ANALYTICAL METHODS OF FOOD AUTHENTICATION

Edited by P.R. Ashurst and M.J. Dennis

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Preface

As we stated in the earlier volume *Food Authentication* the adulteration of food is not a new issue. It has, within the past decade become much sharper, partly because of the trend in Europe and other parts of the world to move from compositional legislation to label declaration and partly because of the development of analytical methods. The legal issues surrounding label declarations are pursued with differing vigour by the various enforcement bodies, but this volume is not concerned as such with that aspect.

Having examined in some detail the main food commodity groups, for it is within commodities that the problem mainly exists, this volume surveys the range of analytical methods that are today applied to the investigation and confirmation of authenticity or adulteration. At the outset it must be recognized that for any food commodity it is invariably the case that there is no single test that will confirm the authenticity of a product. As will be seen almost all types of analysis, both complex and simple, are used in one or another commodity. Two common strands are evident in all authenticity work. One is the creation and maintenance of databases and the other is the handling of the large amounts of data generated.

There is deliberately no chapter in this volume dealing with databases because these will vary to meet the special needs of each commodity. We have, however, included a chapter on the chemometric methods that are now widely used for data processing and evaluation – a direct result of the computing resources that are available universally at relatively low cost.

Stable isotope analyses have become an important tool in the detection of adulteration and chapters are included on both mass spectrometry and nuclear magnetic resonance as alternative ways of obtaining ratios of the key stable isotopes. Mass spectrometry linked to pyrolysis is dealt with in a separate chapter and the significance of both mid- and near-infrared spectroscopy are recognized. Other analytical techniques that each have their own chapters include oligosaccharide analysis, trace elements analysis and the important biochemical techniques such as enzyme based methods, analyses linked to DNA identification and comparison, antibody techniques and electrophoresis. Finally, a short miscellaneous chapter examines some of the simpler but none the less effective tools such as microscopy.

We have also included a chapter on the detection of irradiated foodstuffs which although perhaps not strictly an authenticity issue does rate highly in consensus of consumers and enforcement bodies.

This book is the work of its authors, all eminent and respected specialists in their own fields and the editors are very appreciative of their contributions. We take full responsibility for the overall work 'warts and all' and it is hoped the reader will find this a worthy successor to the original volume.

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1 Introduction to methods for food authentication

M.J. DENNIS and P.R. ASHURST

1.1 Aims of food authentication

When food analysts carry out food authenticity tests, what are they trying to achieve? The answer depends very much on the requirements of the customer. Food producers, food enforcement agencies and national governments each have subtly different needs and these must be taken into account when the choice of method of analysis is made.

1.1.1 *Food producing companies*

Reputable food producers want to ensure the products they manufacture meet the demands of legislation in the countries where they are sold. They also wish to avoid paying a premium price for an inferior commodity. Equally the costs of the authenticity test are important and, in some manufacturing situations, the speed with which the test can be accomplished may be very important. Although food companies will wish to use soundly based methodology to meet their obligations under due diligence legislation, they do not necessarily need the degree of method validation which is appropriate in other situations. Thus, when purchasing raw materials, they are in a position to specify the composition of the material they are buying. They do not necessarily have to justify this specification; they can simply say 'this is not the product I want'. One consequence of this is that the larger, more scientifically aware companies, can be among the first to adopt new technologies in food authentication when these meet a perceived need.

1.1.2 *Enforcement authorities*

In contrast, method validity is of crucial importance to enforcement authorities. It is vital that they can show that the methods used are defensible in a court of law. Thus, the methods must be rigorously defined and their validity established either through long usage, or preferably, through collaborative testing. Enforcement authorities will therefore tend to concentrate on validating and applying methodologies developed by universities and research institutes rather than undertaking cutting-edge research. In this, their position is similar to that of the food companies, who do not consider it their role to devote resources to research but are content to apply the best methods currently available.

1.1.3 *National governments*

The organization of the enforcement of food law differs between countries. However, it is not unusual for law enforcement to be undertaken at a local or district level. This is the case in the UK where enforcement is generally under the direction of local authorities. Nevertheless, national governments must have an interest in co-ordinating and directing these activities. Only at the national or international level can the combination be found of the appreciation of the need to invest in authenticity research and the resources to carry out such work. Governments need to ensure that sound, well-validated methods are available to meet the needs of both industry and enforcement in order to protect the public from misleading or fraudulent labelling. In this they seek to meet the needs of the public, as voters and consumers, and the food industry as an engine of the economy. There is no conflict of interest between these parties. Each needs to know that when they buy a premium product, they get what they pay for.

Governments have a difficult task in deciding where best to direct resources for food authentication. Where existing methods of analysis have not been fully validated or usage has indicated problems, then it is appropriate to fund method development and validation work. Equally, where new technologies arise and have potential application to food authentication (e.g. DNA biotechnology) then further research to develop these applications is merited; particularly where current methods need to be improved. It is also important to maintain an awareness of the market place to ensure that important authenticity issues are not being overlooked.

1.2 Networks

Regular contacts of scientists for the discussion and debate of issues has always been fundamental to the development of the scientific method of study. However, the pace of change in food authentication is unrelenting and it is necessary for all parties to maintain close awareness of both scientific developments, and developments in the market place. In the UK for example, the Ministry of Agriculture's Food Authenticity Working Party allows representatives of consumers, food manufacturers and retailers, food law enforcement co-ordinators and scientists to contribute their views as to how the authenticity of foods should be ensured.

In the United States, the Food and Drug Administration (FDA) is seeking a closer working relationship with the food industry and inviting them to become partners and customers of the planned Joint Institute for Food Safety and Applied Nutrition (JIFSAN) which is being set up in co-operation with the University of Maryland (Anon, 1997). The FDA emphasized the need for information sharing between state and federal regulators, food manufacturers and international agencies. It was recognized that industry is often in a unique position to provide information on dishonest competitors, significant changes in

commodity markets and new technologies. One proposal considered was that US Customs services should co-operate with other countries to prevent exporters seeking alternative ports of entry when adulterated products were rejected at the border. The European Commission has recognized the importance of providing support for food authenticity networks by funding Food Authenticity Issues and Methodologies (FAIM). This group brings together over 40 food scientists to review authenticity issues and analytical methodology in fruit-based products, honey, meat and fish, dairy products, oils and fats, cereals, tea and coffee.

Thus there is considerable international recognition of the need to encourage information exchange in food authenticity. These more formal schemes together with the individual initiatives of scientists working in key commodity areas ensure that resources are targeted towards the most deserving issues. Nevertheless, a clear awareness of progress in ensuring the authenticity of food commodities is most commonly achieved through well-targeted surveys.

1.3 Sampling

1.3.1 *National surveillance*

There are a number of issues which should be considered when a national authenticity surveillance of a specific food (or group of foods) is contemplated. It is necessary to find a basis for determining priorities and the relative importance of the many possible issues which could be studied. Some basis for weighting the various factors must be established. It is, in principle, possible to consider any claim which is made on a food label as an issue requiring testing, but some issues will be more important than others. Many factors may influence this judgement. For instance, is this a problem which consumers are aware of? Or will feel particularly strongly about? (e.g. kosher or vegetarian labels); will the authenticity issue have an effect on the health of a sub-group of the population? (e.g. those allergic to cow's milk might be severely affected if goat's milk yoghurt or cheese contained this ingredient). Is the food commodity sold to a large percentage of the population or will only a few people be affected? Is the issue a recently discovered problem or have manufacturers and retailers known about the problem for sufficient time to put adequate monitoring systems in place? Are suitable methods of analysis available?

If it is decided on the basis of these considerations that a surveillance exercise is appropriate, then a detailed sampling plan, incorporating any constraints which the analytical method imposes, should be prepared. The type of sales outlet for the food may be significant; supermarket, small shop, restaurant or mobile sales van. Will the surveillance cover the whole country or just selected areas? Will it cover premium brands, budget brands and own label brands? How much will the analysis cost? Would it be more effective to evaluate a large number of samples with, perhaps, a cheap but less sensitive method, to gain a

good idea of how widespread a problem may be? Alternatively, is a highly accurate but expensive test required in order to detect a sophisticated adulteration issue? Has a cut-off limit been established from authentic samples by reputable laboratories using the test in question, and will these authentic data cover the range of food commodities and countries of origin that it is proposed to investigate? Have any new processing methods been carefully evaluated and shown not to affect the proposed tests? Is a confirmatory test available which will add validity to the proposed survey? All of these factors and possibly others need to be taken into account when planning a surveillance exercise.

1.3.2 *Food companies*

For the food industry, the emphasis on where authenticity testing should be directed is rather different. Lees (1996) outlined some of the considerations which needed to be taken into account. The analysis of every batch of raw materials before incorporation into the food product is termed positive release. This approach is very effective in detecting adulteration but it carries a high cost. It is appropriate for high risk situations such as when a new supplier is being used, where a regular supplier consistently delivers a product close to an authenticity limit value (and a Gaussian distribution might be expected) or where a previously good supplier has had a batch fail an authenticity test. In situations where a supplier has a good track record of providing authentic materials, then other approaches can be considered. Screening tests which are simple, fast and cheap may help to maintain confidence in a product but are unlikely to detect sophisticated adulterations. The most cost effective approach is to undertake random sampling using the best tests currently available. By using statistics, it is possible to calculate the minimum number of analyses required to ensure, for a given confidence interval (a%) that a p% non-conformity will be detected. So, for 100 sample batches in one year, if it is assumed that 1 in 4 batches are not authentic (p 25%) then only 10 batches need to be analysed to give a 95% probability (a%) of identifying the problem. This level of confidence can be increased by ensuring co-ordination of surveillance activities with retailers, trade associations, importers and producers.

Having established that analysis is required to investigate sample authenticity, it is necessary to ensure that appropriate and sufficient authentic data are available and to specify how subsequent data from unknown samples will be interpreted.

1.4 Interpreting databases

When carrying out food authentication studies, it is best to establish a firm cut-off point at which the sample will be deemed to have failed the test before any tests