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chapter

Introduction to Food Analysis

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1.1 INTRODUCTION

Investigations in food science and technology, whether by the food industry, governmental agencies, or universities, often require determination of food composition and characteristics. Trends and demands of consumers, national and international regulations, and realities of the food industry challenge food scientists as they work to monitor food composition and to ensure the quality and safety of the food supply. As summarized by McGorrin [1] in a review of food analysis history, "the growth and infrastructure of the model food distribution system heavily relies on food analysis (beyond simple characterization) as a tool for new product development, quality control, regulatory enforcement, and problem solving." All food products require analysis of various characteristics (i.e., chemical composition, microbial content, physical properties, sensory properties) as part of a **quality management** program, from raw ingredients, through processing, to the final product. Of course, food analysis is also used extensively for research on foods and food ingredients. The nature of the sample and the specific reason for the analysis commonly dictate the choice of analytical methods. **Speed**, **precision**, **accuracy**, **robustness**, **specificity**, and **sensitivity** are often key factors in this choice. **Validation** of the method for the specific **food matrix** being analyzed is necessary to ensure usefulness of the method.

Making an appropriate choice of analytical technique for a specific application requires a good knowledge of the various techniques (Fig. 1.1). For example, your choice of method to determine the salt content of potato chips would be different if it is for nutrition labeling compared to quality control. The success of any analytical method relies on the proper selection and preparation of the food sample, carefully performing the analysis, and doing the appropriate calculations and interpretation of the data. Methods of analysis developed and endorsed by several nonprofit scientific organizations allow for standardized comparisons of results between different laboratories and for evaluation of less standard procedures. Such **official methods** are critical in the analysis of foods, to ensure that they meet the legal requirements established by governmental agencies. **Government regulations** and **international standards** most relevant to the analysis of foods are mentioned here but covered in more detail in Chap. 2, and nutrition labeling regulations in the United States are covered in Chap. 3.

1.2 REASONS FOR ANALYZING FOODS AND TYPES OF SAMPLES ANALYZED

1.2.1 Overview

Consumer trends and demands, national and international regulations, and the food industry's need to manage product quality dictate the need for analysis of food ingredients and products (Table 1.1) and explain the types of samples analyzed.

1.2.2 Consumer Trends and Demands

Consumers have many choices regarding their food supply, so they can be very selective about the products they purchase. They demand a wide variety of products that are safe, nutritious, and of high quality and offer a good value. Consumer demand has driven significant growth in products making claims, many of which bring an increased need for food
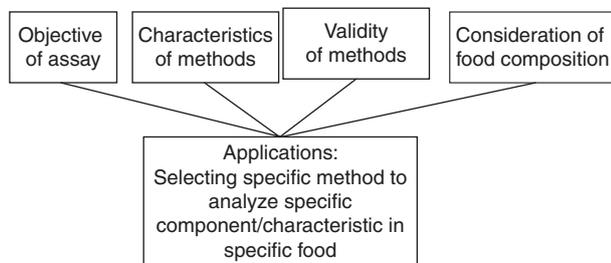
Method selection in food analysis

1.1 table   Reasons for analyzing foods

1. Food safety
2. Government regulations
   (a) Nutrition labeling
   (b) Standards – mandatory and voluntary
   (c) Food inspection and grading
   (d) Authenticity
3. Quality control
4. Research and development

analysis. For example, the consumer-driven, gluten-free claim has led to increased raw material/ingredient testing and finished product testing, required to comply in the United States with the definition of this claim established by the Food and Drug Administration. Many consumers are interested in the relationship between diet and health, including functional foods that may provide health benefits beyond basic nutrition. The use of social media by consumers has changed expectations and raised questions about the food supply. Such trends and demands by consumers increase the need for food analysis and present some unique challenges regarding analytical techniques [2, 3].
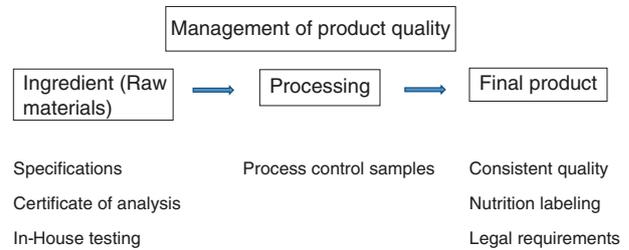
1.2.3 Government Regulations and International Standards and Policies

For food companies to market safe, high-quality foods effectively in a national and global marketplace, they must pay increasing attention to government regulations and guidelines, and to the policies and standards of international organizations. Food scientists must be aware of these regulations, guidelines, and policies related to food safety and quality, and must know their implications for food analysis. Government regulations related to the composition of foods include nutrition labeling, product claims, standards, inspection and grading, and authenticity. This latter issue of authenticity is a challenge for the food industry, given the constant threat of economic adulteration of food products and ingredients. Detecting untargeted compounds in foods and determining their identity are challenges that require advanced analytical techniques that are powerful, sensitive, and fast [2]. The industry is also challenged and forced to "chase zeros" when laws are written stating zero as the level of certain compounds allowed. US government regulations relevant to food analysis are covered in Chaps. 2 and 3. Also covered in Chap. 2 are organizations active in developing international standards and safety practices relevant to food analysis.

1.2.4 Food Industry Management of Product Quality

1.2.4.1 *Raw Ingredients to Final Product*

To compete in the marketplace, food companies must produce foods that meet consumer demands, comply with government regulations, and meet quality standards of the company. Whether it is new or existing food products or ingredients, the key concern for the food industry is safety of the food, but quality management goes well beyond safety. The management of product quality by the food industry is of increasing importance, beginning with the raw ingredients and extending to the final product eaten by the consumer. Analytical methods must be applied across the entire

Management of product quality		
Ingredient (Raw materials) →	Processing →	Final product
Specifications	Process control samples	Consistent quality
Certificate of analysis		Nutrition labeling
In-House testing		Legal requirements

1.2 **figure** Food industry management of quality

food supply chain to achieve the desired final product quality (Fig. 1.2). It is obvious that the food processor must understand the entire supply chain to successfully manage product quality.

In some cases, the cost of goods is linked directly to the composition as determined by analytical tests. For example, in the dairy field, butterfat content of bulk tank raw milk determines how much money the milk producer is paid for the milk. For flour, the protein content can determine the price and food application for the flour. These examples point to the importance for accurate results from analytical testing.

Traditional quality control and quality assurance concepts are only a portion of a comprehensive quality management system. Food industry employees responsible for quality management work together in teams with other individuals in the company responsible for product development, production, engineering, maintenance, purchasing, marketing, and regulatory and consumer affairs.

Analytical information must be obtained, assessed, and integrated with other relevant information about the food system to address quality-related problems. Making appropriate decisions depends on having knowledge of the analytical methods and equipment utilized to obtain the data related to the quality characteristics. To design experiments in product and process development, and to assess results, one must know the operating principles and capabilities of the analytical methods. Upon completion of these experiments, one must critically evaluate the analytical data collected to determine whether product reformulation is needed or what parts of the process need to be modified for future tests. The situation is similar in the research laboratory, where knowledge of analytical techniques is necessary to design experiments, and the evaluation of data obtained determines the next set of experiments to be conducted.

1.2.4.2 *Types of Samples Analyzed*

Chemical and physical analysis of foods is an important part of a quality assurance program in food processing, from ingredients and raw materials, through

table 1.2	Types of samples analyzed in a quality assurance program for food products

Sample type	Critical questions
Raw materials	Do they meet your specifications? Do they meet required legal specifications? Are they safe and authentic? Will a processing parameter have to be modified because of any change in the composition of raw materials? Are the quality and composition the same as for previous deliveries? How does the material from a potential new supplier compare to that from the current supplier?
Process samples control	Did a specific processing step result in a product of acceptable composition or characteristics? Does a further processing step need to be modified to obtain a final product of acceptable quality?
Finished product	Does it meet the legal requirements? What is the nutritive value, so that label information can be developed? Or is the nutritive value as specified on an existing label? Does it meet product claim requirements (e.g., "low fat," "gluten free")? Will it be acceptable to the consumer? Will it have the appropriate shelf life? If unacceptable and cannot be salvaged, how do you handle it? (trash? rework? seconds?)
Competitor's sample	What are its composition and characteristics? How can we use this information to develop new products?
Complaint sample	How do the composition and characteristics of a complaint sample submitted by a customer differ from a sample with no problems?

Adapted and updated from [6, 7]

processing, to the finished products [4, 5]. Chemical and physical analysis also is important in formulating and developing new products, and evaluating new processes for making food products, and in identifying the source of problems with unacceptable products (Table 1.2). Competitor's samples are increasingly relevant, e.g., store brands versus national brands. For each type of sample listed in Table 1.2 to be analyzed, it may be necessary to determine either just one or many components. The nature of the sample and the way in which the information obtained will be used may dictate the specific method of analysis. For example, process

control samples are usually analyzed by rapid methods, whereas nutritive value information for nutrition labeling generally requires the use of more time-consuming methods of analysis endorsed by scientific organizations. Critical questions, including those listed in Table 1.2, can be answered by analyzing various types of samples in a food processing system.

1.2.4.3 Increasing Dependence on Suppliers

Downsizing in response to increasing competition in the food industry often has pushed the responsibility for ingredient quality to the suppliers. Companies increasingly rely on others to supply high-quality and safe raw ingredients and packaging materials. Many companies have **select suppliers**, on whom they rely to perform the analytical tests to ensure compliance with detailed specifications for ingredients/raw materials. These specifications, and the associated tests, target various chemical, physical, and microbiological properties, as appropriate based on the nature of the ingredient. Such specifications for raw materials/ingredients come in various forms within the food industry, with three commonly used forms described below:

1. **Technical/Product Data Sheet**: supplier uses when salesperson is selling ingredients; gives maximum, minimum, and/or range of values, as appropriate, and methods of analysis
2. **Specifications**: internal company document that defines company (processor) requirements (minimum, maximum, and/or target values) and links these to specific methods of analysis; much of the data come from the Technical/Product Data Sheet; give the requirements for the Certificate of Analysis
3. **Certificate of Analysis** (COA): includes results of analytical tests related to predetermined specifications for specific shipment sent to customer; gives actual values and methods of analysis

As an example of specification information, Table 1.3 gives such information for the ingredient semolina, which is coarse-ground flour from durum wheat (high protein), used especially to make pasta, but also for other cereal grain-based products. For the purposes of this table, specific numbers are not given, as they would be in the actual documents for an ingredient.

Ingredient specifications and COAs are important for making specific food products. For example, with the wrong starch (i.e., with wrong specifications), a specific food product may not process correctly nor have the desired finished product quality attributes. Also, for example, if the COA indicates the granulation size of a specific lot of rolled oats is "out of spec," the finished granola bars may not have the desired properties, which can result in increased

1.3 table Example of information included in technical data sheet, specification, and certificate of analysis (COA) for semolina

Properties	Technical/product data sheet	Specification	Certificate of analysis
Chemical			
Moisture	Max value; AACCI	Max value; AOAC/AACCI	Actual value; AACCI
Protein	Min value; AACCI	Min value; AOAC/AACCI	Actual value; AACCI
Ash	Max value; AACCI	Max value; AOAC/AACCI	Actual value; AACCI
Falling number value	Target value, +/−; AACCI		
Enrichment			
Niacin		Max, min, target value; AOAC/AACCI	
Thiamine mononitrate		Max, min, target value; AOAC/AACCI	
Riboflavin		Max, min, target value; AOAC/AACCI	
Ferrous sulfate		Max, min, target value; AOAC/AACCI	
Folic acid		Max, min, target value; AOAC/AACCI	
Physical			
Bran specks	Max value; Internal	Max value; AOAC/AACCI	Actual value; Internal
Black specks	Max value; Internal	Max value; Internal	Actual value; Internal
Color L, a, b		Max L value, Min b value; AOAC/AACCI	Actual values (Hunter)
Color (linear E)			Actual value (calculated from Hunter LAB)
Extraneous matter	Complies with FDA regulations; AACCI		
Insect fragments		Max value; AOAC/AACCI	
Rodent hair		Max value; AOAC/AACCI	
Granulation	Value, +/−. % over #40, 60, 80, 100 sieve. % thru #100 sieve; Rotap	Min, max, target value. % over #40, 60, 80, 100 sieve. % thru #100 sieve; AOAC/AACCI	Actual value. % over #40, 60, 80, 100 sieve. % thru #100 sieve; Rotap
Microbiological			
Standard plate count; Total plate count	Product is considered not ready to eat and requires further processing, so no microbiological guarantees provided	Target value; FDA BAM	
Yeast		Target value; FDA BAM	
Mold		Target value; FDA BAM	
Vomitoxin	Complies with FDA advisory max level	Max value; FDA BAM	
Shelf life	Number of days at recommended storage conditions		

Actual, minimum, maximum, and/or target value; source/type of method

Source of method: *AOAC*, AOAC International; *AACCI*, AACC International; *FDA BAM*, FDA's Bacteriological Analytical Manual; *Internal*, company method; *Hunter*, Hunter colorimeter; *Rotap*, machine to measure granulation

consumer complaints. Setting the specifications for raw ingredients is commonly the responsibility of product developers, but it is often the production and quality control staff at the processing facility that must deal with challenges that arise when there are problems associated with ingredient specifications. Companies must have in place a means to maintain control of the COAs and react to them. With careful control over the quality of raw ingredients/materials, less testing is required during processing and on the final product.

1.2.4.4 Properties Analyzed

Analyzing foods for the reasons described in Table 1.1 to manage product quality commonly involves testing the following: chemical composition/characteristics,

1.4
table Quality management tests for dried pasta

Component/property being measured	Name of test
Quality test done in-house on semolina	
Moisture content	Rapid moisture analyzer
Color (L*, a*, b* determined to calculate Linear E value)	Colorimeter
Quality tests done in-process	
Moisture	Rapid moisture analyzer
Dimensions (after extrusion)	Micrometer and tape measurements by trained personnel
Metal detection	In-line metal detection (ferrous, nonferrous, stainless steel)
Package weight	In-line check with weight scale
Quality tests done on final product	
Moisture	Rapid moisture analyzer
Color L*, a*, b* determined to calculate Linear E value)	Colorimeter
Dimensions (diameter and shape)	Micrometer and tape measurements by trained personnel
Cooking quality	Sensory test by trained personnel (descriptive test; biting of samples)
Label	Visual inspection (with probability sampling methods)

physical properties, sensory properties, and microbial quality. Table 1.4 shows the quality management tests typically done on dried pasta. Each test indicated would be done at some specified frequency and by a specified method. Note the relationship between the COA information for semolina in Table 1.3 and the quality tests done on in-house on semolina by the processor, as reported in Table 1.4. While the COA generally requires considerable testing, done by official methods, routine in-house testing of ingredients is typically limited in scope and often uses rapid methods. The nature of testing required is largely determined by the nature of the food ingredient/product, but not surprisingly sensory properties (including taste, smell, appearance) are commonly tested across all foods and ingredients. This book focuses only on methods of analysis for testing chemical composition/characteristics and physical properties, and not on sensory properties or microbial quality.

1.3   STEPS IN ANALYSIS

1.3.1   Select and Prepare Sample

In analyzing food samples of the types described previously, all results depend on obtaining a representative sample and converting the sample to a form that can be analyzed. Neither of these is as easy as it sounds! **Sampling** and **sample preparation** are covered in detail in Chap. 5.

Sampling is the initial point for sample identification. Analytical laboratories must keep track of incoming samples and be able to store the analytical data from the analyses. This analytical information often is stored on a **laboratory information management system** (LIMS), which is a computer database program. Especially beneficial to commercial analytical laboratories, this system can capture all data for any specific sample and make it accessible to customers so they can import, review, and analyze as they wish. Customers have real-time access to these data, which also enables them to review testing status. There is no need for hand typing data, which saves times and decreases chances for error, and the system settings can ensure standardized data entry for consistency and compliance to analytical requirements.

1.3.2   Perform the Assay

Performing the assay is unique for each component or characteristic to be analyzed and may be unique to a specific type of food product. Single chapters in this book address sampling and sample preparation (Chap. 5) and data handling (Chap. 4), while the remainder of the book addresses the step of actually performing the assay. Chapters 6, 7, 8, 9, 10, and 11 cover spectroscopy and spectrometry, and Chaps. 12, 13, and 14 cover chromatography. These major general topics are covered before chapters that cover specific methods for chemical composition and characterization, since many analytical methods utilize spectroscopy and chromatography techniques. The descriptions of the various specific procedures are meant to be overviews of the methods. For guidance in actually performing the assays, details regarding chemicals, reagents, apparatus, and step-by-step instructions are found in the books and articles referenced in each chapter. Numerous chapters in this book, and other books devoted to food analysis [8–12], make the point that for

food analysis, we increasingly rely on expensive equipment, some of which requires considerable expertise. Also, it should be noted that numerous analytical methods utilize automated instrumentation, including autosamplers and robotics to speed the analyses.

1.3.3 Calculate and Interpret the Results

To make decisions and take action based on the results obtained from performing the assay that determined the composition or characteristics of a food product, one must make the appropriate calculations to interpret the data correctly. **Data handling**, covered in Chap. 4, includes important statistical principles.

1.4 METHOD SELECTION

1.4.1 Objective of the Assay

Selection of a method depends largely on the objective of the measurement (Fig. 1.1). For example, methods used for rapid online processing measurements may be less accurate than official methods (see Sect. 1.5) used for nutritional labeling purposes. Methods referred to as reference, definitive, official, or primary are most applicable in a well-equipped and staffed analytical lab. Quality control testing of raw ingredients at the processing facility, in-process testing, and final product testing often rely on secondary/rapid quality control methods. This is likely in contrast to primary/official methods used on raw ingredient specification and COAs and the testing for nutrition labeling. Both **primary methods** and **secondary methods** may be necessary and appropriate, with a secondary method calibrated against the primary method. The more rapid secondary or field methods may be more applicable on the manufacturing floor in a food processing facility. For example, refractive index may be used as a rapid, secondary method for sugar analysis (see Chaps. 15 and 19), with results correlated to those of the primary method, high-performance liquid chromatography (HPLC) (see Chaps. 13 and 19). Moisture content data for a product being developed in the pilot plant may be obtained quickly with a rapid moisture analyzer that has been calibrated using a more time-consuming forced air oven method (see Chap. 15). Many companies commonly use unofficial, rapid methods for testing in the processing plants but validate them against official methods. The calibration between these methods is critical.

1.4.2 Characteristics of the Method

Numerous methods often are available to assay food samples for a specific characteristic or component. To select or modify methods used to determine the chemical composition and characteristics of foods, one must be familiar with the principles underlying the procedures and the critical steps. Certain properties of methods and criteria described in Table 1.5 are useful to evaluate the appropriateness of a method in current use or a new method being considered. As highlighted in a review article on food analysis by Cifuentes [2], there is a continual need for the development of methods that can be characterized as being more robust, efficient, sensitive, and cost-effective. Many of the older "wet chemistry" methods have evolved into powerful and commonly used instrumental techniques. This has led to significant increases in accuracy, precision, detection limits, and sample throughput.

1.4.3 Validity of the Method

1.4.3.1 *Overview*

Numerous factors affect the usefulness and validity of the data obtained using a specific analytical method. One must consider certain characteristics of any method, such as specificity, precision, accuracy, and sensitivity (see Table 1.5 and Chap. 4). However, one also must consider how the variability of data from the method for a specific characteristic compares to differences detectable and acceptable to a consumer, and the variability of the specific characteristic inherent in processing of the food. One must consider the nature of the samples collected for the analysis, how representative the samples were of the whole, and the number of samples analyzed (Chap. 5). One must ask whether details of the analytical procedure were followed adequately, such that the results are accurate, repeatable, and comparable to data collected previously. For data to be valid, equipment to conduct the analysis must be standardized and appropriately used, and the performance limitations of the equipment recognized.

1.4.3.2 *Standard Reference Materials*

A major consideration for determining method validity is the analysis of materials used as controls, often referred to as **standard reference materials** or **check samples** [13]. Analyzing check samples concurrently with test samples is an important part of quality control [14]. Standard reference materials (SRMs) can be obtained in the United States from the National Institute of Standards and Technology (NIST) and from US Pharmacopeia (USP), in Europe from the Institute for Reference Materials and Measurements (IRMM), and from other specific organizations for other countries/regions. Besides government-related groups, numerous organizations offer check sample services that provide test samples to evaluate the reliability of a method [13]. For example, **AACC International** (AACCI) (formerly known as the American Association of Cereal Chemists, AACC) has a check sample service in which a subscribing

1.5 table

Criteria for choice of food analysis methods: characteristics of a method

Characteristic	Critical questions
Inherent properties	
Specificity/selectivity	Is the property being measured the same as that claimed to be measured, and is it the only property being measured?
	Are there interferences?
	What steps are being taken to ensure a high degree of specificity?
Precision	What is the precision of the method? Is there within-batch, batch-to-batch, or day-to-day variation?*
	What step in the procedure contributes the greatest variability?
Accuracy	How does the new method compare in accuracy to the old or a standard method?
	What is the percent recovery?
	Is it reproducible between labs?
Applicability of method to laboratory	
Reagents	Can you properly prepare them?
	What equipment is needed? Are they stable? For how long and under what conditions?
Equipment	Is the method very sensitive to slight or moderate changes in the reagents?
	Do you have the appropriate equipment?
	Are personnel competent to operate equipment?
Cost	What is the cost in terms of equipment, reagents, and personnel?
Applicability to food/sample	Destructive or nondestructive?
	Online or off-line?
	Official method/approval?
	Nature of food matrix?
Usefulness	
Time required	How fast is it? How fast does it need to be?
Reliability	How reliable is it from the standpoints of precision and stability?
Need	Does it meet a need or better meet a need?
	Simplicity of operation?
Personnel	
Safety	Is any change in method worth the trouble of the change?
Procedures	Who will do any required calculations?

*In-process samples may not accurately represent finished product; must understand what variation can and should be present

laboratory receives specifically prepared test samples from AACCI. The subscribing laboratory performs the specified analyses on the samples and returns the results to AACCI. The AACCI then provides a statistical evaluation of the analytical results and compares the subscribing laboratory's data with those of other laboratories to inform the subscribing laboratory of its degree of accuracy. The AACCI offers check samples such as flours, semolina, and other cereal-based samples, for analyses such as moisture, ash, protein, vitamins, minerals, sugars, total dietary fiber, and soluble and insoluble dietary fiber. Samples also are available for testing physical properties and for microbiological and sanitation analyses.

The **American Oil Chemists' Society** (AOCS) has a reference sample program that includes oilseeds, oils and fats, marine products, aflatoxins, cholesterol, trace metals, *trans*-fatty acids, and other samples.

Laboratories from many countries participate in the program to check the accuracy of their work, their reagents, and their laboratory apparatus against the statistical norm derived from the group data.

Standard reference materials are important tools to ensure reliable data. However, such materials need not necessarily be obtained from outside organizations. Control samples internal to the laboratory (i.e., **internal reference materials**) can be prepared by carefully selecting an appropriate type of sample, gathering a large quantity of the material, mixing and preparing to ensure homogeneity, packaging the sample in small quantities, storing the samples appropriately, and routinely analyzing the control sample when test samples are analyzed. Whatever the standard reference materials used, these should match closely the matrix of the samples to be analyzed by a specific method. AOAC International has a peer-review

program of matching reference materials with respective official methods of analysis.

1.4.3.3 *ISO Certification*

To help ensure validity of results generated by methods of analysis, commercial, private, and government laboratories are increasingly using **ISO** (International Organization for Standardization) certification (currently 17025) to help assure their customers of the quality of their work (i.e., validity of data) [4, 15]. Certification can be for the entire laboratory and/or just individual assays (i.e., methods and procedures). Once certified, these laboratories are audited for recertification every two years. ISO certification involves review of standard operating procedures, forms, records, work instructions, document control process, and test methods. While ISO certification does not deal with safety or the business, it does address questions about the following: (1) results validity, (2) equipment, (3) training, (4) analysts operating the equipment, (5) reagents and chemicals, and (6) customer communication.

Method certification requires comparison to other laboratories and the use of control samples/standards. For a food company with a corporate laboratory and multiple plant operations, the corporate laboratory does proficiency testing, using check samples (e.g., USP, NIST), and makes control samples that can be tested at both the corporate and plant laboratories for comparison of results.

A benefit of certification can include increased business for the laboratories (i.e., some customers choose ISO-certified laboratories over noncertified laboratories, due to increased confidence in the data). Also, the certification tends to create a mind-set of

continuous improvement within the laboratory. The greatest challenges for laboratories regarding certification are often updating procedures and balancing the need to get results to customers versus the need to check equipment and follow all required procedures. If a company fails an audit, the approach used is root cause analysis and corrective action investigation and implementation.

1.4.4 Consideration of Food Composition

Proximate analysis of foods refers to determining the major components of moisture (Chap. 15), ash (total minerals) (Chap. 16), lipids (Chap. 17), protein (Chap. 18), and carbohydrates (Chap. 19). The performance of many analytical methods is affected by the **food matrix** (i.e., its major chemical components, especially lipid, protein, and carbohydrate). In food analysis, it is usually the food matrix that presents the greatest challenge to the analyst [16]. For example, high-fat or high-sugar foods can cause different types of interferences than do low-fat or low-sugar foods. Digestion procedures and extraction steps necessary for accurate analytical results can be very dependent on the food matrix. The complexity of various food systems often requires having not just one technique available for a specific food component, but rather multiple techniques and procedures, as well as the knowledge about which to apply to a specific food matrix.

A task force of **AOAC International** (formerly known as the Association of Official Analytical Chemists, AOAC) suggested a "triangle scheme" for dividing foods into matrix categories [17–25] (Fig. 1.3). The apexes of the triangle contain food groups that
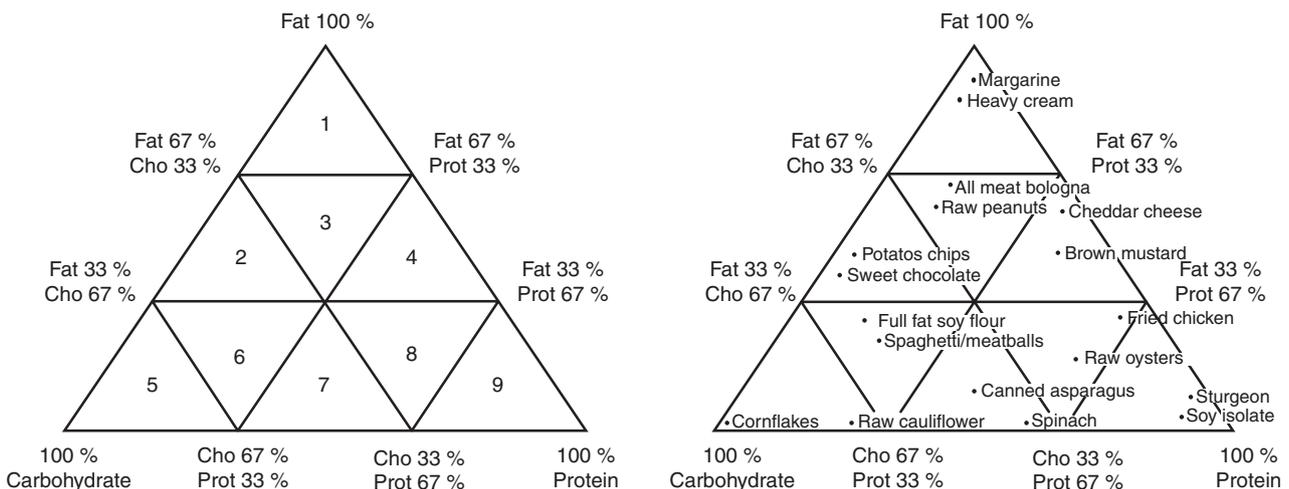
1.3 figure Schematic layout of food matrixes based on protein, fat, and carbohydrate content, excluding moisture and ash (Reprinted with permission from [20], *Inside Laboratory Management*, September 1997, p. 33. Copyright 1997, by AOAC International])

were either 100 % fat, 100 % protein, or 100 % carbohydrate. Foods were rated as "high," "low," or "medium" based on levels of fat, carbohydrate, and proteins, which are the three nutrients expected to have the strongest effect on analytical method performance. This created nine possible combinations of high, medium, and low levels of fat, carbohydrate, and protein. Complex foods were positioned spatially in the triangle according to their content of fat, carbohydrate, and protein, on a normalized basis (i.e., fat, carbohydrate, and protein normalized to total 100 %). General analytical methods ideally would be geared to handle each of the nine combinations, replacing more numerous matrix-dependent methods developed for specific foods. For example, using matrix-dependent methods, one method might be applied to potato chips and chocolates, which are both low-protein, medium-fat, medium-carbohydrate foods, but another might be required for a high-protein, low-fat, high-carbohydrate food such as nonfat dry milk [19]. In contrast, a robust general method could be used for all of the food types.

The AACCI has approved a method studied using the triangle approach [21]. This approach also has been adopted by NIST for categorizing their SRMs [23–25] (see also Sect. 1.4.3.2). These SRMs can be used by laboratories to optimize methods for samples with specific matrices and to confirm that a method can be used reliably for a specific matrix. The food triangle categorization of both samples and reference materials enables analysts to choose the appropriate SRM for method development, for optimization, or for confidence in routine testing. NIST has, as a goal, the development of SRMs and other reference materials in all nine sections of the food matrix triangle [23].

1.5   OFFICIAL METHODS

The choice of method for a specific characteristic or component of a food sample is often made easier by the availability of **official methods**. Several nonprofit scientific organizations have compiled and published these methods of analysis for food products, which have been carefully developed and standardized. They allow for comparability of results between different laboratories that follow the same procedure, and for evaluating results obtained using new or more rapid procedures.

1.5.1   AOAC International

AOAC International, which dates back to 1884, is well known for its program to provide official methods of analysis that fit their intended purpose (i.e., will perform with the necessary accuracy and precision under usual laboratory conditions). The official methods developed are intended for use by regulated industries, regulatory agencies, contract research organizations and testing laboratories, and academic institutions. AOAC International also has programs to develop standards, to test the performance of commercial and analytical methods, and for testing laboratory proficiencies.

AOAC International uses advisory panels, stakeholder panels, working groups, and expert review panels in their collaborative system of voluntary consensus building to develop fit-for-purpose methods and services to ensure quality measurements. The *Official Methods of Analysis* of AOAC International [26] is the widely used compilation of methods adopted over the years. Details of the method validation program are given online and in the front matter of that book. Methods validated and adopted by AOAC International and the data supporting the method validation are published in the *Journal of AOAC International*. AOAC International's *Official Methods of Analysis* was most recently published in 2016, but an online version of the book is available as a "continuous edition," with new and revised methods posted as soon as they are approved. The *Official Methods of Analysis* of AOAC International includes methods appropriate for a wide variety of products and other materials (Table 1.6). These methods often are specified by the FDA with regard to legal requirements for food products. They are generally the methods followed by the FDA and the **Food Safety and Inspection Service** (FSIS) of the **United States Department of Agriculture** (USDA) to check the nutritional labeling information on foods and to check foods for the presence or absence of undesirable residues or residue levels.

1.5.2   Other Endorsed Methods

The AACCI publishes a set of approved laboratory methods, applicable mostly to cereal products (e.g., baking quality, gluten, physical dough tests, staleness/texture). The AACCI process of adopting the *Approved Methods of Analysis* [27] is consistent with the process used by the AOACI and AOCS. Approved methods of the AACCI are continuously reviewed, critiqued, and updated (Table 1.7) and are now available online.

The AOCS publishes a set of official methods and recommended practices, applicable mostly to fat and oil analysis (e.g., vegetable oils, glycerin, lecithin) [28] (Table 1.8). AOCS is a widely used methodology source on the subjects of edible fats and oils, oilseeds and oilseed proteins, soaps and synthetic detergents,

1.6 table	Table of contents of 2016 *Official Methods of Analysis of AOAC International* [26]

Chapter	Title
1	Agriculture liming materials
2	Fertilizers
3	Plants
4	Animal feed
5	Drugs in feeds
6	Disinfectants
7	Pesticide formulations
8	Hazardous substances
9	Metals and other elements at trace levels in foods
10	Pesticide and industrial chemical residues
11	Waters; and salt
12	Microchemical methods
13	Radioactivity
14	Veterinary analytical toxicology
15	Cosmetics
16	Extraneous materials: isolation
17	Microbiological methods
18	Drugs: part I
19	Drugs: part II
20	Drugs: part III
21	Drugs: part IV
22	Drugs: part V
23	Drugs and feed additives in animal tissues
24	Forensic sciences
25	Baking powders and baking chemicals
26	Distilled liquors
27	Malt beverages and brewing materials
28	Wines
29	Nonalcoholic beverages and concentrates
30	Coffee and tea
31	Cacao bean and its products
32	Cereal foods
33	Dairy products
34	Eggs and egg products
35	Fish and other marine products
36	Flavors
37	Fruits and fruit products
38	Gelatin, dessert preparations, and mixes
39	Meat and meat products
40	Nuts and nut products
41	Oils and fats
42	Vegetable products, processed
43	Spices and other condiments
44	Sugars and sugar products
45	Vitamins and other nutrients
46	Color additives
47	Food additives: direct
48	Food additives: indirect
49	Natural toxins
50	Infant formulas, baby foods, and enteral products
51	Dietary supplements

1.7 table	Table of contents of 2010 *Approved Methods of AACC International* [27]

Chapter	Title
2	Acidity
4	Acids
6	Admixture of flours
7	Amino acids
8	Total ash
10	Baking quality
11	Biotechnology
12	Carbon dioxide
14	Color and pigments
20	Ingredients
22	Enzymes
26	Experimental milling
28	Extraneous matter
30	Crude fat
32	Fiber
33	Sensory analysis
38	Gluten
39	Infrared analysis
40	Inorganic constituents
42	Microorganisms
44	Moisture
45	Mycotoxins
46	Nitrogen
48	Oxidizing, bleaching, and maturing agents
54	Physical dough tests
55	Physical tests
56	Physicochemical tests
58	Special properties of fats, oils, and shortenings
61	Rice
62	Preparation of sample
64	Sampling
66	Semolina, pasta, and noodle quality
68	Solutions
74	Staleness/texture
76	Starch
78	Statistical principles
80	Sugars
82	Tables
86	Vitamins
89	Yeast

industrial fats and oils, fatty acids, oleochemicals, glycerin, and lecithin.

Standard Methods for the Examination of Dairy Products [29], published by the American Public Health Association, includes methods for the chemical analysis of milk and dairy products (e.g., acidity, fat, lactose, moisture/solids, added water) (Table 1.9). *Standard Methods for the Examination of Water and Wastewater* [30] is published jointly by the American Public Health Association, the American Water Works Association,

1.8
table

Table of contents of 2009 *Official Methods and Recommended Practices of the American Oil Chemists' Society* [28]

Section	Title
A	Vegetable oil source materials
B	Oilseed by-products
C	Commercial fats and oils
D	Soap and synthetic detergents
E	Glycerin
F	Sulfonated and sulfated oils
G	Soapstocks
H	Specifications for reagents and solvents and apparatus
J	Lecithin
M	Evaluation and design of test methods
S	Official listings
T	Recommended practices for testing industrial oils and derivatives

1.9
table

Contents of Chap. 15 on chemical and physical methods in *Standard Methods for the Examination of Dairy Products* [29]

15.010	Introduction
15.020	Acidity tests
15.030	Adulterant tests
15.040	Ash tests
15.050	Chloride tests
15.060	Contaminant tests
15.070	Extraneous material tests
15.080	Fat determination methods
15.090	Lactose and galactose tests
15.100	Minerals and food additives
15.110	Moisture and solids tests
15.120	Multicomponent tests
15.130	Protein/nitrogen tests
15.140	Rancidity tests
15.150	Sanitizer tests
15.160	Vitamins A, D2, and D3 in milk products, HPLC method
15.170	Functional tests
15.180	Cited references

and the Water Environment Federation. *Food Chemicals Codex* [31], published by USP, contains methods for the analysis of certain food additives. Some trade associations publish standard methods for the analysis of their specific products. The FDA refers analysts to manuals for general laboratory quality practices [32], elemental analysis methods [33], drug and chemical residue analysis methods [34], pesticide analysis methods [35, 36], and macroanalytical procedures (e.g., evaluation by sight, smell, or taste) [37]. The USDA refers analysts to the *Chemistry Laboratory Guidebook* [38], which contains test methods used by USDA laboratories for ensuring the safety and accurate labeling of foods regulated by the USDA (see also Chap. 3, Sect. 3.3).

1.6 SUMMARY

Food scientists and technologists determine the chemical composition and physical characteristics of foods routinely as part of their quality management, product development, or research activities. For example, the types of samples analyzed in a quality management program of a food company can include raw materials, process control samples, finished products, competitors' samples, and consumer complaint samples. Consumer, government, and food industry concern for food quality and safety has increased the importance of analyses that determine composition and critical product characteristics.

To successfully base decisions on results of any analysis, one must correctly conduct all three major steps in the analysis: (1) select and prepare samples, (2) perform the assay, and (3) calculate and interpret the results. The choice of analysis method is usually based on the objective of the analysis, the characteristics of the method itself (e.g., specificity, accuracy, precision, speed, cost of equipment, and training of personnel), and the food matrix involved. Validation of the method is important, as is the use of standard reference materials to ensure quality results. Rapid methods used for quality assessment in a production facility may be less accurate but much faster than official methods used for nutritional labeling. Endorsed methods for the chemical analyses of foods have been compiled and published by AOAC International, AACCI, AOCS, and certain other nonprofit scientific organizations. These methods allow for comparison of results between different laboratories and for evaluation of new or more rapid procedures.

1.7 STUDY QUESTIONS

1. Identify six specific reasons you might need to determine certain chemical characteristics of a food product (or ingredient) as part of a quality management program.
2. You are considering the use of a new method to measure compound X in your food product. List six factors you will consider before adopting this new method in your quality assurance laboratory.
3. In your work at a food company, you mentioned to a coworker something about the *Official Methods of Analysis* published by AOAC International. The coworker asks you what AOAC International does and what the *Official Methods of Analysis* is. Answer your coworker's questions.
4. For each type of product listed below, identify a publication in which you can find standard

methods of analysis appropriate for the product:

(a) Ice cream
(b) Enriched flour
(c) Wastewater (from food processing plant)
(d) Margarine

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