

Introduction to the Chemical Analysis of Foods

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List of Abbreviations

AACC	American Association of Cereal Chemists	DMTA	dynamic mechanical thermal analysis
AAS	atomic absorption spectroscopy	DNFB	1-fluoro-2,4-dinitrobenzene
ADC	analog-to-digital converter	DNP	dinitrophenyl
ADP	adenosine-5'-diphosphate	DSC	differential scanning calorimetry
AES	atomic emission spectroscopy	DTA	differential thermal analysis
AI	artificial intelligence	DTNB	5,5'-dithiobis-2-nitrobenzoic acid
AOAC	Association of Official Analytical Chemists	dwb	dry weight basis
AOCS	American Oil Chemists' Society	ECD	electron capture detector
AOM	active oxygen method	EDTA	ethylenediaminetetraacetic acid
APHA	American Public Health Association	EI	electron impact
ASCI	American Standard for Information Interchange	EIA	enzyme immunoassay
ASTM	American Society for Testing Materials	ELISA	enzyme linked immunosorbent assay
ATP	adenosine-5'-triphosphate	EMF	electromotive force
ATR	attenuated total reflectance	EPA	Environmental Protection Agency
BCA	bicinchoninic acid	ERH	equilibrium relative humidity
BCD	binary coded decimal	ESR	electron spin resonance
Be	degrees Baumé	FAB	fast atom bombardment
BGG	bovine gamma globulin	FAME	fatty acid methyl esters
BHA	butylated hydroxyanisole	FAO/WHO	Food and Agricultural Organization/ World Health Organization
BHT	butylated hydroxytoluene	FDA	Food and Drug Administration
BOD	biochemical oxygen demand	FD&C	Food, Drug and Cosmetic
BSA	bovine serum albumin	FDNB	1-fluoro-2,4-dinitrobenzene
BV	biological value	FFA	free fatty acid
CAST	calf antibiotic and sulfa test	FGIS	Federal Grain Inspection Service
CFR	Code of Federal Regulations	FIA	fluoroimmunoassay
CGC	capillary gas chromatography	FID	flame ionization detector
CI	chemical ionization	FID	free induction decay
CI	confidence interval	FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
CID	Commercial Item Description	FNB/NAS	Food and Nutrition Board of the National Academy of Sciences
COD	chemical oxygen demand	F-6-P	fructose-6-phosphate
CPC	cetylpyridinium chloride	FPD	flame photometric detector
C-PER	calculated protein efficiency ratio	FT	Fourier transform
CPU	central processing unit	FTC	Federal Trade Commission
CQC	2,6-dichloroquinonechloroimide	FT-ESR	Fourier transform - electron spin resonance
CV	coefficient of variation	FTIR	Fourier transform infrared
CVM	Center for Veterinary Medicine	FT-NMR	Fourier transform - nuclear magnetic resonance
CW	continuous wave	Gal-DH	β -galactosidase dehydrogenase or galactose dehydrogenase
DAL	defect action level	GC	gas chromatography
DC	direct current	GC-AED	gas chromatography - atomic emission detector
DC-PER	discriminant calculated protein efficiency ratio	GC-FTIR	gas chromatography - Fourier transform infrared
DHHS	Department of Health and Human Services		
DMF	dimethylformamide		
DMD	D-malate dehydrogenase		

GC-MS	gas chromatography - mass spectrometry	MPIP	Meat Poultry Inspection Program
GFC	gel-filtration chromatography	MRI	magnetic resonance imaging
GHz	gigahertz	MRMs	multiresidue methods
GMP	Good Manufacturing Practices	MS	mass spectrometry (or spectrometer)
G-6-P	glucose-6-phosphate	MW	molecular weight
GPC	gel-permeation chromatography	NAD	nicotinamide-adenine dinucleotide
G6P-DH	glucose-6-phosphate dehydrogenase	NADP	nicotinamide-adenine dinucleotide phosphate
HACCP	Hazard Analysis Critical Control Point	NADPH	reduced NADP
HETP	height equivalent to a theoretical plate	NCWM	National Conferences on Weights and Measures
HK	hexokinase		
HMDS	hexamethyldisilazane	NIR	near-infrared
HMF	5-hydroxymethyl-2-furfural	NIST	National Institute of Standards Technology
HPCE	high-performance capillary electrophoresis		
HPLC	high-performance liquid chromatography	NMFS	National Marine Fisheries Service
HPLC-MS	high-performance liquid chromatography - mass spectroscopy	NMR	nuclear magnetic resonance
HPTLC	high-performance thin-layer chromatography	nOe	nuclear Overhauser enhancement
HRGC	high-resolution gas chromatography	NPR	net protein ratio
HRP-B	horseradish peroxidase-benzidine	NPU	net protein utilization
IC	integrated circuit	NRC	National Research Council
ICP	inductively coupled plasma	NSSP	National Shellfish Sanitation Program
ICP-AES	inductively coupled plasma - atomic emission spectroscopy	OCls	organochlorines
ICTA	International Confederation for Thermal Analysis	ODS	octadecylsilyl
IDEA	immobilized digestive enzyme assay	OPs	organophosphates
IMS	Interstate Milk Shippers	PAGE	polyacrylamide gel electrophoresis
INT	indonitrotetrazolium	PAM I	Pesticide Analytical Manual, Volume I
IR	infrared	PAM II	Pesticide Analytical Manual, Volume II
ISA	ionic strength adjustor	PCBs	polychlorinated biphenyls
ISE	ion-selective electrode	PDCAAS	protein digestibility - corrected amino acid score
IU	International Units	PEEK	polyether ether ketone
IUPAC	International Union of Pure and Applied Chemistry	PER	protein efficiency ratio
KFR	Karl Fischer reagent	PGI	phosphoglucose isomerase
KFReq	Karl Fischer reagent water equivalence	pI	isoelectric point
KHL	keyhole limpet hemocyanin	PID	photoionization detector
KHP	potassium acid phthalate	PMO	Pasteurized Milk Ordinance
LALLS	low-angle laser light scattering	ppb	parts per billion
LAN	local area network	PPD	Purchase Product Description
LC	liquid chromatography	ppm	parts per million
LC-MS	liquid chromatography - mass spectroscopy	PUFA	polyunsaturated fatty acids
LDH	lactate dehydrogenase	PVPP	polyvinylpyrrolidone
LIMS	laboratory information management system	RAC	raw agricultural commodity
MAS	magic angle spinning	RF	radio frequency
MCL	maximum contaminant level	RIA	radioimmunoassay
MECC	micellar electrokinetic capillary chromatography	RPAR	Rebuttable Presumption Against Registration
MFL	million fibers per liter	RPER	relative protein efficiency ratio
MHz	megahertz	RCS	rapid scan correlation
		SD	standard deviation
		SDH	sorbitol dehydrogenase
		SDS	sodium dodecyl sulfate
		SDS-PAGE	sodium dodecyl sulfate - polyacrylamide gel electrophoresis
		SEC	size-exclusion chromatography
		SFC	solid fat content
		SFC	supercritical-fluid chromatography

SFI	solid fat index	TMS	tri-methylsilyl
SIM	selected ion monitoring	TNBS	trinitrobenzenesulphonic acid
S/L	solid/liquid	TOC	total organic carbon
SNF	solids-not-fat	TS-MS	thermospray - mass spectrometry
SO	sulfite oxidase	TSS	total soluble solids
SRMs	single residue methods	TSUSA	Tariff Schedules of the United States of America
STOP	swab test on premises		
TBA	thiobarbituric acid	USCS	United States Customs Service
TCD	thermal conductivity detector	USDA	United States Department of Agriculture
TEMED	tetramethylethylenediamine	UV	ultraviolet
TEPA	tetraethylenepentamine	UV-Vis	ultraviolet-visible
TGA	thermogravimetric analysis	Vis	visible
TIC	total ion current	VPP	vegetable protein product
TLC	thin-layer chromatography	wwb	wet weight basis
TMCS	trimethylchlorosilane		

Preface and Acknowledgments

This book was designed for use as a text primarily for undergraduate students majoring in food science who are currently studying the chemical analysis of foods. It should also be useful to workers in the food industry who do food analysis. Chapter authors are primarily university faculty members who are teaching or have taught a food analysis course, and who are very familiar with the specific topic of the chapters by nature of their research programs. Each chapter has been reviewed by persons working in the food industry who are familiar with and utilize that technique.

The book is not a laboratory manual but instead is designed to provide the lecture materials in an easy-to-follow outline format, with a brief discussion for each section. It provides much of the information on techniques that students must possess before they are able to conduct those laboratory experiments that normally accompany a food analysis course.

This book covers only the analysis of chemical properties of foods, and not physical properties. It is not intended as a detailed reference, but as a general introduction to the techniques used in food analysis. The course instructor can expand on the information in lecture, as desired, providing more details for any particular technique.

General information on sampling and data handling provides the background for discussing specific methods to determine the chemical composition and characteristics of foods. Large sections on spectroscopy and chromatography are included to explain principles of the techniques themselves and how they relate to methods of food analysis. Other methods and instrumentation such as ion selective electrodes, enzymes, immunoassays, and thermal analysis are also covered from the perspective of their use in the chemical analysis of foods. A chapter is included that relates food analysis to government regulations and recommendations.

All topics include information on the basic principles, procedures, advantages, limitations, and applications of food analysis. All chapters have summaries and study questions, and key words or phrases are identified. Many also have practice problems and a comparison of possible methods.

Most of the material covered in this book requires an understanding of general, organic, analytical and food chemistry, as well as biochemistry. With this basic knowledge, along with a food analysis course and

other relevant food science courses, it is hoped that students can function within the food industry as necessary relevant to food analysis. This book will provide a good basis for food scientists and technologists as they begin work in the food industry, whether or not they are the persons directly involved in analysis of the food products. I would greatly appreciate comments from students, instructors and food industry professionals as to how well this book meets their needs, as well as any suggestions for later editions.

I wish to thank the persons who prepared each of the chapters in this book. Those of us who teach or have taught food analysis are indebted to our former students, who gave us the needed perspective as we prepared our chapters, intended to meet the needs of future students. I also wish to thank the authors of articles and books, as well as the publishers and industrial companies, for their permission to reproduce materials used here. Special thanks is extended to Dr. Y. H. Hui who advised me throughout this project. Becky Atkinson-Hitt is acknowledged for providing exceptional secretarial assistance. I thank the many persons from the food industry, government, and academia who kindly reviewed one or more chapters in this book. They offered their assistance on this project with the hope that this book might meet the needs of present employees in their companies, and that it might also meet the needs of students who will be their future employees. They are:

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General Information

Introduction to Food Analysis

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1.1 INTRODUCTION

Investigations in food science and technology, whether in universities, governmental agencies, or the food industry, often require determination of food composition. Various types of samples may require analysis as part of a research program, as new food products are developed, or as part of a **quality assurance** program for existing products. The chemical composition of foods is often determined to establish the acceptability or nutritive value of the food product. The nature of the sample and the specific reason for the analysis commonly dictate the choice of analysis method. Speed, **precision**, and **accuracy** are often key factors that determine the choice of method. In addition to actually performing the assay, the success of any analysis method relies on the proper selection and preparation of the food sample and on the appropriate calculations and interpretation of the data. **Official methods** of analysis developed by several non-profit scientific organizations allow for comparison of results between different laboratories, and for comparisons to 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** most relevant to the chemical analysis of foods will be covered in Chapter 2.

1.2 TYPES OF SAMPLES ANALYZED

The chemical analysis of foods is an important part of the quality assurance program in food processing, from ingredients and raw materials, through processing, to the finished products (1–3). It is also important in formulating and developing new products and evaluating new processes for making food products, and in identifying the source of the problem for unacceptable products (Table 1-1). For each type of product 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 **nutritional labeling** generally requires the use of official methods of analysis. Critical questions, including those listed in Table 1-1, can be answered by analyzing various types of samples in a food processing system.

1.3 STEPS IN ANALYSIS

1.3.1 Select and Prepare Sample

In analyzing food samples of the types described above, all results depend on obtaining a representative

TABLE 1-1. 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? 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 Control Samples	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? Will it be acceptable to the consumer? Will it have the appropriate shelf life?
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 from (4,5)

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 Chapter 3.

1.3.2 Perform the Assay

Performing the assay is the step in food analysis that 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 the other two steps in analysis described here (sections 1.3.1 and 1.3.3), while the remainder of the book addresses this step of actually performing the assay. The descriptions of the various specific procedures are meant to be overviews of the methods. To actually perform the assays, details regarding chemicals, reagents, apparatus, and step-by-step instructions should be found in the referenced books and articles.