Pearson's

Ninth Edition

Composition and Analysis of Foods

Pearson's Composition and Analysis of Foods

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Preface to the Ninth Edition

In this edition we have tried to strike a balance in content to suit the needs of readers who are interested in differing fields in food analysis. This has called for the inclusion of some methods in reasonable detail, with an indication of the main reactions involved and references to alternative procedures, together with some indication of how the results are to be interpreted, especially from the legal viewpoint. As with the eighth edition, the choice of material has been increasingly influenced by the effect of international organisations, particularly the European Community, on methods and standards.

Analysis plays an important role in the assessment and maintenance of food quality and safety, both in industry and for enforcement authorities at the national and international levels. The phrase 'chemical analysis' has been used in previous editions in the restricted sense of only those methods of analysis which use chemical processes; in the eighth edition it was used in the broader sense to encompass physical techniques used to characterise and measure chemical substances, and in this edition the trend has been continued. However, for the purpose of this new edition we have come to the conclusion that the long established title 'Chemical Analysis of Foods' is no longer suitable. In view of the extended inclusion of information on food standards and on food composition we believe that the new title 'Composition and Analysis of Foods' more accurately reflects the contents. We have felt no need to exclude the name of David Pearson from the new title since we believe that the spirit of his long association with the book should be maintained.

Opportunity has been taken to consolidate the changes made in the eighth edition, not only in relation to changes on account of newer EC Directives and other international requirements such as those of the Codex Alimentarius Commission but also by taking into account substantive changes in food legislation which have taken place in the 1980s, to a lesser extent revisions of BSI and ISO standards and methods published and recommended by the Analytical Methods Committee of the Analytical Division of the Royal Society of Chemistry. In all respects we have also tried to retain the benefit of experience which the previous authors have incorporated in the earlier editions, particularly in relation to the readership. This can range from the student of food science to the professional laboratory manager requiring technical information and available methods of analysis, but we hope that with the inclusion in this edition of more compositional data, this book will also be found useful by such workers as dietitians, home economists, etc. We have included a short table of nutritional data in Appendix 13. The text has been further amplified by references

for more detailed or more specialised interests. Although comprehensiveness is impossible in so vast a field we have tried to provide a broad cross-section of methods of analysis of interest to school, college, university, industrial and enforcement laboratories. Minor revisions of the distribution of material between chapters have been made. The reader should note that the use of proprietary names in this book does not imply endorsement of the product or Company.

Harold Egan's untimely death in June 1984 was a sad occasion. His activities in the wider sphere of food science and analysis at the national and international level were such that the scientific community lost a wealth of experience. It was perhaps fortunate that he was able to contribute some of his knowledge to the content of the eighth edition of this book. Although before his retirement he had conveyed to the present authors his intention to withdraw from active participation in the production of a ninth edition, his exceptional energy and drive to complete on target will be missed by both of us.

We would also like to thank all those friends and colleagues both within and without LGC who have troubled to provide comment and criticism on the earlier edition. Many of their suggestions have been incorporated into the present text. We would like to acknowledge the official associations, societies, industrial organisations and others who have given permission to use published and other information in this and earlier editions.

1989 RSK, RS

Abbreviations

used in this book or commonly met in analytical, standards, trade and official documentation dealing with food. See also Appendix 1.

AACC	American Association of Cereal Chemists
AAS	atomic absortion spectroscopy
AMC	Analytical Methods Committee
AOAC	Association of Official Analytical Chemists
AOCS	American Oil Chemists' Society
APA	Association of Public Analysts
BFMIRA	British Food Manufacturing Industries Research Association (now Leatherhead Food RA)
BP	British Pharmacopoeia
BPC	British Pharmaceutical Codex
BS	British Standard
BSI	British Standards Institution
COT	Committee on Toxicology
EC (EEC)	European Community (European Economic Community)
ECD	electron capture detector
FAC (FdAC)	Food Advisory Committee
FACC	Food Additives and Contaminants Committee
FAO	Food and Agriculture Organization
FDA	US Food and Drug Administration
FDF	Food and Drink Federation
FID	flame ionisation detector
FMF	Food Manufacturers' Federation
FSC	Food Standards Committee
FTNMR	Fourier transform NMR
GC/MS	combined gas chromatography and mass spectrometry
GLC	gas–liquid chromatography
GMP	Good manufacturing practice
GSC	gas solid chromatography
HPLC	high performance liquid chromatography
HPTLC	high performance TLC
ICP-OES	inductively coupled plasma-optical emission spectroscopy
ICUMSA	International Commission for Uniform Methods of Sugar Analysis
IDF	International Dairy Federation
IOB	Institute of Brewing
ISO	International Organization for Standardization
IUPAC	International Union for Pure and Applied Chemistry
LACOTS	Local Authorities' Coordinating Body on Trading Standards
LAJAC	Local Authorities' Joint Advisory Committee
LGC	Laboratory of the Government Chemist
MAFF	Ministry of Agriculture, Fisheries and Food
NMR	nuclear magnetic resonance (spectroscopy)
OIV	International Office of Wine

p.p.m.	parts per million (equivalent to mg/kg or mg/l)
RSC	Royal Society of Chemistry
SAC	Society for Analytical Chemistry
SI	Statutory Instrument, also Système International d'Unités
SPA	Society of Public Analysts (now the Analytical Division of the Royal Society of Chemistry)
SR & O	Statutory Rules and Orders
TLC	thin-layer chromatography
USDA	United States Department of Agriculture
WCOT	wall coated open tubular column
WHO	World Health Organization

Contents

1

Introduction. Legislation, Standards and Nutrition

Food analysis

In earlier times the food analyst was mainly concerned with gross adulteration. Nowadays there is an increasing tendency to examine food from a more positive viewpoint. Processed foods are produced within the limits of prescribed manufacturing formulations, set also to comply with legal or other recognised requirements. This is attained by standardising, as far as possible, the process at each of the following stages: at the farm, the 'raw' material, the process itself and finally the product and its storage. Enhanced manufacturing capacity and the complexity of modern products has necessitated the development of techniques which are suitable for rapid assessment and control. Concurrently, attempts have been made to replace subjective methods of assessing various organoleptic qualities by more precise objective procedures. Our knowledge of even minor food constituents has improved markedly due particularly to the application of newer techniques of separation, identification and measurement.

In many food laboratories, most of the routine work comprises methods of proximate analysis and the study of additives and contaminants. The main compositional components of interest are moisture, fat, protein, ash and available and unavailable carbohydrate. In practice the methods used may vary according to the food under examination: they may also be empirical. For example, the results obtained for moisture and fat depend on the procedure adopted and it is often only the free moisture or free fat that is determined. Moisture (or more correctly, loss on drying) values obtained by oven drying may include other volatile matter such as essential oils, traces of volatile acids and amines. Much attention must be paid to the preparation of the sample and the particle size. Sugars from natural sources such as fruits may include a mixture of several compounds but it is often convenient to express these together as the total soluble solids (uncorrected), as measured by a simple refractometric determination. Protein can be calculated from the total nitrogen as determined by the Kjeldahl method using an arbitrary factor which, because of the differing proportions of amino acid groups present, varies according to the food. 'Fibre' and 'ash' are essentially analytical terms. Neither represent a precise component or group of components in the original food but as long as the same standard procedure is applied to the same food each time, the results provide an adequate basis for interpretation. In many estimations allowance has to be made for interference arising from the food itself or from contamination of the reagents and

blank determinations must also be performed. Allowance may sometimes have to be made for changes which occur during storage. Many different alternative methods may be available for some analyses and it is obviously not possible to quote all of these in working detail. We have therefore had to be selective, sometimes on an arbitrary basis, but in doing this we have tried also to indicate where other methods (or reviews of these) may be found. In particular, preference has been given to methods which have been studied collaboratively. Such preference recognises the need to establish tolerance limits which are appropriate not only to the method but also to the matrix analysed. The importance of these factors and the associated sampling problems must be taken into account in the application of analytical procedures to the enforcement of standards of composition and to limits of contamination. In this respect recognition must be given to the need to obtain the performance characteristics of methods in the region of specification or legal limits. Horwitz and Albert (1987) have discussed methods for assessment of analytical procedures; the AMC (1987a, b) has presented recommendations on the estimation, definition and use of detection limits and also on the conduct of trials of methods. Validation of methods of analysis in respect of quality control of the performance of laboratories has also been discussed by Wood (1986).

Internationally recognised reference procedures have been published by bodies such as ISO, IUPAC, Codex Alimentarius and the AOAC. In the UK, standard methods for some foods have been published by the British Standards Institution and the Analytical Division of the Chemical Society (1974), which receives reports from its Analytical Methods Committee (Chirnside and Hamence, 1974).

Frequent reference is made in the text to the AOAC methods book which is the Official Methods of Analysis of the Association of Official Analytical Chemists (editor S. Williams), published by the Association of Official Analytical Chemists, 1111 N 19th Street, Arlington VA, USA. The AOAC methods book is now in its fifteenth edition, published in 1990, in a new format.

Some organisations which serve a particular industry are concerned with the development of analytical methods suitable for use within that industry, e.g. IFJU (fruit juice), ICUMSA (sugar). A list of such organisations is given in Appendix 1.

Some of the problems in food analysis arise from the fact that many of the ingredients used in the manufacture of a composite food are themselves foods of biological origin and vary widely in chemical composition. For example, flour used in the manufacture of bread, cakes and biscuits may be prepared by various milling procedures from different varieties of wheat. The composition of the manufactured article may therefore vary widely, depending on the composition of the natural or prepared ingredients. Difficulties can thus arise when analysis of the final product is contemplated in order to assess the amount of the natural ingredients used in its manufacture, e.g. tomato content of a ketchup, fruit juice content of a soft drink or cocoa content of chocolate. This can only be achieved by the analysis of the product for a major component of the natural ingredient which is not provided significantly by other ingredients used. However, the natural variation in the concentration of the selected indicator component in the ingredient makes it difficult to choose a suitable mean or minimum figure to use in the calculation.

As in other fields of analysis, the availability of good methods is essential if accurate results are to be obtained. The overall importance of cleanliness at the

bench and meticulous attention to detail at each step cannot be over-emphasised. Good analytical results also depend on careful sample preparation and accurate analyses are of little use unless the results can be interpreted meaningfully. It is therefore important that laboratory workers acquire a realistic appreciation of the fundamentals of sampling, statistics and assessment of quality criteria, together with an understanding of the significance of the data obtained. The development in recent years of a wide range of convenience foods has been due both to advances in food science and technology and to changes in the style of food distribution and marketing. Convenience foods include ready meals (dehydrated, frozen or preserved), low calorie dishes and products, instant soups and desserts, sauces and garnishes, bread and cake mixes, pie fillings, fruit drink powders, dietetic and baby foods, snack foods, processed meat products and analogues. It is not possible to cover in detail the composition and analysis of this wide range of food products. Many can be analysed by methods used for examination of the traditional classes of foodstuffs. Certain convenience foods contain technological additives such as emulsifiers, stabilisers, colours and flavours and they may also contain modified ingredients derived from starches, carbohydrates, lipids and proteins.

Furthermore, as a result of the growth in food science and technology, consumer awareness and the ever increasing range of food products, there has been a change in emphasis in food legislation in recent years, both in the EC and in the UK, away from compositional regulation and more toward informative labelling. This has led to new technical challenges to the food analyst, who will be required to confirm the correctness of food label declarations, e.g. to determine which species of meat are present in the sample which may be a cooked product, or whether a pasta product is prepared solely from durum wheat, or whether a so-called non-dairy product contains milk ingredients.

The application of standardised methods of analysis to products which contain substantial amounts of modified ingredients may yield results which are significantly in error; the analyst is advised to proceed with caution in such circumstances and where possible to cross check method performance against products of known or similar composition. Typically, the presence of emulsifiers, stabilisers, gelling agents, modified starches and similar ingredients will significantly affect the performance of extraction processes and in some circumstances the recommended method may prove to be totally inoperable.

Food legislation

Food has been liable to adulteration to a greater or lesser degree since very early times. The first recorded instances of adulteration in Britain date back to the Middle Ages when organised commerce in food began. At later dates, individual statutes for foods such as beer, wine, tea and coffee were introduced by the Excise Authorities in order to protect the revenue. Scientific methods for the detection of the more subtle forms of sophistication were then almost non-existent and it was almost impossible to give the ordinary consumer any real protection.

The turning-point came in the nineteenth century. Between 1820 and 1860, attention was drawn to the prevalence of food adulteration by a few medical practitioners, Members of Parliament and microscopists. In 1850 Wakley, the editor of *The Lancet*, established a Sanitary Commission to review the types of fraud which

were being practised in relation to the food supply. The Commission's reports, published in *The Lancet* from 1851 to 1854, were largely responsible for the Government's decision to set up a Select Committee on the Adulteration of Food in 1855. Subsequently, an Act for Preventing the Adulteration of Articles of Food or Drink became law in 1860. This established the appointment of Public Analysts, who included at that time a number of medically qualified analysts. In the decade after 1860 much attention was paid to the need for a legally acceptable and workable definition of 'adulteration'. One of the early proposals formulated for inclusion in the new Sale of Food Act 1875, was to the effect that a food or drink shall be deemed to be adulterated if:

1. It contains any ingredients which may render it injurious to the health of the consumer.
2. It contains any substance that sensibly increases its weight, bulk, or strength, unless the presence of such substance be due to circumstances necessarily appertaining to its collection or manufacture, or be necessary for its preservation, or be acknowledged at its time of sale.
3. Any important constituent has been wholly or in part abstracted, without acknowledgement being made at the time of sale.
4. It be a colourable imitation of, or be sold under the name of, another article.

The 1875 Sale of Food Act established two principal offences, the mixing of injurious ingredients and selling to the prejudice of the purchaser a food not of the nature, substance or quality demanded. Subsequent Acts in 1928 and 1938 were eventually succeeded by the 1955 Food and Drugs Act; which in turn was replaced by the 1968 Medicines Act and the 1984 Food Act. The two principal offences however have stood the test of time and are the basis of present-day legislation. The first of these requires proof of injuriousness, the second evidence of composition: both are matters of opinion though in some areas Ministers have made regulations which in effect leave room only for analytical opinion. This is especially the case in respect of compositional matters and in the use of permitted additives, which in effect control the commercial adulteration. Contamination may be regarded as the harmful (accidental or deliberate) aspect of adulteration and as such, and with the notable exceptions of lead, arsenic and (more recently) vinyl chloride monomer for which specific regulations apply, are controlled only by general provisions of the present Food Acts.

The main provisions of the Foods Acts in the UK may be summarised as follows. Section 1 of the Food Act 1984 makes it an offence to sell for human consumption any food to which substances have been added or abstracted or which have been processed to render it injurious to health. Section 2 prohibits the sale to the prejudice of the consumer of food not of the nature, substance or quality demanded. Sections 6 and 8 prevent the use of false or misleading descriptions and the sale of unfit food. Powers to make regulations to control composition (including use of additives and extent of contamination) are conferred under Section 4 whilst Section 7 confers similar powers in respect of labelling. New regulations are developed and old regulations are revised taking into account the advice of the Food Advisory Committee.

As this edition goes to print, the Food Safety Bill, successor to the Food Act 1984,

has been published and is expected to become law later in 1990. It is essentially an enabling instrument which, through regulations, will strengthen the enforcement of food law in the UK, particularly with regard to food safety. It will continue to encompass the main features of the 1984 Act relating to food analysis, i.e. nature, substance and quality, composition, labelling, additives and contaminants and will absorb EC obligations into regulations.

The UK joined the European Economic Community (EC) on 1 January 1973. The Treaty of Rome gives the Community institutions the powers and procedures necessary to achieve the objective of establishing a common market by harmonising economic activities and the laws governing these. The Laws of the Community are expressed in the form of Regulations and Directives. The essential difference is that the Regulations must be incorporated into national legislation, whilst requirements of the Directives are built into the framework of individual legislative systems. In general, Regulations are concerned with primary agricultural produce whilst Directives are concerned with detailed compositional aspects of manufactured foods. A list of Directives and Regulations is given in Appendix 3. A comparative study of food law enforcement in the EC Member States has been published by Roberts (1977) and the implications for the analyst have been described by Crosby (1982), Sawyer (1976) and Shenton (1976). Various aspects of the development of EC legislation in relation to the food industry and the impact on UK legislation have been described by Turner (1978) and Goodall (1977); Haigh (1978) has outlined the aims, objectives and philosophies of the European Commission.

Nutritional evaluation

Analysis is widely accepted as a basis for the nutritional evaluation of food, whether natural or processed, and is comprehensively treated as such in the widely accepted publication on the composition of food by Paul and Southgate (1978). Various conversion factors have been used for the calculation of food energy values. The SI unit of energy is the Joule and recommendations to adopt this in place of the calorie as the unit of food energy were made by the Food Standards Committee; these recommendations have been implemented in part by the 1984 Labelling Regulations which require that the expression for energy calculated in kilojoules shall predominate. The Labelling Regulations also include factors for use when calculating food energy values in Joules: 16 kJ/g (3·75 kcal/g) for carbohydrate, expressed as monosaccharide, 17 kJ/g (4·0 kcal/g) for protein, 37 kJ/g (9.0 kcal/g) for fat and 29 kJ/g (7·0 kcal/g) for alcohol. Paul and Southgate list food energy values in both kilocalories (kcal) and kilojoules (kJ) and use the same factors.

The Food Standards Committee's second report on Claims and Misleading Descriptions (1980) supported a recommendation of its first (1966) Report that the labelling of foods, in respect of claims for nutritive value due to vitamin and mineral content, should be controlled by a Code of Practice and that this should be incorporated in future labelling legislation. For a claim to be made, at least one-sixth of the recommended daily amount of a vitamin or mineral should be provided by the amount of the food normally consumed in one day. For the food to be described as a 'rich' or 'excellent' source the amount of vitamin or mineral present must be at least one-half of the recommended daily amount. These proposals, along with others in respect of claims on polyunsaturated fats, cholesterol, slimming properties, energy

content, diabetic and other medical qualities, were all included in the 1984 Food Labelling Regulations. The schedule set out in Table 1.1 gives detailed information regarding the levels of vitamins and minerals which are allowed in food in relation to a specific claim. Each nutrient specified shall include its biologically active equivalent or derivative.

Table 1.1 Recommended daily allowances (RDA) of vitamins and minerals

Recommended name	RDA	To be calculated as
Vitamin A	750 μg	μg of retinol or μg of retinol equivalents on the basis that 6 of beta-carotene or 12 μg of other biologically active carotenoids equals 1 μg of retinol equivalent
Thiamin (vitamin B_1)*	1·2 mg	mg of thiamin hydrochloride
Riboflavin (vitamin B_2)*	1·6 mg	mg of riboflavin
Niacin	18 mg	mg of nicotinic acid or mg of nicotinamide or tryptophan on the basis that 60 mg equals 1 mg of niacin
Folic acid	300 μg	μg of total folic acid
Vitamin B_{12}	2 μg	mg of cobalamins
Vitamin C (ascorbic acid)*	30 mg	mg of ascorbic acid
Vitamin D	2·5 μg	μg of ergocalciferol (vitamin D_2) or μg of cholecalciferol (vitamin D_3)
Calcium	500 mg	mg of calcium
Iodine	140 μg	μg of iodine
Iron	12 mg	mg of iron

* This name may also be added in parenthesis

So far as polyunsaturated fatty acid content is concerned the 1984 Regulations stipulate that the food must contain at least 35 per cent fat by weight and at least 45 per cent of the fatty acid content be polyunsaturated and not more than 25 per cent of the fatty acid content be saturated. The claim regarding cholesterol levels require that the food contains no more than 0·005 per cent cholesterol and that it also satisfies the provisions of the polyunsaturated fatty acid claim.

However, a recent Commission proposal for a Council Directive on compulsory nutrition labelling of foodstuffs (89/C296/04) (OJ No. C296, 24.11.89, p. 3) lists the following energy conversions: carbohydrate 17 kJ/g, 4 kcal/g; sugar alcohols 10, 2·4; protein 17, 4; fat 37, 9; ethanol 29, 7; organic acid 13, 3 respectively. The Directive will also deal with nutritional claims. Protein is defined as N × 6·25 for all foods except where the protein is totally from milk when 6·38 applies. Fat means total lipids including phospholipids. Percentages of the Recommended Daily Allowances (see Table 1.2) may be declared numerically or graphically. Fifteen per cent of the RDA per 100 g or per a less amount should be regarded as a significant amount.

International standards

The United Nations, through the Food and Agriculture Organization, the World Health Organization and the joint Codex Alimentarius Commission, aims to promote international trade through the development of internationally accepted food standards. There are recommended international standards published for the individual foods such as sugars, edible oils, canned fruits and vegetables, fruit juices, quick frozen foods, processed meats, fish products and cocoa products. A list of

Table 1.2 Vitamins and minerals which may be declared and their Recommended Daily Allowances (RDAs). Annex to proposal for a Council Directive on nutrition labelling, see text

Vitamin A (μg)	1000	Vitamin B_{12} (μg)	3
Vitamin D (μg)	5	Biotin (mg)	0·15
Vitamin E (μg)	10	Pantothenic acid (mg)	6
Vitamin C (μg)	60	Calcium (mg)	800
Thiamin (mg)	1·4	Phosphorus (mg)	800
Riboflavin (mg)	1·6	Iron (mg)	12
Niacin (mg)	18	Magnesium (mg)	300
Vitamin B_6 (μg)	2	Zinc (mg)	15
Folacin (μg)	400	Iodine (μg)	150

Codex Standards is given in Appendix 2. All of the standards are detailed specifications most of which require analytical methods for their realisation and enforcement. Consultations have been established between the various international organisations with interests in development of analytical methods. Other bodies concerned include the AOAC, IUPAC and ISO, which also has interests in the development of standards and methods of sampling and analysis especially for primary agricultural produce, together with the various international bodies representing individual commodity interest indicated in Appendix 1. Attention has also been given to the development of a common sampling terminology by international standards organisations; this is in recognition of the need to obtain international agreement on enforcement standards.

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2

General chemical methods

SAMPLING AND SAMPLE PREPARATION

Sampling

The value of the result of a chemical analysis on a well prepared laboratory sample will depend on how representative the sample is of the lot, batch, package or consignment of the particular food from which it was taken and on the kind of chemical information that is required. Foodstuffs and food ingredients are relatively heterogeneous materials, so it is difficult to obtain a single absolutely representative sample for laboratory analysis. The problem may be minimised by selecting several samples from the lot either randomly or according to a plan. These samples may be analysed separately to yield results from which the average composition of the lot may be computed, or in certain cases the samples may be thoroughly mixed to give a single large representative bulk sample from which a sample may be taken for laboratory analysis.

The process of sampling is one facet of statistics and most books on statistics include chapters describing the elementary mathematical principles involved. Kratochvil and Taylor (1981) give a useful short introduction and the same authors (1982) present a survey of recent literature on sampling. The Codex Alimentarius Commission has published sampling plans for prepackaged foods (CAC/RM 42–1969) and the Codex Committee on Methods of Analysis and Sampling has produced a document giving guidance notes for commodity committees on the general principles for Codex sampling procedures (Wood, 1985). Other useful compilations have been published by the Ministry of Defence (1965), Kramer and Twigg (1966) and Herschdoerfer (1967). BS 5309: Part I: 1976 gives an introduction and describes the general principles of methods for sampling chemical products. There are ISO standards for the sampling of various foodstuffs. Because of the practical difficulties and economic aspects of full statistical sampling, and the natural variation in the composition of foodstuffs, food analysis is often carried out on randomly chosen single samples.

Preparation of laboratory samples

In order to obtain precise analytical results, the laboratory sample must be made as homogeneous as possible so that, within the limits of the analytical method used, the replicate analyses agree closely. The method of homogenisation will depend on the type of food being analysed. A number of very efficient electrical mechanical devices