Chimia 51 (1997) 714–716
© Neue Schweizerische Chemische Gesellschaft
ISSN 0009–4293

Analytical Chemistry in Industrial Food Research

Laurent B. Fay* and **Ian Horman**

Abstract. Because of the complexity of food matrices, analytical chemistry has a key role to play in the elucidation of food composition, food stability and its behaviour during processing. In this paper, the application of various analytical techniques (*e.g.*, near IR spectroscopy, electronic nose technology, nuclear magnetic resonance and mass spectrometry) are discussed.

Introduction

Webster's Third New International Dictionary defines analysis as the separation of a whole into its fundamental elements or component parts. To elucidate food composition, its behaviour during processing and its stability, many analytical techniques are required. These include analytical chemistry, a part of science that has revealed the remarkable complexity of food matrices. Looking just at the carbohydrate field, some hundred monosaccharides have been identified. It is thus easy to imagine that the number of oligosaccharides and polysaccharides in foods, formed by combination of monosaccharides, is several orders of magnitude higher.

A constantly increasing array of analytical tools allows to go further and further in the detailed analysis of foods, separating and identifying substances with molecular masses in a range from less than 100 Da to beyond 100 000 Da. However, to cite *Malissa*, the sharper our probes are, ... the more our ability for proper judgement has to be developed [1]. Indeed, analysis, however sophisticated it might be, cannot guarantee alone high and constant product quality [2].

Food quality, as outlined in the *Table*, has three dimensions. Innovation and renovation of food products must take into account all of the three, and among the many disciplines needed for this, analytical chemistry has its role to play. Analytical chemistry is of importance to establish the molecular basis of food aroma, food colour, food texture, *etc.* – namely the *perceivable* qualities – and relating these to the *social* qualities of consumer preference. And, analytical chemistry plays an even bigger role in assuring the *invisible* qualities of food, for food safety and nutritional value – even beyond legislative norms. In fact, analytical chemistry helps driving production technology to join all dimensions of food quality.

Analytical Chemistry in Food Safety

Historically, the analytical laboratory in the food industry has been a gatekeeper, controlling the quality of incoming raw materials and outgoing finished products. For modern food manufacturing, this traditional approach is by far not enough. It allows at best an inventory or recognition of problems but neither prevents nor solves them, and as such does not provide the necessary safety and security.

Table. *The Three Dimensions of Food Quality*

Social quality	Perceivable quality	Invisible quality
Price/Performance	Flavour	Nutritional value
Convenience	Colour	Safety
Cultural/Ethnic	Textdure	
Conviviality	Appearance	
Pleasure	Sound	

Quality system tools like Hazard Analysis at Critical Control Points (HACCP) and Good Manufacturing Practice (GMP) have been developed to render the whole food chain transparent. Their aim is to identify any hazard along the production line that might affect product safety and quality, so as to prevent them from happening. To be effective, these tools need a working base of sound data, and these are in part generated *via* analytical chemistry.

Food laboratories rely on a wide range of analytical techniques and methods which fall into three categories: reference, rapid and robust, and on-line. Reference methods are the most accurate, but can be time-consuming. They mainly use fine analytical techniques such as GC, GC/MS, HPLC or ICP, and are generally validated or registered officially through organisations such as ISO or IUPAC.

For use in the factory laboratory, rapid and robust control methods are needed to ensure quick release of products for sale: these generally involve measuring whole food systems rather than individual components. Here, NIR has almost become a standard for determining residual humidity in dehydrated products such as milk or tomato powders, but is also used for multicomponent analysis as shown in *Fig. 1*. For such analyses, FT/IR also has great potential.

The future will bring an increased use of on-line methods, incorporating targeted sensors in production lines to yield dynamic data of the concentrations of individual compounds or of groups of compounds. The example in *Fig. 2* illustrates the use of *Taguchi* sensors as an artificial nose to recognise and classify instant coffees according to different blends.

Analytical Chemistry and Nutrients

Carbohydrates, lipids and proteins are the three food macronutrients as well as the major building blocks of food structure. Analytical chemistry is an essential

**Correspondence*: Dr. L.B. Fay
Nestlé Research Centre, Nestec Ltd.
Vers-chez-les-Blanc
P.O. Box 44
CH–1000 Lausanne 26
Tel.: +41 21 785 86 09
Fax: +41 21 785 85 54

CHIMIA 51 (1997) Nr. 10 (Oktober)

tool to understand their physicochemical functions in food and their nutritional role along with vitamins and minerals, in ensuring health and well-being.

Carbohydrates

Gas and liquid chromatography (GC and HPLC) are the 'classical' techniques for monosaccharide analysis. However, GC calls for extensive cleanup and derivatisation steps, while HPLC with detection by UV or refraction index (RI) suffers from low selectivity and sensitivity. Size-exclusion chromatography applied to oligosaccharide analysis also shows limited resolution. Recently, high performance anion exchange chromatography (HPAEC) coupled with pulsed amperometric detection (PAD) showed up as an interesting technology for determination of mono- and oligosaccharides at low levels, without the need for derivatisation and only minimal sample cleanup [3]. It exploits the weak acidity of carbohydrates to give highly selective separations on a strong ion-exchange stationary phase and can be applied routinely, yielding fast and reliable data on free sugars in foods or structural information on polysaccharides.

Lipids

Unsaturated fats readily oxidise and can impair flavour and nutritional value. To determine the status of lipid oxidation, the resistance to oxidation and antioxidant activity, accelerated storage tests like the *Schaal* oven or the *Rancimat* have been used traditionally. These involve applying high temperatures, high oxygen concentration or oxidation catalysts, but these results do not necessarily reflect the real situation under normal food storage conditions. Thus, more representative techniques for early detection of lipid oxidation have been developed, *e.g.*, measuring headspace oxygen consumption or the concentration of phospholipid hydroperoxides.

Oxidation profiles of isolated lipid fractions can be completely different from profiles obtained in the food matrix, thus the trend goes to measuring oxidation directly in foods, *e.g.*, by monitoring O_2 consumption with a specific electrode. Other analytical techniques include using electron spin resonance spectroscopy (ESR) to quantify free radicals formed in food products.

Proteins

Over the past two decades, mass spectrometry has become a tool with considerable impact in the field of protein analysis. The feasibility of on-line coupling between separation techniques such as liquid chromatography or capillary electrophoresis and the mass spectrometer has led to using fast atom bombardment and electrospray ionisation to analyse samples without derivatisation. Typical applications are the mass determination of proteins and peptides and the elucidation of their amino-acid sequence [4]. Initially used by the pharmaceutical industry and in life sciences, these sophisticated techniques are now entering the food science laboratory.

Minerals and Trace Elements

The analysis of minerals and trace elements in foods covers two different aspects: The determination of the absolute content, and the investigation of the bioavailability, namely the fraction of the content that is really absorbed in the human body. As an example, spinach contains 4 mg iron per 100 g, but only *ca.* 1% of this is absorbed, as the high content of oxalic acid forms an insoluble complex (contrary to oxalic acid, ascorbic acid promotes iron absorption).

Thermal ionisation isotope mass spectrometry permits accurate determination of the bioavailability of minerals and trace elements, and a typical experiment for calcium is illustrated in *Fig. 3*.

Mandatory nutritional labelling in the US and the need for rapid release of food products have brought advanced multi-element techniques such as inductively coupled plasma atomic emission spectrometry (ICP-AES) right into the food factory. Products can be analysed for nine or more elements within 3 h [5]. Impend-

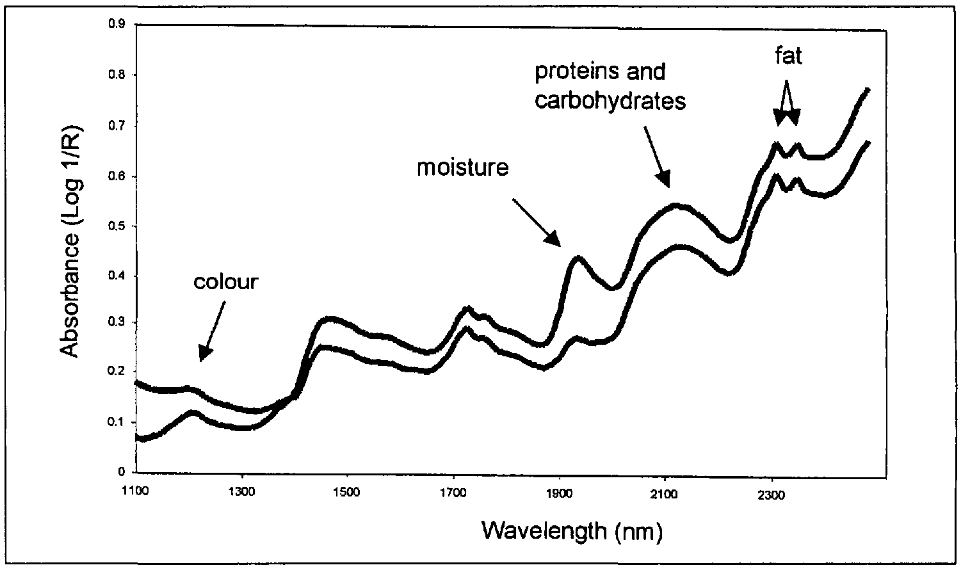

Fig. 1. *Near IR spectra of whole roast coffee beans at two levels of roasting, showing characteristic peaks for different components.* A typical quantitative analysis takes 2 min once the instrument is calibrated relative to known standards.

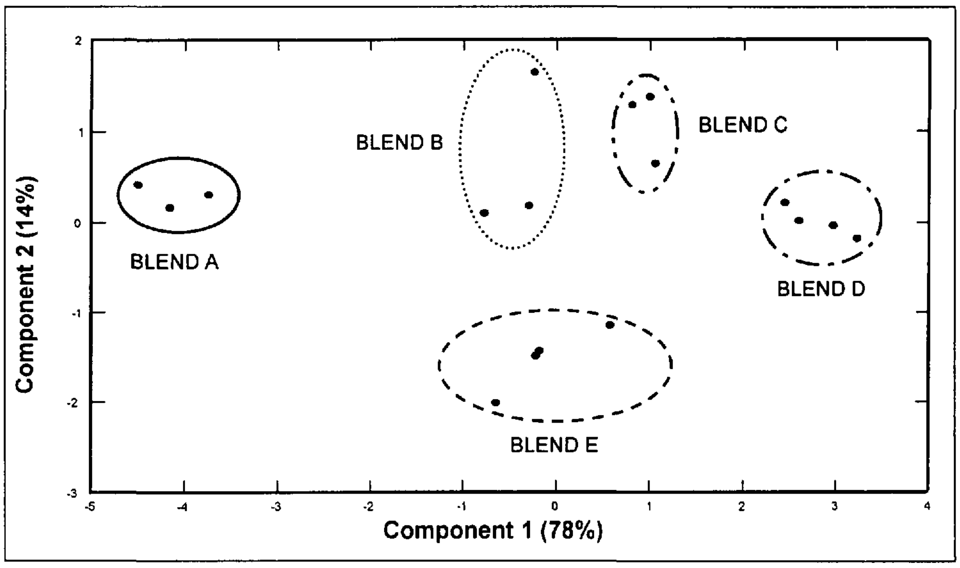

Fig. 2. *Differentiation of instant coffee blends using 'electronic nose' data and principal components analysis*

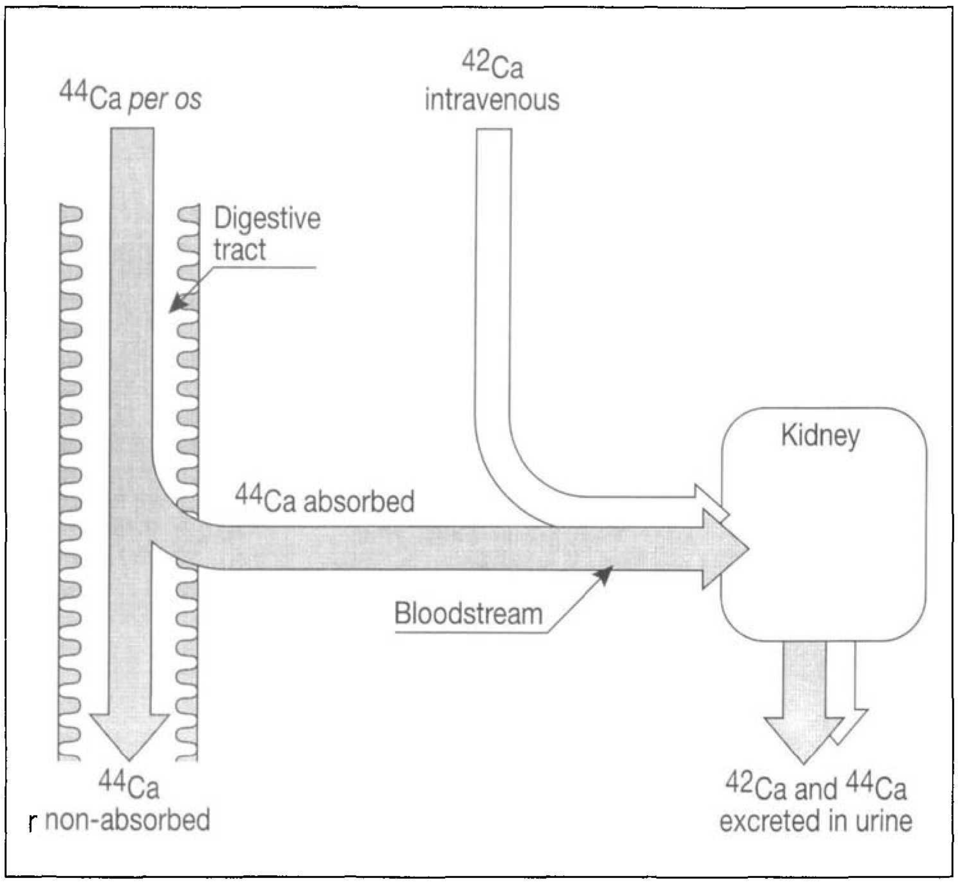

Fig. 3. *The dual isotope technique for measuring bioavailability of calcium uses one stable isotope injected intravenously as a standard to detect the proportion of a second isotope absorbed* via *the gut after oral ingestion in a food matrix*

ing European legislation for lead and cadmium limits in raw materials will demand advanced techniques such as graphite furnace-atomic absorption spectrometry or inductively coupled plasma mass spectrometry (ICP-MS) [6].

Analytical Chemistry and Perceivable Food Quality

The sensory aspects of foods, outlined in the *Table,* play a key role in food choice. 'Hi-tech' analytical techniques like high resolution nuclear magnetic resonance or mass spectrometry have a long history in various fields of food science. They have been used extensively to study flavours, colours or textures of food products, and through the results, to guide new product development.

Nuclear Magnetic Resonance

High-resolution NMR today aims at structure elucidation of complex macromolecules like proteins, starches and fibres, glycoconjugates and DNA fragments. This technique has been employed for structural elucidation of constituents in raw or processed materials, of metabolites generated in the body and for quantification of individual components in whole food systems [7]. The use of isotope-labelled analogues of aroma compounds has allowed following the fate of these compounds during food processing.

During the last few years, a number of new techniques have emerged, largely expanding the potential applications of NMR in food research. Solid-state NMR for studies of insoluble plant cell walls, high resolution magic angle spinning for gels, field gradient spin echo based diffusion measurements for emulsions, on-line coupling of HPLC to NMR and, eventually, NMR microscopy are some future NMR techniques of food analysts.

Gas Chromatography

Steam distillation was long used to isolate food volatiles but, for obvious reasons, always gave a 'cooked' aroma. Thus, a number of techniques has been developed to obtain representative samples. These include headspace analysis, simultaneous distillation-extraction under vacuum [8] or, more recently, solid-phase microextraction [9]. Linking between sensory and instrumental analysis, GC-olfactometry permits determination of the odour threshold of individual volatile compounds [10]. Nevertheless, GC and GC/MS remain the flavour analyst's workhorses, with high sensitivity and selectivity, backed up by extensive databases containing sensory, chromatographic and spectroscopic attributes of thousands of volatile compounds [11].

Mass Spectrometry

Today, tandem mass spectrometry is becoming more and more popular [12]. The commercial availability of triple quadrupole instruments and quadrupole ion trap mass spectrometers at accessible prices has brought this tool into the food chemist's laboratories. Improvements in instrument design and computer control now offer the user machines that can be operated under routine conditions. As an example, flavour analysis deals with complex matrices of several hundreds of components, where 'key components' are often present only in traces. Since tandem mass spectrometry offers structural identification as well as quantitative determinations, we will see an expansion of its use, hyphenated or not to chromatographic separations, in food analysis.

The authors would like to thank their colleagues from the *Nestlé Research Center*, Lausanne, for providing the necessary background information to prepare this article.

Received: August 29, 1997

[1] H. Malissa, *Fresenius J. Anal. Chem.* **1990**, *337*, 159.

[2] R. Stalder, *Laboratory Technology International* **1997**, 162–164.

[3] M.R. Hardy, R.R. Townsend, Y.C. Lee, *Anal. Biochem.* **1988**, *170*, 54.

[4] K.A. Walsh, L.H. Ericsson, K. Resing, R.J. Johnson, in 'Method in Protein Sequence Analysis', Eds. K. Imahori, and F. Sakiyama, Plenum Press, New York, 1993.

[5] K.W. Barnes, E. Debrah, *Atom. Spect.* **1997**, *18*, 41.

[6] G. Ellen, J.W. Van Loon, *Food Addit. Contam.* **1990**, *7*, 265.

[7] I. Horman, in 'Analysis of Food and Beverages', Ed. G. Charalambous, Publ. Academic Press Inc., London, 1984.

[8] L. Maignial, P. Pibarot, G. Bonetti, A. Chaintreau, J. P. Marion, *J. Chromatogr.* **1992**, *606*, 87.

[9] C.L. Arthur, J. B. Pawliszyn, *Anal. Chem.* **1990**, *62*, 2145.

[10] I. Blank, in 'Techniques for Analysing Food Aroma', Ed. R. Marsili, M. Dekker Inc., New York, 1997.

[11] R.A. Hearmon, *Fresenius J. Anal. Chem.* **1992**, *344*, 164.

[12] L.B. Fay, I. Blank, C. Cerny, in 'Flavour Science: Recent Developments', Eds. A.J. Taylor, and D.S. Mottram, The Royal Society of Chemistry, Cambridge, 1996.