all the vegetable tissues, it contains a small amount of mineral matter that is left as an ash after cotton is burned. The amount of ash is about 1–1.5%. The mineral matter in cotton consists of chlorides, carbonates and phosphates of potassium, calcium and magnesium. A large variation is observed in the amount of coloring matter found in cotton. The small amount of vegetable protein found in cotton is a little over 1%. The impurity

Table 1.1 Chemical analysis of cotton fiber by McCall and Jurgens, 1951[4]

	Mature cotton (%)	Immature cotton (%)
Cellulose	96.41	92.44
Protein	1.00	2.00
Wax	0.45	1.14
Ash	0.79	1.32
Undetermined	1.35	3.10

found in the largest amount consists of pectinous materials. Also, a small amount of fatty material, which is mostly cottonseed oil, is found in raw cotton. This probably comes from cotton seeds that are slightly damaged during ginning.

Raw cotton contains about 0.5% of a waxy substance which serves as a protective coating on the surface of the fiber. Cotton wax is insoluble in water and because of this, raw cotton is very hard to wet. It is well known that unbleached cotton will not soak up water as easily as bleached cotton. After cotton is purified, all of these impurities are reduced to a total of about 1%.[1,2]

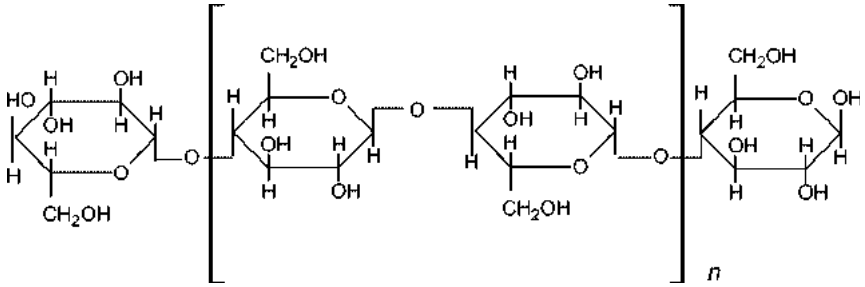
There are several factors that influence the chemical analysis of raw cotton, but we can obtain a general idea from the following figures. If the impurities are calculated on an oven-dry basis, the percentage of fiber should be around 90%. It would be close to 99% after purification.[2,3] Chemical analysis of the cotton fiber has shown the probable composition given in Table 1.1.[4]

The waxes are important in the spinning process but a hindrance to the dyeing operation. Owing to their water-repellent nature, waxes prevent the proper absorption of the dyestuff. The natural color of cotton and the other impurities also interfere, but to a lesser degree, with the ordinary processes of dyeing, printing and finishing.[5]

When all the impurities have been removed from the natural cotton fiber, cellulose remains. Cellulose is a long-chain polymer produced by linking together a large number of glucose units (see Fig. 1.1). Its empirical formula is $(C_6H_{10}O_5)_n$.[3]

Cellulose fiber contains both crystalline and amorphous regions. X-ray diffraction (XRD) diagrams give discernable patterns that indicate the existence of a crystalline arrangement of the molecules in the cellulose fiber. On the other hand, the spots on the XRD diagrams are somewhat blurred and not as clearly defined as those from conventional crystalline substrates.

The main objective in bleaching cotton is the removal of natural and adventitious colorants to produce pure white material. The coloring matters of cotton are associated partly with waxes, proteins and pectins. Some of these coloring matters are partly removed by scouring. However, there is residual natural coloring matter that can only be removed by bleaching. Little is known about these substances, but they appear to contain conjugated carbon double bonds and nitrogen. This coloring matter is capable of being changed into colorless compounds by means of nascent hydrogen, or oxidized into simpler soluble colorless compounds by nascent



1.1 Cellulose structure.

oxygen. The chemicals used for these purposes are termed bleaching agents. The whiteness produced by reducing bleaching agents is not permanent, turning yellow or brown upon exposure to air. The whiteness given by oxidizing bleaching agents does not, as a rule, become yellow. The choice of a bleaching agent is governed by the nature of the fiber, the degree of bleaching required and the cost of the process.[6]

One major treatment that the cotton fibers undergo during processing is mercerization which consists in impregnating the material with a concentrated solution of cold 20–25% sodium hydroxide, keeping the material in contact with this cold solution for a given time with tension and subsequently rinsing it. The structural changes that occur during mercerization induce shrinkage in width and length; this shrinkage has been shown to be caused by swelling which leads to changes in the cross-section of the fibers and improved luster. Ugbolue[7] has summarized the effect of mercerization and other chemical treatments on cotton. It is agreed that mercerized cotton fibers have greater moisture absorption capacity, are more receptive to aqueous chemical agents and are more accessible to dye molecules than unmercerized cottons. The structure of cotton grafted by radiation-induced polymerization with butyl methacrylate and acrylonitrile has been investigated by light and electron microscopy.

The internal structure of the fibers is studied using the transmission electron microscope and changes in fiber surface and type of damage caused by abrasion are evaluated using the scanning electron microscope. The results indicate that the grafting procedure changes the surface as well as the internal fibrillar character of the fibers; the extent of such changes increased with increasing graft polymer content. In a recent study a significant amount of grafting of the polymer on the cotton fabrics was observed and the samples were characterized using SEM and FTIR.[8] Thus, information about textile wet processes helps provide additional resources in our understanding of the chemical testing of these fibers and yarns.

1.2.2 Other vegetable fibers

Some of the other important vegetable fibers include flax, jute, kenaf, hemp, sisal, coir, banana and pineapple. Generally, in vegetable fibers such as cotton and flax,

cellulose is the material that provides the thread-like molecule and fibrillar structure. Differences in the properties of natural fibers of similar chemical constitution can be explained in part by variations in the state of alignment of the molecules. Flax and cotton are chemically almost identical and are both cellulose fibers. But flax has tensile properties quite different from those of cotton; flax has a tenacity of up to 6.3 grams per denier (gpd) compared with 3–5 gpd for cotton. Flax and ramie have highly oriented molecules along the fiber axis and consequently, high tensile strengths. Cotton, with an angle of spirality of about 31 ° has a much greater elongation at break than flax, with its spiral angle of 5 °.

Stout[9] has written a detailed review on jute and kenaf. X-ray diffraction patterns show the basic cellulose crystal structure, although in jute and kenaf the crystalline orientation is high and the degree of lateral order is lower than in flax.[9] Batra[10] in a comprehensive review has highlighted the morphological structures and physical, mechanical and chemical properties of other long vegetable fibers.

Gel-permeation chromatography[11] is used to compare the pore structure of jute, scoured jute and purified cotton cellulose. Both native and scoured jute have shown greater pore volumes than cotton. The effects of alkali and acid treatment on the mechanical properties of coir fibers are reported.[12] Scanning electron micrographs of the fractured surfaces of the fibers have revealed extensive fibrillation. Tenacity and extension-at-break decrease with chemical treatment and ultraviolet radiation, whereas an increase in initial modulus and crystallinity is observed with alkali treatment. FTIR spectroscopy shows that the major structural changes that occur when coir fibers are heated isothermally in an air oven (at 100, 150 and 200 ºC for 1 h) are attributable to oxidation, dehydration and depolymerization of the cellulose component.

1.2.3 Protein fibers

Animal fibers are made from proteins and the long molecules are built from some 20 or so different types of amino acid molecule. The proportion and arrangement of these different units determine the structure of the protein molecule and the nature of the protein itself. Wool cells come in two different types: the para cortex and the ortho cortex, which lie on opposite sides of the fiber and grow at slightly different rates. This causes a three-dimensional corkscrew pattern of coiled springs, giving wool high elasticity and a ‘memory’ that allows the fibers to recover and resume normal dimensions.

Wool fibers can be stretched up to 30% without rupturing and still bounce back. The closed-packed wool molecules are joined together by chemical links. These cross-links ensure that when the molecules are stretched out of their normal folded shape, they return to that shape when the stretching force is removed. Also, under suitable conditions wool can absorb half its own weight of water. Cook[13] has suggested that hot water or steam can destroy the cross-links so that the molecules are free to stay in the new positions that they reach when the fibers are stretched.

Moreover, prolonged heating will actually cause new links to form which anchor the molecules firmly in their new position, a transition from α-keratin to β-keratin. Electron microscopy, X-ray diffraction and other forms of evidence indicate that about one-third of the total length of the protein chains in wool is in the coiled-coil, α-helix conformation.

Silk, like wool, is an animal fiber and unlike wool, its molecules are in extended form. The protein molecules in silk are highly oriented in the direction of the fiber and can pack tightly together. The forces of attraction between the molecules interact effectively to give the molecular bundles very great strength. The effect of heat on silk is similar to its effect on wool. Thus, at a high temperature, silk will burn. But the molecules in silk are not joined together by cross-links as in wool; so, there are no cross-links between the molecules to break down or rebuild. Silk is therefore able to withstand higher temperatures than wool.

1.3 Regenerated fibers

Interest in the manufacture of different forms of rayon has resulted in the production of regular rayon, hollow viscose, spun-dyed filaments and staple rayon, crimped rayon and surface modified fibers, high tenacity rayon and high wet modulus (polynosic) rayon fibers. In chemical composition, viscose rayon and cotton are alike; they are both cellulose.

In cotton, the cellulose molecule consists of some 2000 to 10 000 anhydroglucosidic units linked together to give a high proportion of crystalline material (70–85%). The crystallites in cotton are orientated with respect to each other, forming microfibrils which in turn are arranged into fibrils, and the fibrils into filaments. During the manufacture of viscose, natural cellulose fibers are dissolved, resulting in some depolymerization and reduced crystallinity of the cellulose in viscose rayon. This renders the fiber more responsive to water. Thus, viscose rayon will absorb twice as much water naturally from air as cotton does. Viscose rayon has a moisture regain of 13% under standard conditions, and when soaked in water it will increase in length by 3–5% and swell to double its original volume. Viscose rayon loses as much as half its strength when wet, and is more easily stretched.

The differences between regular and high-tenacity rayon are to be found in the degree of degradation of the cellulose which has occurred during preparation of the viscose, the degree of crystallization, the size of the crystallites, the degree of orientation and the fine structure and uniformity of the filament.

1.4 Fiber identification

The identification of textile fibers is a task frequently performed in a textile laboratory. The need to identify fibers arises in fibers research as well as during fabric production and processing. The identification of an unknown fiber in a yarn

or fabric made up of a mixture of fibers is also often carried out. Identification tests are performed by utilizing tests that take advantage of the different chemical (and to some extent physical) characteristics of fibers. While there are some very elaborate analytical instruments that can be used to identify the chemical composition of materials and fibers in most circumstances, the textile laboratory relishes simple qualitative methods of identification of fibers. Here, three procedures are introduced, involving burning, solubility and dye staining tests of the fibers. These experiments will enable the reader to gain some insight in identifying various textile fibers using simple techniques.

1.4.1 Fiber identification by burning

Purpose: To make some observations about the reaction of various fibers to an open flame.

Procedure: Obtain 1–2 cm lengths or tufts of the various fibers or yarns to be tested from the samples provided. Perform the following tasks and carefully record the information in Worksheet 1 provided below. Worksheets may be adjusted as necessary to suit the purpose of the reader.

1. Hold the individual fiber samples to be tested in tweezers or tongs and bring the fibers slowly to the side of a Bunsen burner flame. Make observations. What is the initial reaction? Does the fiber shrink? Melt? Anything else?
2. Place the fiber in the flame and slowly withdraw it. Does the fiber burn?
3. If burning occurs, describe the flame. Color? Sooty?
4. Does burning continue or is fiber self-extinguishing?
5. If burning continues, extinguish it and carefully smell the smoke. Describe the smell.
6. Observe the remains of the ash (burn) product. Color? Black? Pale brown? Does it crumble? Is it hard? Bead-like?

1.4.2 Fiber identification by solubility

Purpose: To examine the reaction of fibers to solvents and to use these observations to identify fibers.

Background: In the burning test, natural fibers like cellulose and wool could be distinguished from the synthetic fibers (nylon, polyester, acrylic) fairly well. Among these synthetic fibers there is some confusion using the burning test. Since fibers are polymeric materials they can react with solvents in different ways. Thermoplastic fibers may dissolve in a common solvent like acetone. Also some highly semi-crystalline (thermoplastic) fibers like nylon and polyester will dissolve only in harsh ‘solvents’ like formic acid or boiling dimethylformamide (DMF). Here chemical dissolution of the fiber polymer occurs. Natural fibers like the cellulosics and protein fibers are thermosetting polymers. They are found to

Worksheet 1 Fiber identification by burning

	Initial reaction	Burning	Description of flame	Self-extinguishing	Smell	Remains
Cotton						
Wool						
Silk						
Acetate						
Polyester						
Acrylic						
Nylon						
Viscose						
Polyolefin						
Glass						

dissolve chemically in strong acid or base solutions. Other 'solvents' have been found that are quite specific in dissolving certain fibers. It is this specificity of solubility that allows for the determination of the quantitative composition of various fibers in blended fiber fabrics, e.g. polyester/cotton, nylon/wool. In this experiment, one can study the solubility behavior of various fibers in a series of solvents and observe the unique solubility behavior of textile fibers.

Worksheet 2 Fiber identification by solubility: synthetic fibers

	Acetone, room temperature	Formic acid, room temperature	DMF, 90 °C
Acetate			
Modacrylic (SEF)			
Polyester (Dacron 64)			
Nylon 6			
Nylon 6, 6			
Acrylic (Orlon)			
Polyolefin (polypropylene)			
Fiber glass			

Procedure

(A) *Identification of synthetic fibers*: Set up a series of 15 test tubes containing about one (1) ml of the following: five with acetone, five with 90% formic acid and five with DMF. (ALL THREE OF THESE SOLVENTS ARE HAZARDOUS!!! EXPERIMENTS MUST BE CARRIED OUT IN THE HOOD AND CARE TAKEN NOT TO SPILL ANYTHING.) To each of the solvents add a 0.5–1 cm length of yarn to determine its solubility. The five fibers to be tested are acetate, polyester, acrylic, nylon and viscose. Gently swirl each test tube and use a glass rod to poke and stir the fiber to effect solubility. Make observations on Worksheet 2, supplied above. Note that it may take a few minutes for these samples to dissolve. For the DMF samples, using a test tube holder, place the test tube in a water bath (IN THE HOOD!!!) and make some observations. Note that complete dissolution may not be possible and may mean weakening to a jelly-like mass of the fiber in the

Worksheet 3 Fiber identification by solubility: natural fibers

	NaOH 10% at room temperature		
	Immediate effect	After 10 min	After 20 min
Cotton			
Wool			
Silk			

	NaOH 10% at 40 °C		
	Immediate effect	After 10 min	After 20 min
Cotton			
Wool			
Silk			

	Sulfuric acid 70% at 40 °C		
	Immediate effect	After 10 min	After 20 min
Cotton			
Wool			
Silk			

liquid. Check this with a glass rod. Make all your observations about the solubility of these five fibers in the three solvents in Worksheet 2.

(B) *Natural fiber solubility tests*: Here the same general procedure and test tube set-up will be used except that we will be testing the solubility of three natural fibers – cotton, wool and silk in two solvents:

- Use three test tubes each with 10% caustic soda (NaOH) at room temperature (and eventually at 40 ºC)
- Use three test tubes each with 70% sulfuric acid at 40 ºC.

Make the same type of observations as described for the synthetic fibers section. Record all the observations in the Worksheets.

1.4.3 Fiber identification by dye staining

Procedure: Following the instructions provided for using DuPont Fabric Dyestain #4, identify the unknown fabric samples assigned for identification. Be sure to:

1. Put a 1 cm wide strip of 'multifiber fabric' in your dyebath as an internal control.
2. Confirm the identity of your fabric by performing a burning or a solubility test on your unknown materials.
3. Report the results of your unknown determinations on Worksheet 4.

Worksheet 4 Fiber identification by dye staining

1. Unknown # (mount samples in spaces)	Identified as Dyestain test: _____ Confirm test: _____
2. Unknown #	 Dyestain test: _____ Confirm test: _____
Multifiber control (to be mounted here)	

1.4.4 DuPont Dyestain #4 instructions

(DuPont fiber identification dye #4)

Source: PYLAM Products Company Inc, 2175 East Cedar Street, Tempe, AZ 85281-7431, USA. Tel: +1 480-929-0070, Fax: +1 480-929-0078

Product use: Identify fiber/yarn/fabric types by color 'staining' technique. Also useful for identifying polymers in general.

Product composition: Dye mixture – Acid Blue 298, Acid Red 182, Direct Blue 218, Disperse Orange 25, Disperse Yellow 3, Direct Yellow 11.

Procedure

1. Wet out material (unknown) with hot water.
2. Place material into boiling 1% by weight water solution of DuPont #4 dye stain. Use a 20:1 (liquor:fiber) bath ratio.
3. Boil for 1 min.
4. Remove, rinse and dry.
5. Compare color results with 'standard' color strip.

1.4.5 Quantitative determination of the percentage of fiber in a yarn/fabric blend (solubility test)

Procedure

1. Weigh a watch glass.
2. Take a piece of blend fabric in which one of the components is either cotton or wool and the other is a synthetic. Place it on the watch glass and dry it in a 100 ºC oven for 10 min. Weigh the fabric and watch glass and calculate the weight of fabric.
3. In a fume hood:
   - If it contains cotton, put it in a beaker of 70% sulfuric acid.
   - If it contains wool, put it in a beaker of 5% caustic soda and 'Clorox' bleach.

   Leave for 15 min, stirring occasionally.
4. Remove what is left (the synthetic part of the blend) into a beaker of water with care, and stir once more.
5. After 5 min, remove the synthetic component, rinse carefully and transfer to the watch glass. Dry in the oven.
6. Weigh the synthetic component and calculate the weight of the synthetic component.
7. Calculate the percentage of the blend fabric.
8. Fill the table below with your data.

Fabric tested: Blend of _____

Table 1.2 Calculation of percentage makeup of blend

Weight of watch glass	*a*	g*
Weight of blend fabric and watch glass	*b*	g
Weight of fabric	*b–a*	g
Weight of synthetic component and watch glass	*c*	g
Weight of synthetic component	*c–a*	g
% of synthetic in the blend	100 × (*c–a*)/(*b–a*)	%

*Add data to this column.

Result: Fabric tested consists of % _____ and % _____ .

Table 1.3 Summary of typical observations

Fibre	Burning characteristics	Odour	Ash
Cotton	Burns with a flame. Has an afterglow	Burning paper	Black and powdery
Polyester	Melts and burns with a sputtering flame. Gives off thick black smoke	Faintly sweet, slight geranium odour	Hard, black, round and shiny
Polypropylene	Melts and burns with steady flame. Clear flame, no smoke. Looks like melting glass. Melted portion is clear.	Very little odour. Slight celery odour	Hard, turns opaque
Nylon	Melts and burns with sputtering flame. Gives off white smoke	Burning garbage	Hard, round, gray or brown, shiny
Nomex	Very slow to ignite. Will not support combustion. No melting. Material chars and curls up	Faintly sweet	Black, dull finish crushes into black powder
Acrylic	Melts and burns rapidly. Sputtering flame. Thick black smoke	Faintly sweet, slight 'hot iron' odour	Resembles burned head of wooden match; crucibles into black or brownish orange powder

Details of single fiber analysis have been given by Bresee.[14] Therefore, in this chapter, only highlights of some of the important methods for fiber identification have been considered.

1.5 Density measurement

The density of any material is defined as its weight per unit volume. Textile fiber density is more conveniently determined indirectly by comparing the sample with standards of known density. The two commonly used techniques are the sink–float and gradient density methods.

The sink–float method requires a beaker, pipette, burette and two liquids. The liquids must be miscible and inert to the fiber being tested. One liquid must be less dense than the fiber and the other liquid must be more dense. A known volume of liquid A is pipetted into a beaker and the fiber is immersed in the liquid. The second liquid, B, is then added dropwise from a burette to the beaker with constant stirring. As the density of the liquid solution in the beaker changes, a point is reached where the density of the liquid precisely equals that of the fiber and the fiber will neither sink nor float but will remain suspended in the liquids. The volume of the second liquid added to the beaker is recorded and the density of the fiber is calculated:

$$d_{\text{solution}} = d_{\text{fiber}} = \frac{d_{\text{A}}V_{\text{A}} + d_{\text{B}}V_{\text{B}}}{V_{\text{A}} + V_{\text{B}}} \qquad [1.1]$$

where d refers to density, V refers to volume and A and B refer to the first and second liquids respectively.

The density gradient method is more complicated and can provide density measurements to five significant figures. A density gradient apparatus can either be purchased or constructed inexpensively as detailed elsewhere.[15] Density gradient analysis consists of preparing a density gradient column, calibrating the column and then introducing the fiber sample to the column for measurement of its density. The column is a vertical tube containing two miscible liquids such that the density in the tube changes continuously from top to bottom. The column is calibrated by immersing several objects of known density in the column and then plotting their locations in the column versus their densities on graph paper. Glass spheres of known densities are used as calibration floats. The fiber is dropped in the column and when it has settled in the column, its location is recorded. Finally, the density of the fiber is determined by noting the density on the calibration curve corresponding to its location in the column.

Density measurements can be used in the calculation of the percentage crystallinity of fibers. Fiber degradation has also been monitored by both sink–float and density gradient measurements.[16]

1.6 Use of infrared spectroscopy

The appearance of the first research grade Fourier transform infrared (FTIR) spectroscopy in the early 1970s initiated a renaissance and opened the door for the use of the technique in many analyses, including textile fiber identification. The basis of FITR spectoscopy is the two-beam interferometer. Details of the design, techniques and applications are adequately covered elsewhere.[17,18] The fundamental equation for spectrometric quantitative analysis is known as Beer–Lambert–Bouguer law, sometimes shortened to the Beer–Lambert law. To ensure an acceptable quantitative method, it is important to obtain reference spectra of the analyte and all other components. Also, the best method of sampling must be employed and the system calibrated. Finally, a validation sample must be prepared for evaluation. Polymers and fibers are usually analyzed as pressed films although solid samples can be analyzed directly if the FTIR apparatus has an appropriately attached microscopic unit. Sometimes, absorption band ratios are used and give the best results.

Positive identification of synthetic fibers can be made using standard analytical methods published by AATCC (American Association of Textile Chemists and Colorists), ASTM (American Society of Testing Materials) and The Textile Institute, Manchester.

1.7 Other methods of surface analysis

Many new instruments are now available which can be used to characterize various depths of a specimen. A brief account of the use of these techniques will be presented here.

1.7.1 ESCA (XPS)

Electron spectroscopy for chemical analysis (ESCA) is used for characterizing polymer surfaces and is also known as X-ray photoelectron spectroscopy (XPS). This method is based on the observation that electrons are emitted by atoms under X-ray irradiation. The energy of the emitted electrons yields the binding energy of the electron to the particular atom.[19]

1.7.2 SEM

Scanning electron microscopy (SEM) constitutes one of the older and one of the most widely used instruments for surface analysis. It provides a three-dimensional visual image and, thus, the quantitative analysis is relatively straightforward.

1.7.3 SSIMS

Static secondary ion mass spectroscopy (SSIMS) ranks with XPS as one of the principal surface analytical techniques. Treatment of polymer surfaces to improve their properties with respect to wetting or water repulsion and to adhesion, is by now a standard procedure. The treatment is designed to change the chemistry of the outermost groups in the polymer without affecting bulk properties. One popular surface treatment is plasma etching. The use of SSIMS is most amenable to the surface evaluation of such treated materials.

1.8 References

1 Wingate I B and Mohler J F, *Textile Fabrics and their Selection*, Prentice-Hall, Englewood Cliffs, NJ, 1984.
2 Ward K, *Chemistry and Chemical Technology of Cotton*, New York, Interscience Publishers, 1955.
3 Hall, A J, *Cotton-cellulose: Its Chemistry and Technology*, New York, D. Van Nostrand, 1924.
4 McCall, E R and Jurgens, J F, 'Chemical composition of cotton', *Textile Res. J.*, 1951, **21**, 19.
5 Trotman, E R, *Textile Scouring and Bleaching*, London, Charles Griffin & Company, 1968.
6 Bean, P and McCleary, W A, *The Chemistry and Practice of Finishing : A Practical Treatise on Bleaching, and the Finishing of White, Dyed, and Printed Cotton Goods*, Manchester, UK, Kirkham & Pratt, 1912.
7 Ugbolue, S C, in Structure/Property Relationships in Textile Fibres, *Textile Progress*, 20, No.4, P W Harrison (ed), Manchester, UK, The Textile Institute, 1990, p 4.
8 Karthik R, Fan, Q and Ugbolue, S C, Thermo-responsive finishing of cellulose fabrics using PNIPAAm grafting for smart applications, *226th ACS National Meeting*, New York, September 7–11, 2003.
9 Stout, H P, in *Handbook of Fiber Science and Technology,Vol.IV, Fiber Chemistry*, M Lewin and E M Pearce (eds), New York, Dekkar, 1985, p 701.
10 Batra, S K, in *Handbook of Fiber Science and Technology, Vol.IV, Fiber Chemistry*, M Lewin and E M Pearce (eds), New York, Dekkar, 1985, p 727.
11 Bertoniere, N R, Rowland, S P, Kabir, M and Rahman, A Q, 'Gel permeation characteristics of jute and cotton', *Textile Res. J.*, 1984, **54**, 434.
12 Varma, D S, Varma, M and Varma, I K, 'Part 1: Effect of physical and chemical treatments on properties', *Textile Res. J.*, 1984, **54**, 827.
13 Cook G J, *Handbook of Textile Fibres, Manmade Fibres*, Merrow Publishing, Sheldon, UK, 1984, p xiii.
14 Bresee, R R, 'Single fibre analysis', in *Analytical Methods for a Textile Laboratory*, J W Weaver (ed), AATCC, Research Triangle Park, NC, 1984, Chapter 2, pp 9–28.
15 ASTM, *Annual Book of ASTM Standards, Part 35*, American Society for Testing Materials, Philadelphia, 1979, D1505–68, pp 533–539.
16 Stock, C R and Scofield, E R, 'Application of the specific gravity column to the quantitative determination of additives in a base material', *Textile Res. J.*, 1951, **21**, 521.

17 H Gunzler and A Williams (eds), *Handbook of Analytical Techniques*, Vols 1 & 2, Cambridge, WileyVCH, 2001.
18 Nettles, J E, *Handbook of Chemical Specialities: Textile Fiber Processing, Preparation, and Bleaching*, New York, John Wiley & Sons, 1983.
19 Sperling, L H, *Introduction to Physical Polymer Science*, third edition, New York, Wiley-Interscience, 2001, p 529.

2

Chemical analysis of feather and down textile materials

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2.1 Introduction

2.1.1 Feathers and down

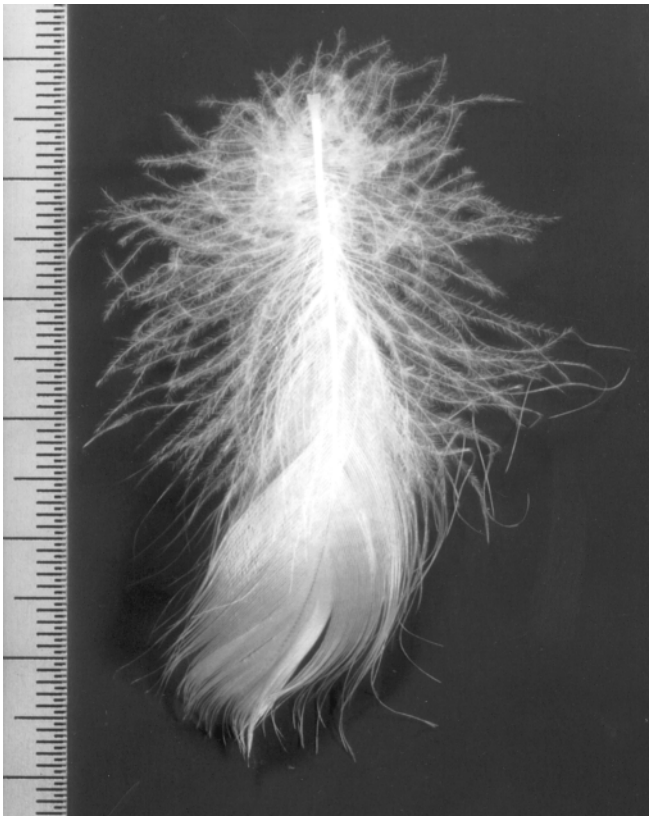
Feathers and down are two of nature's most marvelous products. Together they form the bird's plumage, but they are very different in structure. A feather has a two-dimensional structure, a quill from which barbules extend in two opposite directions like vanes and a compact and flat tip (EN 1885, 1998, p 4). Down has no quill but a small core, from which small clusters of barbs (with barbules and nodes) extend in three dimensions (EN 1885, 1998, p 4). It would be erroneous to assume that down is a small feather or would eventually develop into a feather.

Feathers form the outer, protective coat of the animal's body. They have structural functions, best seen in the strong wing and tail feathers. They are highly resilient. Down, only found in waterfowl, provides the necessary warmth insulation for the bird. The barbs are fluffy-elastic and at least as resilient as feathers. Thanks to its structure, down can trap a large volume of air, resulting in an almost unsurpassed heat insulation capability with respect to weight.

2.1.2 Test standards

Few people buy commodities knowing everything about them, nevertheless most consumers would like to feel comfortable understanding what is on the product labels. This is important because the standardized information provided on the labels enables producers and merchants of down- and feather-filled products to inform customers of their products' quality, and thus sell more goods.

In order to write, read, compare and understand the quality or performance data of down and feathers in a universal language, the International Down and Feather Bureau (IDFB), together with international and national standardization organizations, has developed methods and published standards for both testing and



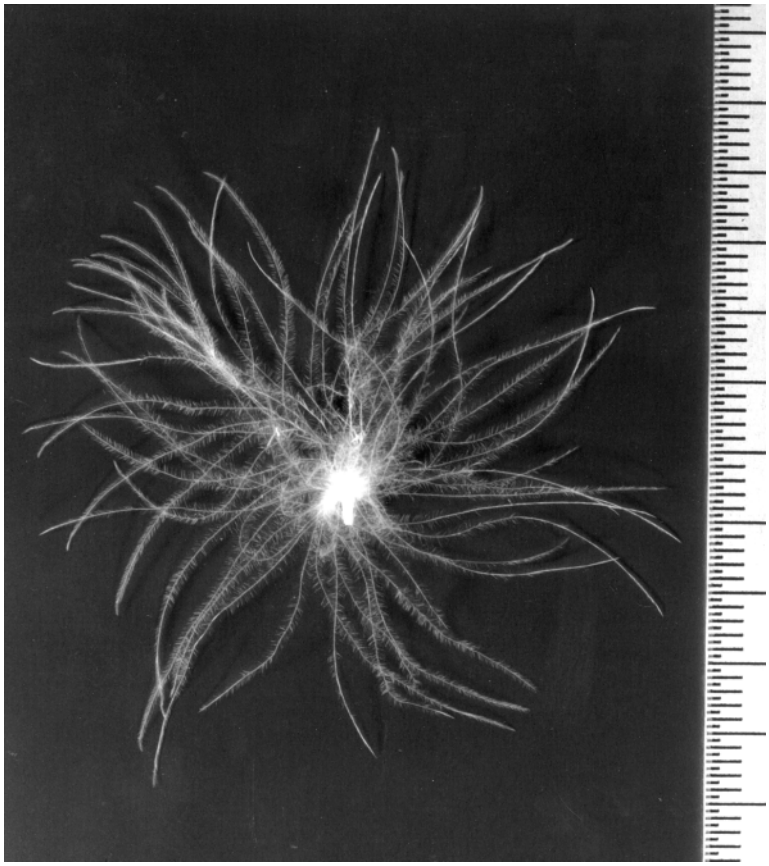
2.1 Micrograph of a feather (*source*: IDFL – International Down and Feather Laboratory and Institute).

characterizing down and feather material and products. In this chapter, information and data on the quality control of feathers and down are summarized, primarily from IDFB testing regulations and from European standards. European standards (EN) are determined by committees acting for the European Union in its effort to standardize regulations for inter-Europe commerce. References are also made to British and Japanese norms**.** When applying any of these methods, it is important that the procedures are followed in their entirety.

The characterization and testing of all materials, natural or synthetic, are determined by their physical structure and chemical composition.

2.1.3 The physical structure of feathers and down

As far as we know, few of nature's products are fully symmetrical, although if we could observe the nano-range of matter not currently visible to our eyes or through current technical vision aids, we may find many more. No feather, for example, is exactly the same on the left and the right side of the shaft, and none are entirely



2.2 Micrograph of a down cluster (*source*: IDFL).

straight. The barbs are not symmetrical (see Fig. 2.1). Furthermore, it is clear that an individual down cluster is not at all as symmetric as an individual snowflake, with which down is sometimes compared (see Fig. 2.2).

2.1.4 The chemical composition of feathers and down

Nature's polymer chemistry has selected two basic molecular building blocks for fibrous products.

- Flora: Cellulose is the construction material for the structural cells in plants. It is probably found in its purest form in cotton, having the general formula of a polysaccharide:

$$-[C_x(H_2O)_y)]_n- \quad [2.1]$$

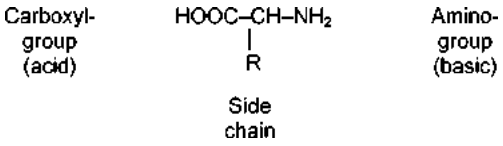
- Fauna: Animal and human tissue, like skin and leather, as well as fibers such as

hair, wool, and feathers and down, have proteins as their main building blocks. These are large molecules, assembled by nature from a variety of not much more than 20 different amino acids. Keratin is the predominant protein in fibers. It, too, is a macromolecule, with cysteine as one of the main amino acids:

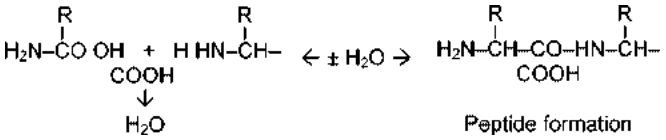
$$HS–CH_2–CH(NH_2)–COOH \quad [2.2]$$

Amino acids: building blocks for proteins

Proteins are composed by nature from (by weight) about 50% carbon (C), 20–25% oxygen (O), 15–17% nitrogen (N), 7% hydrogen (H) and, particularly in the keratins (the protein building blocks of hair and feathers), between 2.5 and 5% sulfur (S). A closer look at amino acid molecules reveals that they actually have an acidic head (carboxyl group) and an alkaline tail (amino group) (see Fig. 2.3). With this structure, they are capable of head-to-tail reactions, forming first peptides and then proteins, which are large molecules containing chains of amino acids linked by peptide bonds (see Fig. 2.4).



2.3 General chemical formula of amino acids (*source*: IDFL).



2.4 Peptides (*source*: IDFL).

The role of water

As can be seen from the chemical composition of down and feathers, water is an essential part of their chemical makeup. Not only is water a by-product of the polymerization of amino acids to proteins, it can also reverse this chemical reaction. Boiling water will split proteins into shorter chains of polypeptides, which is an intermediate step in the biochemical analysis of down and feathers.

In a manner similar to that of hair and wool, down and feathers have the ability to physically and even chemically absorb moisture from the atmosphere surround-

ing them. The rate is about 13% at standard atmospheric conditions (note: IDFB Testing Regulations Part 1, see References, Section 2.6.3, states that these are 20 ± 2 ºC and 65 ± 2% relative humidity).

As will be shown in Section 2.4, moisture plays an important role for the structural revival or reconditioning of down and feathers that have been vigorously compressed as bulk raw material in bales, or in a finished product, for example in a tightly rolled-up down sleeping bag.

2.2 Chemical analysis of feathers and down

2.2.1 Representative sampling

As the photos above (Fig. 2.1 and Fig. 2.2) indicate, down and feathers are not homogeneous. It is therefore very important to draw representative samples in testing either the raw material or the finished products. If only a single item is to be tested (bag, bale or manufactured article), three individual samples should be collected at three different sites in the item, i.e. from the upper, the middle, and the lower part, respectively. In the case of several packages or items belonging to one lot or batch, the number and quantity of samples are determined according to the following tables. (Sampling norms are somewhat different, and as this difference may be decisive in legal cases**,** the IDFB and the EN figures are shown in Table 2.1 and Table 2.2.

Table 2.1 Sampling of down and feather products or packages (>500 g/item)

Extent of delivery or lot		Number of packages or items sampled		Weight of each individual sample		Total sample quantity	
IDFB Part 2	EN 1883	IDFB Part 2	EN 1883	IDFB Part 2(g)	EN 1883(g)	IDFB Part 2(g)	EN 1883(g)
1	1	1	1	135	135	405	405
2–8	2–15	2	2	70	70	420	420
9–25	16–25	3	3	45	45	405	405
26–90	26–50	5	4	30	35	450	420
	51–90	(5)	5	(30)	30	(450)	450
91–280	91–150	7	7	20	20	420	420
	151–280	(7)	10	(20)	20	(420)	600
281–500	281–500	9	15	20	15	540	675
501–1200	501–1200	11	20	20	15	660	900
1201–3200	> 1200	15	25	15	15	670	1125
3201–10000		19	(25)	15	(15)	860	(1125)

Source: EN 1883, 1998, Table A.1: Packages filled with more than 500 g (Section 2.6.2); IDFB Testing Regulations Part 2 (Section 2.6.3).

Table 2.2 Sampling of down and feather products or packages excluding pillows (<500 g/item)

Extent of delivery or lot		Number of packages or items sampled		Weight of each individual sample		Total sample quantity	
IDFB Part 2	EN 1883	IDFB Part 2	EN 1883	IDFB Part 2(g)	EN 1883(g)	IDFB Part 2(g)	EN 1883(g)
1	1	1	1	35	40	105	120
2–25	–	2	(2)	17	(20)	102	(120)
–	2-90	–	2	–	20	–	120
26–280	–	3	–	13	–	102	–
	91–150	(3)	3	(13)	14	(102)	126
	151–280	(3)	4	(13)	10	(102)	120
281–500	281–500	5	6	7	7	105	126
501–1200	501–1200	7	7	5	6	105	126
1201–3200	> 1200	9	9	5	5	135	135

Source: EN 1883, 1998, Table A.2: Package(s) filled up to 500 g and manufactured articles (Section 2.6.2); IDFB Testing Regulations Part 2 (Section 2.6.3).

2.2.2 Determination of moisture content

While a lower than normal moisture content may be observed in very dry climates, down should be able to return to its natural moisture percentage of 13% if stored under standard climate conditions. If it does not, however, this may be an indication that the down has been mistreated thermally or chemically during the preliminary processes or while in use. A higher than normal moisture content may indicate that the material has been stored or transported under wet conditions, which may result in microbiological or pest damage. If the moisture content is too low, feathers and down lose their resilience and may become brittle. The determination of moisture content is therefore of chemical, physical and biological importance in the quality control of feathers and down. The IDFB Testing Regulation, Part 5 demonstrates how this is done (also refer to EN 1161: 1995E relating to moisture content, see References, Section 2.6.2):

Four to five grams of the representative down–feather specimen are placed into a weighing bottle which has been dried at 105–110 ºC for at least 1 h, then cooled in a desiccator. The sample is dried at 105–110 °C for 2 h, and then allowed to cool in the desiccator. The weight loss is measured (repeating until the weight is constant within 1 mg), calculated and reported as *xx.x*%.

2.2.3 Ash chemical analysis

The chemical formula of cysteine:

$$HS–CH_2–CH(NH_2)–COOH \qquad [2.3]$$

illustrates that the proteins in feathers and down, mainly keratin, contain sulfur (S) in addition to the main components of amino acids: carbon (C), hydrogen (H), oxygen (O) and nitrogen (N).

Ash analytical methods may thus be used to test for evidence of protein-based matter including waterfowl down and feathers. But the characterization or even identification of bird species can hardly be definitely determined on the basis of chemical element percentage alone. One reason is the difficulty of collecting a chemically representative sample from a fairly non-homogeneous natural product.

Nonetheless, the ash chemical methods used for identifying chemical elements present in down and feathers do serve to find and identify foreign matter and undesirable additives. Two examples are:

1. Barium salts added to increase (illegally) the final weight and to give artificial whiteness to raw or pre-processed down.
2. Metal-based chemicals and halogen compounds added to reduce the (in itself not very risky) potential flammability of feathers and down, or additionally, for pest control. Most of these substances are currently prohibited as additives in natural or synthetic materials meant for human use.

In short, the traditional chemical–analytical methods for detecting specific elements or molecules is (despite the application of modern identification detectors) predominantly used in basic research or for detective investigations, but are rarely applied in the day-to-day quality control of down and feather filling materials.

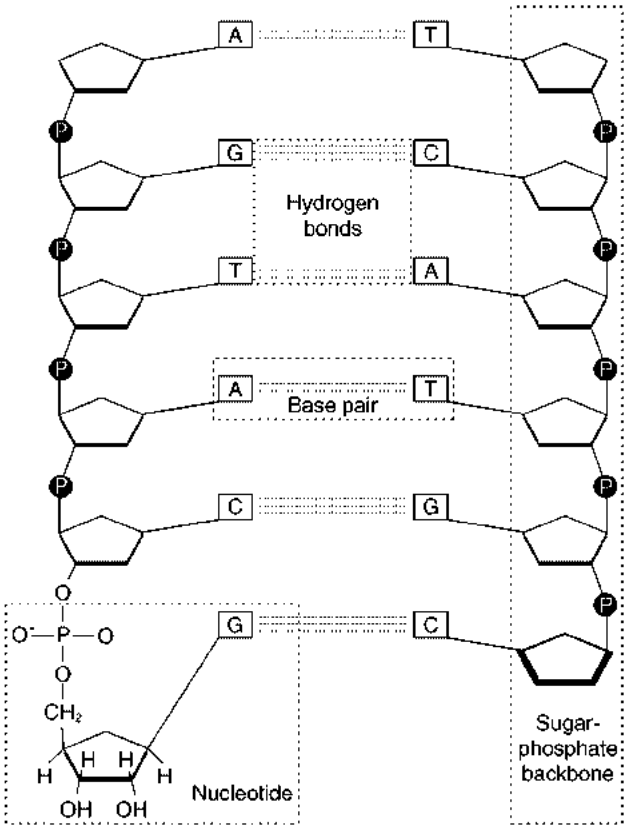
2.2.4 Biochemical analysis

Each cell of an animal, including the structural cells of down and feathers, carries megabytes of genetic information. This is stored in an orderly manner in strands of DNA, nature's 'read only' memory. They most often are found paired with a partner strand, wrapped around each other in the familiar double helix. Most of this information remains stored and protected when it is not actively used either to propagate information or for repairs should it become corrupted (see Fig. 2.5 DNA Chain).

SIAM method for species identification

Specific Identification of Animals by MALDI–TOF mass spectrometry (SIAM) was developed by Wolfgang Altmeyer, Gene-Facts GbR in cooperation with Klaus Hollemeyer and Elmar Heinzle, Saarland University, Saarbrücken, Germany (http://www.gene-facts.com/ENGLISH/SIAM/siam.html).

In this method, proteins are cleaved by specific enzymes, the restriction sites being defined by the amino acid sequences. The fragments generated by this treatment differ in length and molecular weight, according to the species-specific



2.5 DNA chain (*source*: IDFL) The code is quite easy to read. The cells read it by scanning down a messenger RNA (copied from the DNA) and use ribosomes to build proteins based on the code that is read. Researchers read the code by stepping down one nucleotide at a time, clipping it off to identify it, thus determining the sequence of a DNA strand. Hundreds of different proteins are built in to interact with the information contained in the DNA. Their sequence is genetically determined and varies between different animal species.

composition of proteins. Owing to the high content of repetitive sequences, many species-specific (as well as unspecific) peptides are created. These peptides are then separated by highly sensitive MALDI-TOF mass spectrometry (matrix assisted laser desorption ionization time-of-flight mass spectrometry), generating patterns of peaks according to the different molecular weights. The peaks are then analyzed for species-specific patterns.

General description of test method

The feather or down material to be analyzed is put into a PCR (polymerase chain reaction) container, containing a solution of 25 mM NH_4HCO_3 and

2-mercaptoethanol. The PCR container is then placed into a boiling water bath for 20 min, during which time the disulfide bridges of the proteins are opened, thus becoming accessible to the enzymes. The container is then immediately cooled down on ice.

A solution of 25 mM NH_4HCO_3 and trypsin (a protein-cutting enzyme) is added and incubated for 3–4 h at 37 °C. Following this incubation, the enzyme is chemically deactivated and 1 ° µl of the liquid is brought onto a sample plate and evaporated. The sample plate is then placed into the mass spectrometer and analyzed. The time of flight in the mass spectrometer is dependent on the molecular mass of the fragments produced during the preceding 'digestive' procedure. As stated above, the spectrogram can now be analyzed for patterns specific to the bird species.

Test accuracy

To date, the MALDI-TOF spectrometer provides the most specific separation spectrograms. Based on numerous test series with a known reference material, an extensive data base of spectrograms has been acquired. For goose, duck, eider duck, chicken, pheasant and turkey, the accuracy of bird species identification is almost 100%. Accuracy of the visual quantitative method is ±5%. For most quality control purposes, this is sufficient (see also Section 2.4, Visual analytical methods).

2.2.5 Isotopic analysis

Because radioactive elements decay at a known rate, they are increasingly used for identifying sites and objects. Stable isotope ratios can be propagated through the environment from geological formations into water or from food into body tissue. Thus flora and fauna living in such environments will receive a geochemical stamp allowing isotopic analysis to provide information on the origin of materials. This procedure has, for example, been accomplished for meat and grapevines. It may eventually be applied to down and feathers, making it possible to identify the plumage of birds according to their native geographic areas, based on isotopic ratios of trace elements and on isotopic data available on agricultural soil and, in consequence, on plant food consumed by ducks and geese (during analysis, however, it must be taken into account that birds fly long distances, even between continents, and that animal food may also be transported over similar distances).

Until now, these methods have primarily been used in archeology, including the study of ancient nutrition sources of plants or animal diets determined from potsherd residues. However, by plotting data of several elements together, and with more geochemical information, specific maps are becoming available that give very localized indications on the plant source of the food chain of humans or animals. Such maps have already been successfully applied to modern day criminology.

Current instrumentation allows for the analysis of very small samples, and such progress in separation chemistry means that using the above approach is now feasible. Accelerator mass spectrometry (AMS) for detecting the radioactive isotope of carbon (^{14}C) in relation to the stable isotopes once required specimen sizes of one gram or more four decades ago, to sample masses today containing only 0.1–1 mg of carbon are adequate. Measurement times have been reduced from weeks to hours. Stable isotopes of lighter elements such as carbon, nitrogen, oxygen, and even hydrogen (e.g. from proteins) can now be measured with very high precision using isotope ratio mass spectrometry (IRMS).

2.3 Chemical analysis of extracts

Raw down and feathers are obviously not clean and must be washed and separated in various steps. Depending upon how effective the washing equipment is, varying amounts of impurities can be detected through the use of water and solvents.

2.3.1 Aqueous extracts

Acidity (pH)

According to IDFB Testing Regulations, Part 6 (see Section 2.6.3), acidity (pH) is determined in the following manner:

Sample preparation
In a 250 ml Erlenmeyer flask 1 g ± 0.01 g of the test specimen (cut to pieces of approximately 1.5 mm) is macerated with 5 ml of boiled distilled water until all material is wet. Then 65 ml of boiled distilled water is added and the flask is stoppered and allowed to stand for 3 h at room temperature. It must be occasionally shaken mechanically or by hand.

Measurement
Without removing the material, the temperature is adjusted to 25 ± 1 ºC and the pH is measured potentiometrically. Report the pH to the nearest 0.1 pH unit. Occasionally the acidity is measured from the aqueous extract prepared for the determination of oxygen number and turbidity (see below) but this method is not standardized.

Oxygen number (based on IDFB Testing Regulations, Part 7 – see Section 2.6.3; EN 1162, 1996– Section 2.6.3)

Sample preparation
Ten grams ± 0.1 g of the representative down or feather sample is placed into a 2 l plastic jar. One liter of distilled or de-ionized water (grade 3) is added and the jar is closed with a watertight lid. It is then shaken at least 10–15 times by hand to ensure

that the plumage begins to absorb water. Then the jar is placed in a horizontal position on the shaking machine. The jar is shaken at room temperature for 30 min (European Norm (EN) 1162 requires at least 1 h) at a speed of 150 shakes per minute. The resulting liquid is filtered through a sintered glass filter with pore size P160 without squeezing or wringing the liquid from the down and feathers.

Measurement

A 100 ml sample of the liquid is transferred into a 400 ml beaker and acidified by adding 3 ml of 3 mol l^{-1} (ca 25%) sulfuric acid; the beaker is placed onto a magnetic stirrer. By adding N/10 (0.02 mol l^{-1}) potassium permanganate ($KMnO_4$) 0.02 ml at a time, the liquid is titrated until a faint pink color persists for 60 s. The test is repeated with another 100 ml of the liquid sample and a blind test is done with plain water.

Calculation

A = quantity in ml of potassium permanganate solution used by the test sample, *B* = quantity in ml of potassium permanganate solution used in the blank test, oxygen number = 80 × (A–B). Use the arithmetical mean of the two measurements, rounded to 0.1.

Turbidity

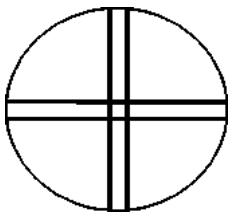
Sample preparation

The test liquid is prepared in the same manner as for the oxygen number; thus, the test liquid prepared for the oxygen number can also be measured for turbidity. Turbidity is measured with a glass turbidity tube (according to IDFB Testing Regulations, Part 11-B – see Section 2.6.3; EN 1164, 1998 – see Section 2.6.2).

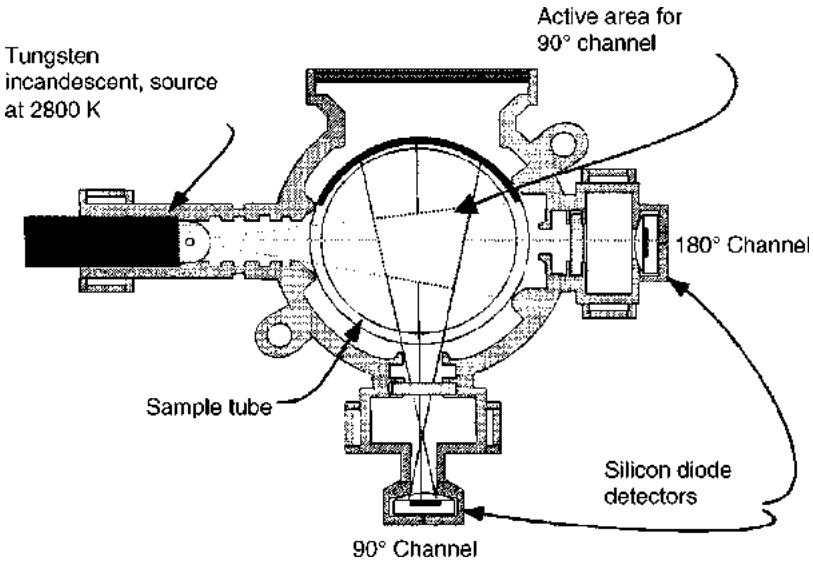
A disk with a double-cross marking (see Fig. 2.6) is placed at the bottom of a 550 mm or longer glass cylinder or tube. A light source of at least 600 lux (e.g. a strong flashlight) is properly positioned to illuminate the double cross marker. The cylinder is then filled with the test liquid. After 60 s, the level of the liquid is lowered until the cross is visible through the liquid. The height of the liquid is at that point recorded as 'turbidity in mm.' Test liquid is again added and then lowered until the cross is visible. The two visibility readings should not differ more than 10 mm, or the refill and lowering steps must be repeated. (EN 1164 recommends connecting the measuring tube to a communicating flask, allowing the liquid level to be raised or lowered more easily to the height of the visibility of the double cross.)

Turbidity is measured with an automated NTU meter (according to IDFB Testing Regulations, Part 11-A – see Section 2.6.3).

In an extensive IDFB round robin test made during spring 2002, the accuracy of the conventional tube-visibility test and the nephelometric measurement of turbidity units, or NTU, was compared. The findings were presented at the June 2002



2.6 Turbidity visibility chip. (*source*: IDFL).



2.7 LaMotte 2020 nephelometric turbidity meter. Light passing through clear water will travel in a straight line. Particles in turbid water will cause the light to scatter, giving it a cloudy or foggy appearance. The turbidity of a sample is determined by measuring the amount of scatter when light is passed through a sample. The higher the turbidity, the greater the amount of scatter. (*source*: *LaMotte 2020 Turbidimeter Instruction Manual*, p 9, source: online www.lamotte.com 2020 Turbidity Meter Instruction Manual; reprinted by permission).

IDFB meeting in Vancouver BC and both methods were approved as equally accurate (source: International Down and Feather Laboratory, IDFL; results on file).

The IDFB Testing Regulations, Part 11-A, as well as the data shown below, are based on tests performed with the LaMotte 2020 turbidity meter (see Fig. 2.7).

Procedure

The vial of the turbidity meter is filled with the liquid which is prepared as prescribed for the oxygen number test. Next, the NTU value is measured as

Table 2.3 Conversion of NTU to mm visibility height (*source*: IDFL)

Test tube visibility height (mm)	100	200	300	400	500	600	750	1000
Nephelometric turbidity unit (NTU)	30.0	15.0	10.0	7.5	6.0	5.0	4.0	3.0

prescribed in the operating instructions of the instrument (a very easy push-button test). At least three readings are taken without removing the vial.

Comparison/conversion

For the range of the 0–1000 mm tube, visibility height and the 0–50 NTU range of nephelometric measurements with the LaMotte 2020 turbidity meter, a conversion of the following relation was determined:

$$y = 3341.2x^{-1.0379} \quad [2.4]$$

where x is mm visibility height and y are the NTU turbidity units.

For practical use, the above relation may be rounded to:

$$x = 3000/y \quad [2.5]$$

For a first estimate, Table 2.3, conversion of NTU to mm visibility height, may be helpful.

The results of the turbidity tests, both tube and NTU above, are indicators for the cleanliness of the down and feather product. It is for this reason that some government regulations already request an NTU value of no more than ten (equaling a visibility level greater than 300 mm).

2.3.2 Solvent extracts

Oil and fat content

Oil and fat content is determined according to IDFB Testing Regulations, Part 4 (see Section 2.6.3) and EN 1163 (1996) – see Section 2.6.2.

Sample preparation

A representative sample of 4–5 g is weighed, put into an extraction thimble and oven-dried for 1 h at 105–110 ºC, then allowed to cool to room temperature in a desiccator. Then it is re-weighed in the same manner as in determining the moisture content (see Section 2.2).

The dried and weighed sample is put into an extraction thimble which is then placed in the Soxhlet extractor, from which the solvent-soluble ingredients are extracted from the down and feathers through at least 20 siphonings.

Solvents

Different norms specify different solvents. Diethyl ether, formerly widely used,

has lost its importance owing to the fear of peroxide explosions and also because its fumes are much heavier than air, allowing them to accumulate on the floor in potentially inflammable concentrations. Furthermore, it has a fairly wide flammability concentration range. Dichloromethane is still specified in some norms, but chlorinated solvents are increasingly being banned from general use by environmental regulations. Therefore, IDFB Testing Regulations Part 4 specifies petroleum, 60–80 ºC fraction (also called naphtha or petroleum ether):

The extract is filtered and the solvent first distilled off in the hot water bath, then evaporated at 100–105 ºC until at a constant weight. The oil and fat content is then calculated as a percentage of the weight of the test sample.

An oil and fat content of 0.5 to 1.5 percent in the pre-processed down–feather filling material is not only considered normal, but also desirable. Markedly higher oil–fat content may indicate insufficient cleanliness. Down and feathers with a too low oil and fat content become brittle and will therefore not be suitable for long-term use. (This is the main reason washing is preferred over chemical cleaning for down-filled products.)

Chemical analysis of the solvent extract

Odor is a first chemical indicator. For instance, natural fats and oils smell different from synthetic finishing chemicals. Chromatographic separation methods will reveal in more detail what has been left on the feathers during cleaning and what has been added during processing. These and other methods for the analysis of solvent extracts are particularly valuable for the verification of biological claims, or for the detection and even identification of traces of both permitted and prohibited chemical treatment methods. Each of these methods is, however, not usually part of routine testing, but is applied in more extensive quality control of down and feather products and the methods are often tools used to investigate complaints further.

2.4 Visual analytical methods

2.4.1 Content analysis

Content analysis according to IDFB Regulations, Part 3 (Section 2.6.3) and EN 12131 (1998) (Section 2.6.2) is as follows:

A separating cabinet (see Fig. 2.8) is equipped with enough weighing containers, usually glass beakers, to segregate the components and to contain them during weighing. A representative sample is placed in the bottom section of the cabinet. This is a 6-g sample with a declared or expected down content equal to or less than 30% or 4 g if the down content is higher than 30%.

First separation
Using tweezers, the individual pieces of plumage are separated into (A) whole

2.8 Separation cabinet (*source*: Peter Lieber).

waterfowl feathers, (B) whole land fowl feathers, (C) broken or damaged waterfowl feathers, (D) residue, and (Q) quill feathers (feathers which are over 100 mm in length or which have a quill point exceeding 9.5 mm in length). In this first separation, down clusters, plumules, nestling down, down fiber, and feather fiber are collected in container (E). The contents of all containers are weighed to an accuracy of 0.1 mg calculated as a percentage of the total weight of all components. (EN 12131 tolerates a maximum weight loss of 2% during this separation step.)

Second separation

A representative minimum sample of 0.2 g from the down/fiber container (E) is then subdivided in a second separation into down clusters (F), down fiber (G), waterfowl feather fiber (H), land fowl feathers and land fowl feather fiber (I) and residue (K). These components are also weighed and expressed as a percentage of the total weight of all components.

Reporting

The components, according to IDFB Testing Regulations Part 3 are reported as follows:

- F % down cluster
- G % down fiber
- A % waterfowl feathers

- H % waterfowl fiber
- C % broken and damaged waterfowl feathers
- Q % quill feathers
- B + I % land fowl feathers and fiber
- D + K % residue
- 100 %

Evaluation and labeling

To represent the visual analysis of the contents of the different components, several quality and labeling standards have been established. Based on EN 12934, down and feather filling materials are qualified in classes (Table 2.4). A problem for both consumer and manufacturer is that it is impossible to produce, for example, products with consistent 100% down content. There are often disparities between what is printed on the label (the manufacturer's claims for the product) and the actual composition of the down–feather filling after it has been laboratory tested. Standards have been developed detailing permissible deviations from the goal percentage. Table 2.5 represents permissible labeling–test disparities. Table 2.6 shows the range of percentages of goose and duck allowed for specific content labeling claims.

2.4.2 Microscopic specie analysis

Biochemical analysis of bird species as described in Section 2.2.4 would in most cases provide a result with the best possible accuracy. However, this method is not readily accessible for production plants using on-site quality control. It is for this reason that visual tests by means of a microscope or microfiche (see Fig. 2.9) are

Table 2.4 Quality classification according to EN 12934 (1999)

Fowl species	Classification	Content of other elements (%)	Down/feather composition
Waterfowl	Class I (also: 'new')	up to 5	down %, feather %
	Class II	more than 5 to 15	down %, feather %
	Class III	more than 15	down %, feather %, other elements %
Land fowl and blends of land- and waterfowl	Class IV (also: 'new')	up to 5	down %, feather %
	Class V	more than 5 to 15	down %, feather %
	Class VI	more than 15	down %, feather %, other elements %
Land fowl and/or waterfowl	Class VII	not specified	unspecified composition

Table 2.5 Permissible disparities between test results and labeling (content analysis)

Denomination on label (%)	Test result range (%)
100	95.0–100.0
90	85.0–94.9
80	75.0–84.9
70	65.0–74.9
60	55.0–64.9
50	45.0–54.9
40	35.0–44.9
30	25.0–34.9
20	17.5–24.9
15	12.5–17.5
10	7.5–12.4

Source: EN 12934 (1999), Table 2, p 8 – see Section 2.6.2

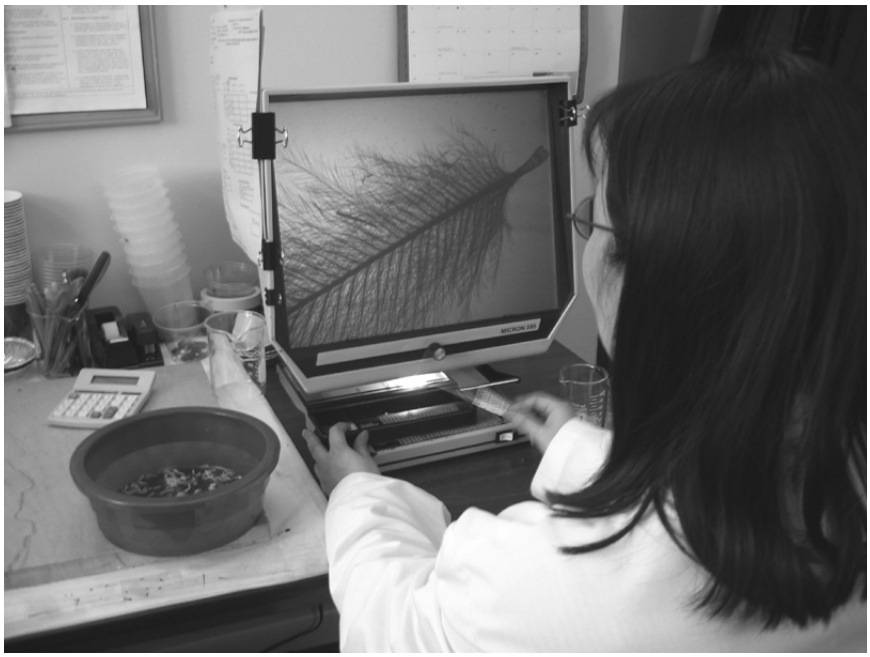
Table 2.6 Goose–duck percentages for labeling

Composition (%)		Denomination
70.0–89.9	goose	
10.0–29.9	duck	goose
50.0–69.9	goose	
30.0–49.9	duck	goose/duck
30.0–49.9	goose	
50.0–69.9	duck	duck/goose
10.0–29.9	goose	
70.0–89.9	duck	duck
0–9.9	goose	
90.0–100	duck	pure duck

Source: EN 12934 (1999), Table 3, p 9 – see Section 2.6.2

still widely performed. Fortunately, nature has equipped the birds' plumage with certain distinctive marks. Best known are the nodes on the down or feather barbules.

IDFB Test Regulation Part 12, Determination of Feather and Down Specie, requires the visual evaluation of all feathers in a 1 g feather sample and all down in a 0.2 g down sample. Several years of research show that testing all pieces at a specific weight is more accurate than testing a certain number of pieces, as was formerly the common practice. This IDFB testing regulation describes the nodes ('fingerprints') of goose, duck, and land fowl feathers and down as follows (see Figs 2.10, 2.11 and 2.12).



2.9 Species identification (*source*: Peter Lieber).

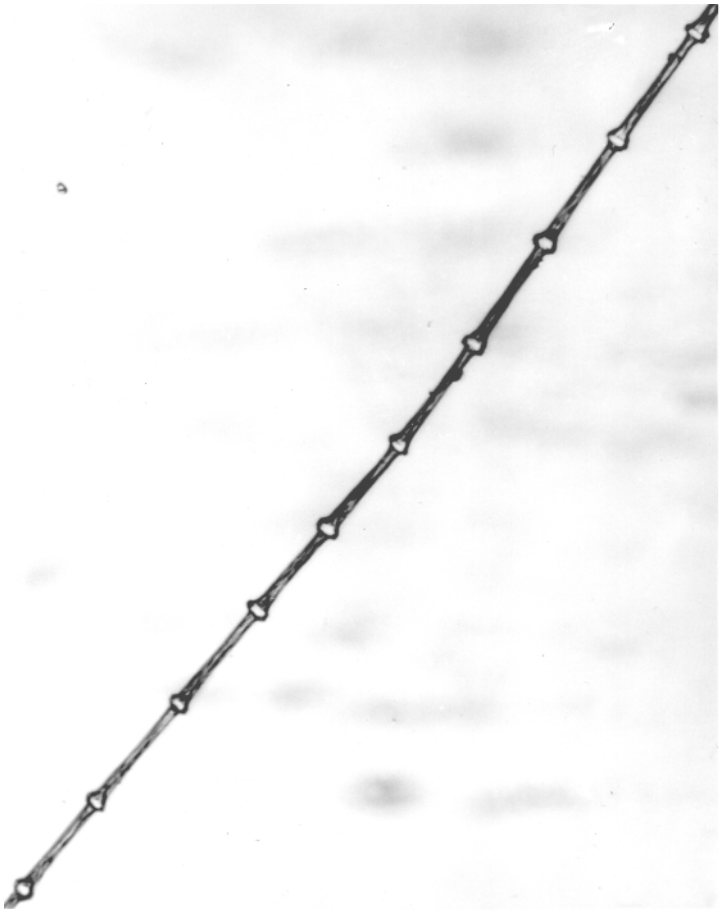
2.4.3 Volume measurement

Filling power

One important feature of feathers, and in particular of down, is their capability to trap air and keep the bird from loosing heat by convection or conduction with a minimum of structural material – a minimum of weight per volume unit, lower than any synthetic product. However, if this were not combined with high resilience and a unique structural 'memory effect' (the sum being what is called filling power or fillpower), feathers and down would be worthless to humans. Several methods are in use; the following description is based on IDFB Testing Regulations Part 10 (see Fig. 2.13):

To measure filling power, 30 g of the down–feather specimen are loosely filled into a cylinder (other test standards use 20 g or one US-ounce) and, according to IDFB Regulations Part 10**,** loaded with a plunger at a specific pressure of 0.149 g cm^{-2} (slightly higher according to the Japanese Industrial Standards, JIS norm). The filling power of down and feathers is usually first measured immediately after the washing and sorting process, and this value is used as one of the quality indicators or claims.

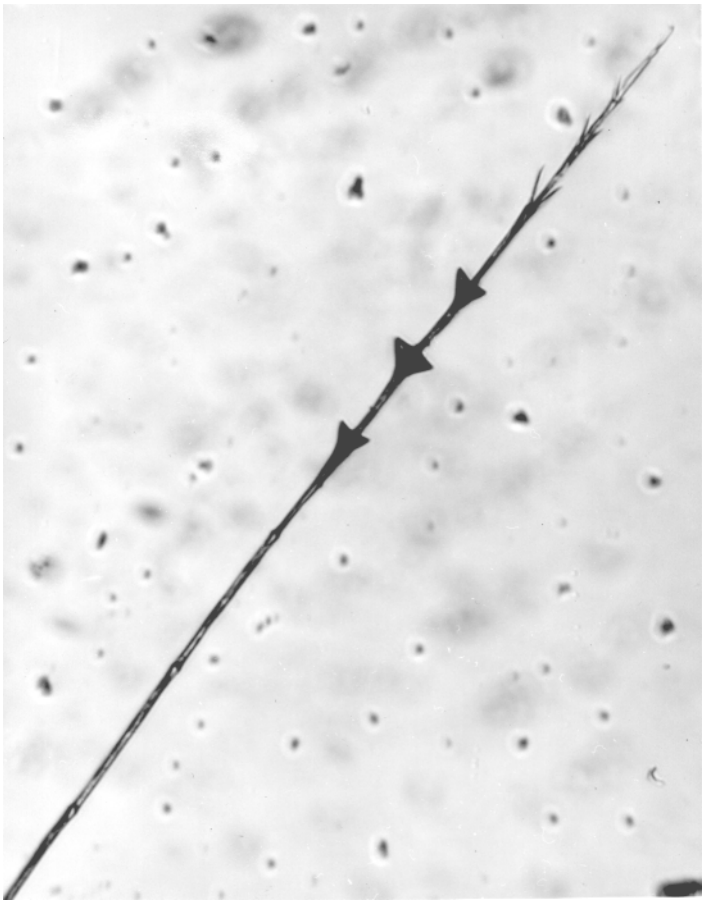
Filling power (in EN 12130, 1998, also called 'massic volume') is expressed in various ways:



2.10 Goose plumage has small nodes which generally begin in the middle area of the barbule. The distance between nodes of a goose feather or down is two times or more the distance between the nodes of a duck (*source*: IDFL).

- in millimeters filling height in a standardized measuring cylinder
- in volume per weight unit, in $cm^3\ g^{-1}$ or $l\ kg^{-1}$ (according to EN 12130, 1998)
- in volume per weight unit, in cubic inches per ounce, cu in/oz (US standards)
- in weight per volume, in $g\ cm^{-3}$ (apparent density)
- in centimeters filling height (according to Japanese standards).

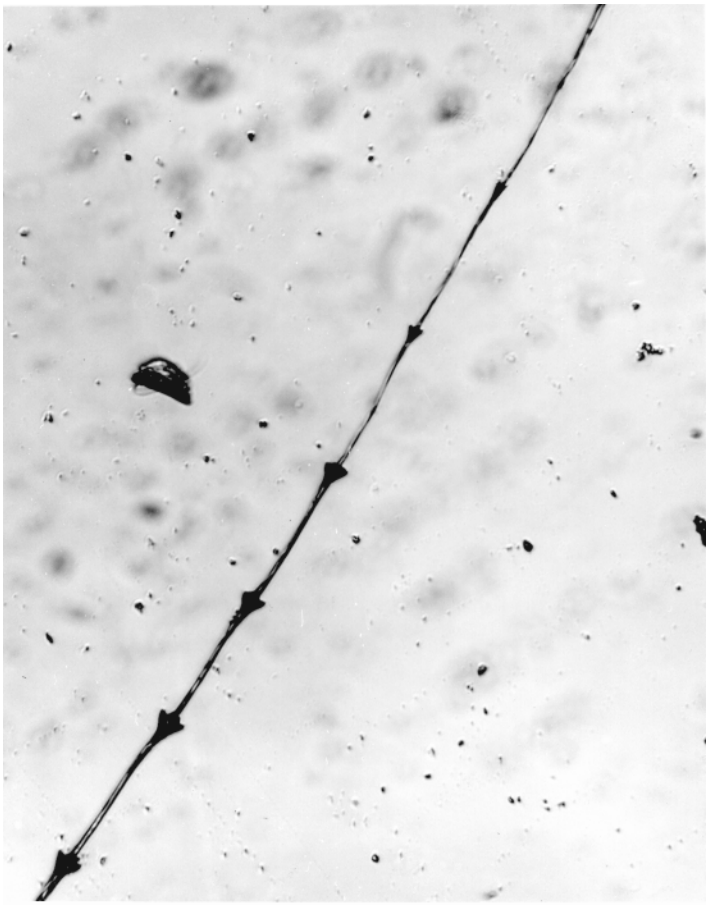
Down and feathers are often vigorously compressed either as processed bulk material in the form of bales or in finished products (e.g. sleeping bags or garments). In this compressed form, they can be transported and stored over longer periods of time. Compression temporarily lowers the filling power of down and feathers.



2.11 Duck plumage has between one and six (often three) nodes near the tip of the barbule. These nodes are relatively large. The distance between the nodes of a duck feather is very short. Prongs are often found beyond the most distant duck node (but are not used in species identification) (*source*: IDFL).

However, in most cases filling power is restored during use and laundering of the product.

Manufacturers, retail buyers and end-use customers would like to be able to verify the filling power quality of the product. This is difficult to do without actually using the product, so product use is often replicated in a laboratory setting. The testing of physical behavior of a natural organic product often makes little sense without taking chemical characteristics into consideration. This is particularly demonstrated in the case of down and feathers owing to their interaction with water molecules. It is this interaction that helps the birds'



2.12 Land fowl (chicken) plumage has a series of evenly spaced slight nodes or swellings which give the barbule the appearance of bamboo. The protrusions or nodes of land fowl extend nearly the entire distance of the barbule (*source*: IDFL).

plumage activate its structural memory and revive it to its filling power potential.

As in any chemical reaction, the absorption or desorption of water is reversible and is connected to a reaction constant, incorporating these physicochemical rules:

- Concentration: obviously, down absorbs water in its liquid form (i.e. the down is submerged in the water) faster than from water present in the atmosphere as humidity. Likewise, it absorbs more in moist air (e.g. steam) than in dry air.
- Time: every chemical reaction, even the most vigorous explosion, takes time each reaction has a specific speed, based on its individual reaction constant.

2.13 Filling power cylinders. Manual (left) and automated (right) (*source*: Peter Lieber).

- Temperature: a chemical reaction generally doubles its speed with every 10 °C rise in temperature.
- Motion: down and feather barbules may be physically restricted from regaining their original geometrical form. Shaking will loosen these restrictions.
- Conditioning: down and feathers require conditioning. Usually, they are box conditioned in screened boxes which are placed in a standard climate room (see Fig. 2.14). At least 72 h of adjusting must be allowed for loose down specimens. Elevated temperatures help speed up the revival. Knowing this, manufacturers and testers have simulated what is done in the cleaning process through tumble drying.

A further developmental step in conditioning is the simulation of body perspiration (which is known to help sleeping bags or comforters to 'rise') by adding a wet towel to the tumbler. Reconditioning the down and feathers to their original volume quality level, which is measured immediately after the original washing and drying, may require 'steaming', that is, direct steam injection into the conditioning box, then allowing the sample to dry off by means of a hair dryer and finally allowing it to adjust to standard climate. Looking at the graph (see Fig. 2.15), it becomes obvious that about 80% of the original filling power (here in

2.14 Conditioning box (*source*: Peter Lieber).

cu in/oz) may be reached in a short time. But to regain the final 20%, the down needs either time or a temperature/moisture boost.

Warmth–insulation

Down and feathers are designed to keep the bird warm. Likewise, we use them to keep ourselves warm and comfortable. It follows that manufacturers and quality control organizations are constantly searching for a meaningful comfort factor. TOG values (describing the thermal insulation properties of textiles as 1 TOG =

0.1 m^2 K W^{-1}) are well known in Great Britain and are used for bed covers as well as for down clothing. The sleeping bag industry has also developed regulations that specify the comfort range of environment temperatures. Other branches of the down and feather industry will soon follow with similar specifications or classifications. In the end, these comfort factors all relate to filling power.

Down–feather ratio

It is obvious from Figs 2.1 and 2.2 that down provides a better insulation per unit of weight than do the feathers. It is therefore useful to include with this description of quality control methods for feathers and down, a graph which shows the filling power in approximate relation to the down/feather ratio (see Fig. 2.16).

2.5 Finished product quality

Textile fabric testing is vital to the quality control of down and feather products. Fabric that can confine down and feathers within predetermined volumes and shapes also prevents dust or mites from contaminating the down filling. Consequently, some basic testing of textiles is necessary for good quality control.

Figure 2.16 does not take into consideration the absolute top range of filling power qualities of around 900 cu in/oz. It does demonstrate that the filling power value drops quickly with even a small addition of feathers. But the resilience-force of feathers is usually higher, making them (or admixtures to down) well suited for pillows.

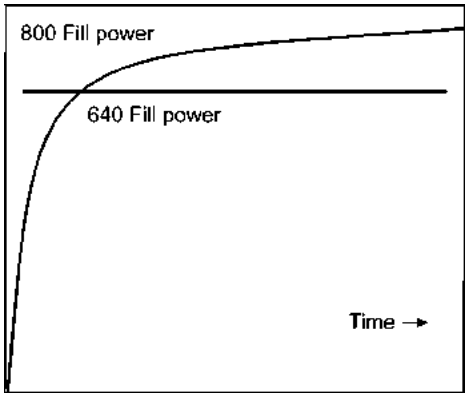
Figure 2.16 raises two questions:

Q1: Why is filling power represented in cu in/oz and not in metric units, cm^{-3} g^{-1} or g cm^{-3}, or in millimeters filling height, the actual reading the instrument takes before converting it into filling power values based on the filling weight?

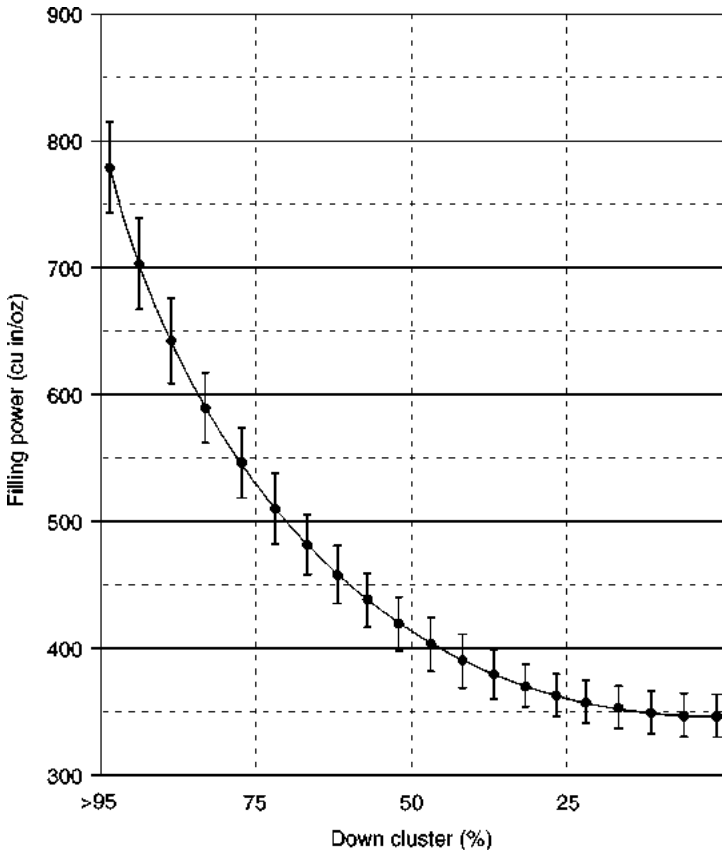
A1: As an example, 800 cu in/oz, if converted to the metric unit (at an identical specific load of 0.149 g cm^{-2}), equals 436.8 l kg^{-1}, and is expressed in this form according to EN 12130 (1998). Psychologically, the larger cubic inch measurement is automatically identified with better quality. In addition, the easy to read, comma-free three digit cubic inch figure (in most cases displayed only in 10 or even 50 unit steps, which is easier yet to read and to conceive) is historically older. It is most likely to remain in friendly co-existence with metric units.

Q2: Does 900 filling power (i.e. 900 cu in/oz) really exist, as sometimes claimed?

A2: Yes, but only in very rare cases and the supply of such down is very small. Since market demand may be much higher than the available down, buyers should be cautious before accepting a 900 filling power claim.



2.15 Filling power increases with conditioning time. (*source*: IDFL).



2.16 Filling power plotted against down cluster (content 0–95%) (*source*: IDFL).

2.5.1 Physical downproofness

Downproofness describes the ability of fabric to keep down and feathers enclosed. There are several test methods which simulate the conditions of actual product use in a time-lapse manner.

US method

A small pillow made of the fabric to be used in the finished product is filled with down and feathers and tumbled in a chamber with rubber stoppers. Both chamber and pillow are examined for leakage counts and rated from 1 (not downproof) to 5 (excellent downproofness) on a comparative basis.

EN method

EN methods EN 12132-1 (1998) and 12132-2 (1998) describe methods of determining downproofness. Sample cushions are filled with specified down and feather mixtures, then tested by either a rubbing motion (EN 12132-1) or by impact (EN 12132-2), simulating the shaking and pounding that occurs in the normal use of bedding or clothing. The samples are then inspected for feathers penetrating the fabric and are rated (see Table 2.7).

2.5.2 Air permeability

Determining air permeability of a fabric is another standard test used in the quality analysis of textiles. Measuring air flow through a specific area of the fabric at standardized differential pressure is a good indicator of downproofness. In general, the lower the air permeability of the fabric tested, the less likely it is that feathers and down will penetrate it. However, these test results are not necessarily conclusive. Some fabrics may fail the air permeability test but pass the physical downproof test and vice versa.

2.5.3 Thread count

Thread count claims are commonly printed on down product labels. The number

Table 2.7 Down proof tests according to EN 12132 (1998)

Impact test (EN 12132-1)	Quality rating	Rubbing test (EN 12132-2)
0 to 5 penetrations	Good	0 to 10 penetrations
6 to 15 penetrations	Acceptable	11 to 20 penetrations
More than 15 penetrations	Not acceptable	More than 20 penetrations

of threads used in weaving is counted by the square inch (or square centimeter). For example, a thread count of 180 may mean a weave of 100 vertical threads (the warp) and 80 horizontal threads (the weft) per square inch. In metric units, it would be designated a 40–32 weave (40 warp threads and 32 weave threads per square centimeter). The thickness of the thread is also a factor in this equation. The finer the thread, the more there will be in one square unit, producing a higher thread count. Normally, the higher the thread count, the better the downproofness. However, yarn size and weaving techniques make some high thread count material unacceptable for down products whereas some low thread count fabrics are acceptable. Before using new fabrics, a careful evaluation of downproofness should be completed.

What is considered a 'high thread count' in down and feather products? A thread count of between 200 and 400 (per square inch) is sufficient, but thread count can go as high as 800 or 1000 threads. (It is important to note that for down and feather quality control purposes, two-ply threads are counted as one thread).

2.5.4 Product labels

As mentioned at the beginning of this chapter, uniform testing procedures provide the methods used to control the quality of down and feather products. By using standardized terms to reflect the quality of down and feather products on the product label, the consumer can be confident that a pillow labeled '100% goose down,' is what it is claimed to be. In Europe, for instance, the European Norm (EN) is standard. In reference to product labeling, the following norms are applied:

EN 12131 (1998) 'Determination of the quantitative composition of feather and down,' describes how the composition of down samples is to be analyzed, calculated and reported.

EN 1885 (1998) 'Terms and definitions,' supports EN 12131 by identifying and defining the components that are to be separated and includes pictures.

EN 12934 (1999) 'Composition labeling of processed feathers and down for use as the sole filling material,' establishes provisions for how the composition of the plumage for use as fillings, and of the fowl species from which such components are derived, are to be represented on the product label (see also Figs 2.9 and 2.10 in Section 2.4.2).

Other norms are created to present these values on labels in a concise form. Despite such attempts to standardize labeling, the problem of standardizing norms world-wide is on-going.

Although the CEN (European Committee for Standardization) is working to solve this problem within Europe, these standardized procedures have not necessarily been adopted by manufacturers outside Europe, whereas the quality control testing procedures are more widely accepted. However, tolerances used to classify feather and down products can vary widely. As an example, EN 12934 (1999)

clearly requires down content ranges to be identified in numbers. According to Table 2.5 in Section 2.4.1, a product labeled '90% down' must test between 85.0% and 94.9% down clusters. Yet in other areas of the world, a product containing a minimum of 75% down may still be labeled as 'down.' The problem is summed up well in EN 12934: 'Fillings of feather and down have not been covered by national standards dealing with denomination and composition in all CEN countries. Where standards exist they often differ from each other.'

2.6 References

2.6.1 Normative references

Table 2.8 shows which IDFB Regulations and European Norms should be followed for each type of test.

2.6.2 Sources for citations

CEN (European Committee for Standardization/Comité Européen de Normalisation), rue de Stassart, 36 B-1050 Brussels.

EN 1161: 1996 E, 'Feather and down – Test methods – Determination of moisture content'. CEN: Brussels, 1996.

EN 1162: 1996 E, 'Feather and down – Test methods – Determination of the oxygen index number'. CEN: Brussels, 1996.

EN 1163: 1996 E, 'Feather and down – Test methods – Determination of the oil and fat content'. CEN: Brussels, 1996.

EN 1164: 1998 E, 'Feather and down –Test methods –Determination of the turbidity of an aqueous extract'. CEN: Brussels, 1998.

Table 2.8 Normative references

Test	IDFB-Regulation	European Norm
Feather and down–sampling in view of tests	Part 2	EN 1883
Moisture content	Part 5	EN 1161
Acidity (pH)	Part 6	N/A
Oxygen number	Part 7	EN 1162
Turbidity (nephelometric)	Part 11-A	N/A
Turbidity (tube visibility)	Part 11B	EN 1164
Oil and fat content	Part 4	EN 1163
Content analysis	Part 3	EN 12131
Quality classes	N/A	EN 12934
Terms and definitions	N/A	EN 1885
Filling power (volume measurement)	Part 10	N/A
Comfort range (sleeping bags)	N/A	EN 13537
Downproofness	N/A	EN 12132-1/2
Species identification	Part 12	N/A

EN 1883: 1998E, 'Feather and down – Sampling in view of tests: Table A.1 and Table A.2. CEN: Brussels, 1998.
EN 1885: 1998/prA1:2003, 'Feather and down – Terms and definitions – Amendment 1'. CEN: Brussels, 1998.
EN 12130: 1998 E, 'Feather and down – Test methods – Determination of the filling power (massic volume)'. CEN: Brussels, 1998.
EN 12131: 1998 E, 'Feather and down – Test methods – Determination of the quantitative composition of feather and down (manual method)'. CEN: Brussels, 1998.
EN 12132-1: 1998 E, 'Feather and down – Methods of testing the down proof properties of fabrics – Part 1: Rubbing test'. CEN: Brussels, 1998.
EN 12132-2: 1998/prA1: 2003 E, 'Feather and down – Methods of testing the down proof properties of fabrics – Part 2: Impact test'. CEN: Brussels, 2003.
EN 12934: 1999 E, 'Feather and down – Composition labeling of processed feathers and down for use as sole filling material'. CEN: Brussels, 1999.

2.6.3 IDFB Testing Regulations

The IDFB Testing Regulations cited below were published by IDFB, Aschaffenburg, Germany. The IDFB moved to Dornbirn in January 2005 (see below). These documents are not paginated, nor are they available on-line.

IDFB (International Down and Feather Bureau)
Current address: Marktplatz 9, A-6850 Dornbirn, Austria
Contact: Mr Siegfried Böhler, Secretary
Tel: +43 5572 382223
Fax: +43 5572 31393
Email: idfb@idfb.org

IDFB Testing Regulations:

1. Part 1 Conditioning
2. Part 2 Sampling
3. Part 3 Determination of the composition (content analysis)
4. Part 4 Determination of the oil and fat content
5. Part 5 Determination of the moisture content
6. Part 6 Determination of the acidity (pH factor)
7. Part 7 Determination of oxygen number
8. Part 10 Determination of filling power (volume measurement)
9. Part 11-A Determination of turbidity (with automated NTU meter)
10. Part 11-B Determination of turbidity (with glass turbidity tube)
11. Part 12 Determination of feather and down species

2.6.4 Other sources

For the 'LaMotte 2020 Turbidity Meter' in Fig. 2.7, see www.lamotte.com/pages/common/pdf/manuals/1799.pdf

Although the above is the internet page title, if you type it in, the page is apparently unavailable. To find it, go to www.lamotte.com, click on 'product manuals' and then click on '2020 Turbidity Meter.' This takes you to the 2020 Turbidity Meter Instruction Manual. The image shown in Fig. 2.7 is on page 9 and the specification for 'Nephelometric turbidity, calibrated in NTU' is given on page 5.