Governme

Setting standards
in analytical science

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Government Chemist

Review 2007

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Front Cover: Dr Rebeca Santamaria-Fernandez using laser ablation high resolution multi-collector ICP-MS for high accuracy isotope ratio measurements on solid samples

Government Chemist

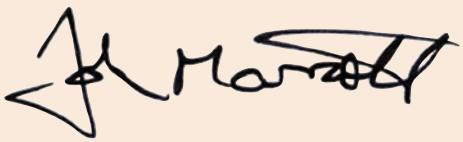
Foreword

My term of office as the Government Chemist ends in May 2008, so this will be my sixth and last Review. Looking back over the period, I see huge advances in the technology we now have at our disposal.

When I was appointed in 2002 we had just taken delivery of a new Fourier transform mass spectrometer, which promised unprecedented sensitivity, mass accuracy and resolution. Since then we have upgraded the instrument and its software several times and installed a robotic workstation to the front end, enabling us routinely to carry out sophisticated isotope exchange experiments to probe the structure of complex proteins in solution. Over the period we have added a range of advanced mass spectrometers, with such acronyms as MALDI-ToF/ToF, LC-QToF, multicollector high resolution ICP-MS; all acquired to help us probe the composition of increasingly complex samples with greater precision, enhanced accuracy and higher sensitivity.

This technology has enabled us to confirm the presence or absence of banned colourants in foods at trace levels, positively identify harmful mycotoxins derived from moulds growing on food, and study the detailed chemistry of the key trace element selenium, both in fortified foods and clinical samples, where it is being studied for its anti-cancer properties. The instruments may have changed significantly over the last six years, but there have been unimaginable changes since the first Government Chemist, George Phillips, set up his laboratory in 1842. Then the microscope, analytical balance, volumetric glassware and hydrometer were the tools of the trade. Nevertheless the job was still done and over the years a succession of Government Chemists has maintained the role of using sound analytical science in support of policy and regulation.

I hope you find the current Review interesting and informative. It summarises our activities over the year, including details of the referee analysis which we undertake to resolve disputes over analytical results. Although we have sophisticated instruments and advanced techniques in our armoury, we should not underestimate the exceptional skills of the analysts at LGC who undertake the work and maintain the credibility of the Government Chemist as the referee analyst. I am deeply indebted to their expertise and dedication.



John Marriott
BSc PhD FRSC
Government Chemist

1 Sound science for all

As referee analyst, the Government Chemist (GC) is the national focus of technical appeal for analytical measurements made pursuant to certain Acts of Parliament. Affiliated to the UK National Measurement System, we provide independent advice addressed to all affected by the dependencies between policy, standards, regulation and analytical science.

The Government Chemist is a UK public body with expertise in the area of analytical chemistry and bioscience, and a remit to help safeguard the quality of public science in this context. Specific functions include:

- Duties prescribed by Acts of Parliament and other UK legislation (Box 1), particularly in relation to food and agriculture, most often in the form of a 'referee function'. As referee, we provide decisive evidence and opinion to help avoid or resolve disputes arising in connection with analytical measurements made by business operators and UK regulatory enforcement authorities;

- Advice to Government and the wider community, provided in fulfilment of a covenant with the Secretary of State for Innovation, Universities and Skills, on the ways in which analytical science can support policy, standards and regulation.

We share the Secretary of State's aim of embedding scientific evidence and advice in all policy making[1].

Figure 1: Dr Will Burkitt studies protein conformation by hydrogen-deuterium exchange and FTICRMS

[1]John Denham speech: Science and Society. DIUS, 16 January 2008

Box 1: The Government Chemist in legislation

The duties of the GC as referee analyst are defined in or under:
Food Safety Act 1990
Food Safety (Sampling and Qualifications) Regulations 1990
Food (Northern Ireland) Order 1989
Food Safety (Northern Ireland) Order 1991
Food Safety (Sampling and Qualifications) Regulations (Northern Ireland) 1991
Poultry Meat (Water Content) Regulations 1984
Materials and Articles in Contact with Food Regulations 2007
Plastic Materials and Articles in Contact with Food Regulations 2006
Plastic Materials and Articles in Contact with Food (Lid Gaskets) Regulations 2007
Natural Mineral Water, Spring Water and Bottled Drinking Water Regulations 2007

Agriculture Act 1970
Feed (Hygiene and Enforcement) Regulations 2005
Genetically Modified Animal Feed Regulations 2004

Medicines Act 1968
Farm and Garden Chemicals Act 1967

The GC is named and has other scientific responsibilities under:
Merchant Shipping Act 1995
Hydrocarbon Oil Duties Act 1979
Poisons Act 1972

The status and territorial extent of the GC are understood with reference to:
Freedom of Information Act 2000
Scotland Act 1998 (Cross-Border Public Authorities) (Specification) Order 1999
Scottish Parliament (Disqualification) Order 2007
Administrative Provisions Act (Northern Ireland) 1928

For effective regulators

Legislators have long recognised the need for an impartial guardian of sound science by embedding the GC referee function into the architecture of technical regulation. As a safeguard of independence, we are funded by DIUS rather than by our regulatory stakeholders.

Our wider advisory remit underpins regulatory effectiveness from policy development, through the formulation and drafting of legislation, to effective implementation and enforcement. We share scientific knowledge and awareness of regulatory developments with UK enforcement authorities, and particularly with the supporting network of Public Analysts. We therefore need to be proactive about developments at all levels which could affect the official controls system.

For business

The GC functions as an independent scientific service, between regulator and regulated; businesses are therefore full stakeholders. We provide cost-effective dispute resolution, enabling expensive risk management measures such as unnecessary product withdrawal to be avoided, and maintain fair markets for diligent traders by providing advice to uphold standards of enforcement science.

The Sainsbury[2] Review recognises the role of regulation in fostering innovation. Typically, regulatory requirements create a rationale for the GC to initiate R&D projects, the results being disseminated for the wider benefit of industry. For example, advances in methods for the measurement of vitamins help companies both to comply with labelling regulations and market the nutritional benefits of their products more confidently. Sainsbury also highlighted that the increasingly modular organisation of global manufacturing chains can drive innovation. As value chains become more fragmented, the confidence of downstream companies in their suppliers comes to rely on sound analytical science. In a knowledge-based UK, the GC has a role in underpinning measurement science - not only to verify the safety of imports, but also of a calibre to meet the requirements of trading partners in global export markets.

For skills and development

We are playing our part to address the growing requirement for scientific skills and specialised expertise. For example, the GC Science Manager's annual residential course, coordinated in his capacity as Training Officer for the Association of Public Analysts (APA), is increasingly popular. We also support LGC's development of training courses to provide underpinning skills in analytical science, which, as the Sainsbury Review recognises, build on the laboratory's strengths, linked to its designation as the UK measurement institute for chemical and biochemical measurements.

For the public good

Whether through Government, industry or the scientific community, the GC serves the public interest by contributing to the knowledge economy whilst protecting safety, wellbeing and the environment. Some of our work spins out more direct benefits; for example, current research could help allergy sufferers to a fuller understanding of potential risks arising from the food chain, while completed projects have helped put particular nutritional or functional claims into context. To spread awareness, we liaise with representative bodies such as the Consumers' Association and the Anaphylaxis Campaign.

[2]Lord Sainsbury of Turville, The Race to the Top: A Review of Government's Science and Innovation Policies. HMSO, October 2007

Underpinning our capabilities

The Department for Innovation, Universities and Skills (DIUS) enables us to carry out our remit effectively by funding the Government Chemist programme, and other underpinning R&D projects through the UK National Measurement System (NMS). Staff at LGC manage and deliver the projects (Figure 2). The Department has appointed an independent adviser, Dr David Ferguson, to help monitor the discharge of the Government Chemist function by LGC.

As this year's Review goes to print, we near the end of the GC programme's three-yearly cycle. The expectations of our stakeholders have evolved significantly since 2005, and the programme has in turn developed the foresight, technology and knowledge transfer capabilities needed to meet them. The new GC Programme (2008-2011) will reflect ongoing safety concerns such as the prevalence of contamination by toxins derived from moulds in consignments of imported food, as well as emerging issues, for example the risks arising to food-allergic consumers from products containing allergens. Reflecting the Government Chemist's duty to advise on wider issues linked with analytical science - for instance, as an *ex officio* member of the Chemical Weapons Convention National Authority Advisory Committee - the new programme will also appraise our potential contribution to emergency and incident prevention.

Figure 2: The Government Chemist at LGC.

Key: *Left to right: Michael Walker (Science Manager and Nominated Officer*), Peter Bedson (Programme Manager), Rita Harte (Assistant Programme Manager), John Francis (Project Manager, Advisory Function), Peter Roper (Nominated Officer*), John Marriott (Government Chemist), Ian Lumley (GC Deputy), Selvarani Elahi (Project Manager, Statutory Function and Nominated Officer*), Peter Colwell (Project Manager, Referee Function), Rattanjit Gill (Project Manager).*

**Nominated Officers are appointed by the GC to act on his behalf. They have overall responsibility for the supervision of statutory analytical casework, including the preparation of certificates. Certificates of Analysis are signed by a Nominated Officer and countersigned by the GC or his Deputy.*

2 Our casework

In 2007 we received 21 samples for formal analysis under the provisions of the Food Safety Act 1990 (Table 1). Toxins derived from contaminating moulds continued to be a prominent concern. In this section we also highlight cases and outcomes relating to the authenticity of meat products and contamination by illegal dyes. A further study illustrates casework under the Agriculture Act 1970.

Table 1: Numbers of official samples received by the GC in 2007

Sample description	Issue to be resolved	Number
Almond	Aflatoxin contamination	5
Chilli	Aflatoxin contamination	1
Fig	Aflatoxin contamination	1
Groundnut	Aflatoxin contamination	1
Hazelnut	Aflatoxin contamination	1
Ice cream sauce	Variety of fruit present	6
Lamb mince	Presence of other species	1
Nut products	Aflatoxin contamination	3
Oregano	Whether irradiated	1
Pistachio	Aflatoxin contamination	1

Benefits for food and agricultural analysis

By underwriting sound measurement science for the food and agricultural sectors we:

- Contribute to public health and the quality of life, by reinforcing standards of consumer safety and choice;
- Protect the reputations and finances of responsible traders by ensuring effective law enforcement, including their right to a supplementary expert opinion;
- Ensure that the enforcement system and the judiciary can access scientific evidence of the required standard;
- Provide a cost-effective source of expertise to help address questions and uncertainties relating to regulatory chemical and bioanalysis.

Food safety - genotoxic contaminants

Managing the risks

Moulds that are widespread in food crops under certain climatic conditions produce a diversity of substances toxic to humans and animals (mycotoxins), some of which cause cancer. If the toxins are chemically stable, they can present a risk in both raw and processed foods. Exposure through the diet cannot be entirely avoided, so legislators aim to limit these contaminants to the lowest concentrations reasonably achievable. The limits are specified by Regulation (EC) No 1881/2006 Setting Maximum Levels for Certain Contaminants in Foodstuffs.

Recent evidence demonstrates the scale of the hazard. An estimate that 25 % of the world's food crops are affected by mycotoxins is widely accepted. Our own monthly monitoring of food recalls during 2007 corroborates the 2006 results from the EU Rapid Alert System for Food and Feed (RASFF), showing that mycotoxins are the biggest cause of incidents arising from food grown around the world. Samples suspected of containing aflatoxins, a group of at least 18 related chemicals produced by the genus *Aspergillus*, are now submitted to the Government Chemist more often than any others. Repeated findings by official control laboratories that imported consignments exceed the legally binding limits have often been coupled to uncertainties over the effectiveness of sampling inhomogeneously distributed mould in very large consignments of food commodities.

Case studies

In response to the potential threat, and following the Food Standards Agency's advice, Port Health Authorities implemented higher sampling rates for potential aflatoxin contamination in consignments of almonds. This, coupled with the right of business operators to a supplementary expert opinion[2], contributed to our caseload. Embargoed consignments incur daily containment fees at the ports, and to address this cost burden on business operators we honed our procedures to fast-track the related samples whilst maintaining full quality assurance. We also phased in the use of LC-MS/MS analysis as a confirmatory procedure (see Underpinning Science). These improvements resulted in a reduction in our turnaround times for the determination of aflatoxins by as much as 50 %.

Table 2 gives information about scientific issues that were encountered during the analysis of individual samples. In all completed cases this year, the GC confirmed the results obtained by the official control (Public Analyst) laboratory, and the consignment was returned to the country of origin or otherwise exported.

[2]Article 11(5), Regulation (EC) No 882/2004 *on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules*

Table 2: Samples received by the Government Chemist in 2007 for aflatoxin analysis

Sample group	Samples received	Case notes
Chillies	Crushed chillies	The legal concentration limits for aflatoxins in chillies are specified by the Annex to Regulation (EC) No 1881/2006 Setting Maximum Levels for Certain Contaminants in Foodstuffs, alongside those for other sample types. We reported that this sample contained the B1 form of aflatoxin in excess of the legal limit (5 μg kg^{-1}). During preparatory experiments using blank crushed chillies to which known quantities of aflatoxins had been added, we compared the extraction of samples with or without a preliminary water-based slurrying step. Results indicated that slurrying recovers the aflatoxins more effectively.
Figs	Dried figs	The samples contained a substance which interfered with measurement of the G forms of aflatoxin by the established chromatographic method. We resolved the interference by modifying the elution gradient.
Nuts	Australian almonds Blanched almond pieces Californian shelled almonds Whole almonds Groundnuts Roasted, sliced hazelnut Pistachios	As the sample descriptions show, there is significant variation in the way nuts are presented to the consumer - for example, almonds may be whole, shelled or blanched. In our experience, the recovery of aflatoxins for analysis is dependent on the way the product has been prepared for market; we had to account for this by carrying out case-specific research and control experiments. Reference materials are an important element of our quality assurance, but the range of sample types remains very limited, and the concentrations of regulated substances that they contain are not always relevant to current legislative limits. We were able to use suitable FAPAS proficiency testing materials for the quality assurance of cases relating to almonds and pistachios this year. The case involving hazelnuts was abandoned by the owner shortly after we received the samples. However, in anticipation of receiving the sample, we had undertaken a preparatory study to upgrade information on the recovery of aflatoxins from this type of product. We completed the work, which involved extraction of the hazelnuts and a successful comparison of LC-fluorescence and LC-MS/MS methods.
Compound samples[3]	Nougat containing pistachio nuts Peanut bites	We modified the standard extraction technique because of the high sugar content in these confectionery-type products. It was known that only certain components - pistachio and peanut respectively - were potential sources of aflatoxins in these products. The nut component could not be separated from either sample for analysis. The whole product was tested on the basis that all aflatoxins present derive from the nut component, in relation to which there is a specific legal limit. We took into account quantitative data on the manufacturing processes for the compound products, in order to report the results in a manner which - in recognition of the fact that food law is criminal law - gave the owner of the sample the benefit of the doubt.

Our casework

Underpinning science

High confidence identification

The GC always aims to improve the fitness of science as a sound basis both for effective trade and regulatory enforcement. Alongside the caseload of referee samples, we piloted LC-MS/MS, a powerful technique capable of identifying many chemical substances with exceptional confidence, to confirm the presence of the five regulated aflatoxins: B1, B2, G1, G2 and M1 (Figure 3). Although each type of food sample required a new investigation because its overall composition, or matrix, could have interfered with measurement, we successfully optimised the LC parameters to enable valid analysis in milk, chilli powder, baby food and peanuts.

Representative sampling

The growth of moulds tends to be patchy, so there has been concern that random sampling strategies could miss contamination hot spots. Because there is no published data on this we led a coordinated effort, involving Public Analysts (PAs) and a Port Health Authority (PHA), to assess the sampling procedure for aflatoxin contamination specified in Regulation (EC) No 401/2006 Laying Down the Methods of Sampling and Analysis for the Official Control of the Levels of Mycotoxins in Foodstuffs. The GC and the PHA each took samples from a contaminated consignment of peanuts. The samples were sent out to three PA laboratories that prepare samples for the determination of aflatoxins. As required, the PAs split the samples into three portions and used a slurrying technique to homogenise each. Three subsamples of each portion were sent for analysis as if they were being distributed among the parties to a dispute (trader, enforcement authority and referee), although for the purposes of the study, all three were in fact analysed by the GC. A full statistical assessment showed that the protocol was fit for the purpose of detecting contamination in consignments, and confirmed that the three parties are treated fairly in that they receive equivalent samples.

Measuring lower limits

The health hazards of ochratoxins, another class of mycotoxins, include kidney damage and related cancers. We developed a method capable of determining compliance with the latest regulatory limits for ochratoxin in cereal-based baby food by optimising an immunoaffinity clean-up method and scaling down the dimensions of an LC column used to isolate the substances of concern[4].

Figure 3: Reliable sample preparation partners cutting edge LC-MS/MS to boost confidence in aflatoxin measurements

[3] *A note from the Government Chemist on sampling and analysis for aflatoxin contamination in compound products containing nuts* is available at www.governmentchemist.org.uk

[4] A technical report, *Development and validation of methods with the required sensitivity to enforce the Contaminants in Food Regulations*, is available on our website, www.governmentchemist.org.uk

Food safety - authenticity of meat products

Consumers of meat products wish to know the species of animal from which any given food item is derived; religious, ethical and cultural factors make this information an important precondition for consumer choice. We have many years' experience in the field of meat speciation, and often receive related requests for referee analysis or advice (Box 2).

Box 2: A recent case requiring referee analysis to determine the species present in meat

An official sample described as lamb mince was taken from a butcher under the provisions of section 29 of the Food Safety Act 1990 by the Trading Standards Service responsible for the enforcement of food law in that locality. In accordance with the Food Safety (Sampling and Qualifications) Regulations 1990, the authorised officer divided the sample into three parts. The Public Analyst, acting on behalf of the enforcement authority, tested one part of the sample and found that it contained pork and beef as well as lamb. The owner of the sample appointed a laboratory to carry out independent tests on the second part, the results of which indicated that only lamb was present. The owner appealed to the GC as referee analyst, and we received the third part of the sample in July. We employed three independent methods and on this occasion, our results confirmed those of the Public Analyst. As well as sending a warning letter, Trading Standards discussed methods for improving procedures with the butcher.

Past cases suggest that some traders may be tempted to extend meat derived from a high value species with material from other animals, but is not always straightforward to prove that a product has been deliberately adulterated. One possible legal defence is that a degree of cross-contamination by 'drip' - fluids exuded from another species - might have arisen accidentally, perhaps during storage or processing. We undertook further characterisation studies which could help to test such a defence, but more work is required to determine the value of this approach.

Our casework

Food safety - hazardous dye contamination

Although the GC's evidence is often used to settle disputes efficiently and avoid recourse to the courts, staff can also be called as expert witnesses. This duty usually relates to a sample that has been taken under statutory provisions by an enforcement authority. However, in May we were called into a four-day civil trial by a food manufacturer in dispute with an upstream supplier of chilli powder.

We are experienced in the determination of a range of hazardous dyes[5], the use of which in food is forbidden. The manufacturer sought to prove that chilli powder supplied in 2005 was the source of Para Red dye which had been detected in its products, leading to the recall and destruction of over 100 000 retail items in accordance with FSA advice at the time. The upstream supplier used the industry standard LC method of the time and found no such contamination. Subsequently, the chilli powder was reanalysed by LC-MS/MS and found to contain Para Red at a concentration of 200 µg kg^{-1}. The court required advice on the competing claims. The questions put to our expert witness were:

- Was Para Red present in the chilli powder?
- Was LC-MS/MS a reliable method to use for the determination of Para Red in chilli powder?

We produced an expert witness report and gave evidence at the trial, answering yes to both questions after assessing all the scientific evidence. The limit of determination for the industry standard LC method was 500-1000 µg kg^{-1}, but LC-MS/MS had been validly applied at the lower concentrations relevant to this case. The court accepted the opinion of our expert witness, and on the basis of this and other evidence ruled in favour of the food manufacturer on 26 July.

Animal feed - case study

Part IV of the Agriculture Act 1970 lays down a framework of requirements for certain information to be given to the purchaser concerning material sold as an animal feeding stuff. Last year, we received a sample taken from a premium brand of mixed rabbit food. A Public Analyst, having applied a statutory method of analysis, reported that the sample was deficient in the trace nutrient copper. These results were disputed by the owner of the sample, so the local authority asked the GC to investigate.

We applied the statutory method, and cross-checked the results by microwave digestion with inductively coupled plasma optical emission spectrometry (ICP-OES). We found that the sample contained 8 mg kg^{-1} of copper, confirming that the concentration was less than the value declared on the label (25 mg kg^{-1}). The local authority consulted a nutrition expert about the welfare implications of this breach, who, after reviewing relevant research papers, found the concentration of copper to be just high enough to meet a rabbit's dietary needs. The authority therefore decided not to take legal action, but the manufacturer was advised to review quality assurance. This is the first time we have encountered a 'nutritional need' approach to deciding the outcome of a labelling case.

[5]For details, see www.governmentchemist.org.uk

3 Preparing for future action

The Government Chemist's underpinning research and knowledge transfer programme upgrades the UK referee analyst capability, thus improving responsiveness to projected demand and benefiting all stakeholders. We helped to defend the safety of the food chain by a review of marketing applications for new feed additives, and the validation of analytical methods for nut allergens. Progress supporting the verification of label claims relating to B-group vitamins and high value ingredients will help ensure that requirements for nutritional and consumer choice are met. As well as presenting our own achievements to a wider audience, we contributed to effective risk management by collating and channelling global data on food recalls to the key stakeholders.

Food safety

Allergens

Food allergy is becoming more prevalent; there are now at least 10 related deaths in the UK every year. The wider impact on the quality of life for allergy sufferers is self-evident. Costs are also escalating, in healthcare and in ensuring that food businesses comply with legislation and adopt good practice, for example by controlling the risks of cross-contamination between products. The use of any of 14 potential food allergens as an ingredient in pre-packed food is regulated: in 2007, Directive 2007/68/EC updated the scope of the requirements, and national Food Labelling (Declaration of Allergens) Regulations extended the implementing legislation. Moreover, a key Parliamentary report[6] recently recommended that food labels should clearly specify the amount of each of the allergens where it is present in a product.

This is an emerging field for analytical science, with few validated methods in the public domain, leaving significant scope for divergent results and opinion. From the outset, the GC consulted expert bodies in the UK and globally so that together we could address the main knowledge gaps as efficiently as possible. Although the scoping phase suggested that peanut and gluten were among the most likely allergens to be received for referee analysis, the acute lack of validation data for tree nuts as an allergen would have left regulatory scientists unprepared. So our first project successfully assessed commercially available kits that incorporate DNA and protein assay techniques - real-time PCR and ELISA respectively. We validated an ELISA kit to detect walnut allergens in varieties of chocolate and biscuits, and established the suitability of this method for a wider range of sample types such as breakfast cereals. Alongside the existing kit methods, we envisage that novel approaches will provide confirmatory analysis, enable increased sampling frequencies and improve coverage of the lengthening list of substances that are regarded as potentially allergenic.

Preparing for future action

Farm to fork

Requirements to apply for product authorisation introduced by Regulation (EC) No 1831/2003 on Additives for Use in Animal Nutrition led to demand for the scrutiny of applications by analytical science experts. To help ensure that the methods of analysis proposed by an applicant are fit for use by official laboratories protecting the food chain and animal welfare, one Member State produces an initial evaluation report and the other countries then have a chance to comment. This year we commented on the initial reports for nine products. While many of the methods appeared fundamentally sound, we highlighted some scientific issues for further consideration, including:

- Gaps in data needed for full confidence in the method. For example, conclusions about performance at safety-critical concentrations of an additive cannot necessarily be drawn from the limit of quantitation;
- Justification for tests that had been carried out on a chemically related compound, rather than the active substance to be marketed;
- Improvements in the specificity of detection possible by switching from older titrimetric methods to LC.

Nutrition

Vitamins are essential nutrients. Claims about products including them must be accurate, but established microbiological assay methods for determination of the B-group vitamins are slow, laborious and temperamental. In addition to the general requirements of food and feed law, specific drivers for regulatory analytical measurement of these vitamins include:

- Conditions placed on nutrition claims and labelling (Food Labelling Regulations 1996);
- Mandatory restoration of thiamin in processed flour (Bread and Flour Regulations 1998);
- Compositional standards in legislation covering baby food and slimming products;
- Restrictions on the use of riboflavin as a colourant (Miscellaneous Additives in Food Regulations 1995);
- Specification of the chemical forms of vitamins which may be used in dietary supplements (Food Supplements Regulations 2003; issues relating to quantitative limits are currently under discussion at EU level and internationally).

As part of a continuing effort to establish more robust methods of analysis, we challenged the state of the art for vitamins B1 (thiamin) and B2 (riboflavin). Our study took account of in-house R&D alongside the work of RSC's Analytical Methods Committee, CEN and USP. We showed that thiamin and riboflavin in dietary supplements can be determined effectively by LC after an acidic extraction step. For food and feed samples, enzymes can be used to isolate thiamin and riboflavin simultaneously, but care is needed in their selection. Whereas the natural fluorescence of riboflavin can be directly detected, thiamin must be converted to a fluorescent derivative, thiochrome, before the LC step, and the performance characteristics of the method can be improved by including a clean-up procedure[7].

Consumer choice

The Food Labelling Regulations 1996 as amended protect consumer nutrition, choice, and the value chain for legitimate traders by requiring the amounts of key ingredients to be stated. For protein-providing foods such as meat and fish, the accuracy of these statements can be enforced by measuring the nitrogen content and comparing it with the expected value for the species in question, known as the nitrogen factor. This factor can depend on a range of variables including the species, geographic origin and size of the animal, the tissues or cut used, the time of year, farming procedures and processing technologies. Unless these variables are investigated and quantified, they can open up grounds for challenge and dispute.

We are currently reviewing nitrogen factors for farmed Atlantic salmon. In collaboration with Trading Standards Officers and the UK's largest salmon processor, we completed a statistically determined programme of factory visits to collect samples, allowing all the main variables, including the season of the year, to be studied efficiently. We are in the process of carrying out nearly 2000 analyses to derive an authoritative modern nitrogen factor for farmed salmon.

[6]House of Lords Select Committee on Science and Technology, Sixth Report of Session 2006-2007: Allergy. HL 166; 26 September 2007

[7]For more information, see *Analytical procedures for the determination of vitamins B1 and B2 in foods, feeds and supplements* at www.governmentchemist.org.uk

DNA delivers on choice and quality

Stringent authorisation and labelling legislation allows consumers to make clear choices about products containing or produced from GMOs. In the UK, the Genetically Modified Food Regulations 2004 and the Genetically Modified Animal Feed Regulations 2004 apply. To underpin capability for referee analysis in this area, we:

- Continued to validate methods for the determination of specific DNA sequences in food and feed markets, completing seven trials in conjunction with the European Commission's Joint Research Centre;
- Completed a comparative review of the performance of microarrays as measurement platforms for GM studies, the results of which will be published through the EU collaboration Co-Extra[8];
- Modelled the effect of changing the number of replicate samples or calibration standards on the overall precision of GM determinations, and showed that under certain conditions quality need not be sacrificed to gain efficiency savings;
- Gave practical input to an interlaboratory trial coordinated by Co-Extra, which will compare the suitability of plasmid and genomic standards for quantitation of the GM content in food samples;
- Worked for improved understanding of method performance and data interpretation as part of the European Network of GMO Laboratories (ENGL).

DNA methods also contributed to our success in correctly identifying all species in 12 samples during an FSA ring trial of a technology platform for fish species determination by UK Public Analysts.

Outreach

We renewed efforts to make modern official control techniques more accessible, raise our profile with industry, and share the latest scientific developments with all stakeholders. Highlights included:

- A third residential training week for Public Analysts, titled Analysis and Examination of Food and Water, which attracted a record 24 attendees and, in an advisory capacity, the chief examiner for the Mastership in Chemical Analysis (MChemA);
- An active role in regular LACORS Food Sampling and Analysis Focus Group meetings, resulting in further dissemination of our research on illegal dyes and aflatoxins sampling - and greater interaction with LACORS' regional organs, for example in presenting to a specialist group from the Central England Trading Standards Authority (CEnTSA) on 7 June;
- Involvement in FSA-coordinated expert fora, including the new Food Irradiation Stakeholder Group (FISG);
- Contact with the British Retail Consortium, resulting in advice to the retail sector on verifying QUID declarations, and progress towards an authoritative opinion on the role of analytical testing alongside other control and audit systems;
- Enhanced awareness and dialogue concerning consumer issues through Foodaware;
- Participation in a new working group of CEN Technical Committee 327 established to develop standards for the preparation of animal feed samples;
- Global networking that included a November visit to the Hong Kong Government Laboratory and a conference presentation on food analysis in nearby Guangzhou.

All stakeholders were welcome at our third annual dissemination event, London, 1 May 2007, and over 60 attended. Richard Burt, President of the Institute of Food Science and Technology (IFST), chaired the day. The varied and compelling programme culminated in an exposition of NMS-funded research on the identification of selenium compounds, and its implications for nutritional science.

[8]GM and non-GM supply chains: their co-existence and traceability

Preparing for future action

Information hub

In safeguarding the food chain, access to a robust evidence base is as vital for effective regulatory services as it is for commercial operators bearing the brunt of the responsibility to counter key risks. We have collated evidence of global food recalls since mid-2006, publishing monthly updates at www.governmentchemist.org.uk. In line with our recent experience of demand for referee analysis, mycotoxins remain the most frequent cause of concern. We now have enough data to reflect on possible trends; for example, recalls concerning materials and articles in contact with food, such as packaging or kitchenware, are seen to be rising steadily when the data are aggregated on a quarterly basis (Figure 4).

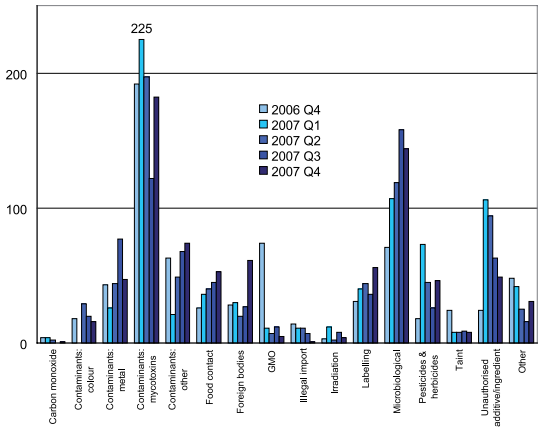


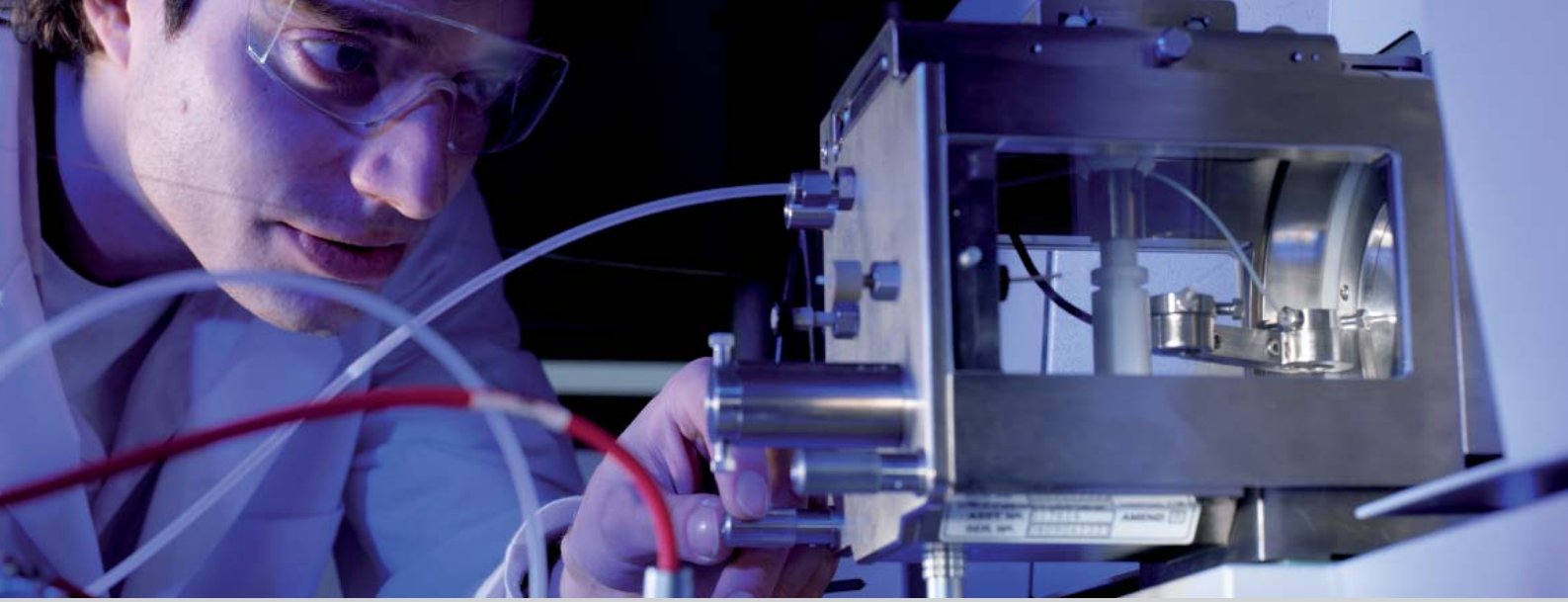
Figure 4: Global food recalls: data for the last 15 months

Forward look

Priorities for the next phase of our underpinning programme (2008-11) have been determined by an independent panel including scientific, regulatory, business and consumer representatives (Table 3).

Table 3: Food chain science priorities for the GC from April 2008

Project	Scope
Food allergens	Enhance and broaden capability for major regulated allergens
Food fraud by DNA	Develop and independently validate methods for adulterant
Mycotoxins: LC-MS/MS	Underpin requirements of Contaminants in Food Regulations
Vitamins: optimised methods	Update techniques applicable to foods, feeds and supplements
Probiotics methods assessment	Detect and enumerate 'good bacteria' in food and supplements
Food contact materials	Evaluate flexible, high performance measurement options
Aflatoxins: optimised methods	Validate and disseminate matrix-specific procedures
GM in food and feed	Develop novel practical approaches for improved quantitation
Improving feed analysis	Innovate to prepare for referee cases on undesirable substances



4 Our science base

As the home of the Government Chemist, LGC provides access to science that complements the GC remit and helps maintain strategic collaboration. This year's highlights from the laboratory include novel sampling capabilities for mass spectrometry, a broadened commitment to protein science and improved characterisation of cell culture techniques. Action to support innovators helped us maintain a broader perspective on analytical science. We give examples of some of the work.

Sample-friendly spectrometry

The National Measurement System's new Chemical and Biological Metrology programme (www.nmschembio.org.uk) takes forward a number of exciting opportunities that could find future application in regulatory analytical science. For example, there are challenging measurement issues around the transfer of chemical substances to a mass spectrometer reproducibly, at trace levels, from high throughput arrays or even intact solid samples.

A technique which has emerged in the last two years is desorption electrospray ionisation (DESI). It can operate rapidly, without conventional sample preparation, or even the usual hurdle of high vacuum operation which is a requirement for comparator techniques - such as matrix-assisted laser desorption/ionisation (MALDI) and secondary ion mass spectrometry (SIMS). These characteristics conduce to miniaturisation, opening up the prospect of portable DESI mass spectrometers for use in the field.

In collaboration with our colleagues at NPL, we have established the tools for a systematic study of direct sampling techniques. Progress included development of a method for the controlled preparation of sampling surfaces, and construction of a novel DESI source interfaced to a high performance commercial mass spectrometer (Figure 5). Scanning programs ensure that each part of the sample is probed in turn, providing traceability between the results of analysis and the region sampled so that a molecular image of the whole item or array can be generated. The study will go on to examine metrological issues critical to wider application, such as reproducible data acquisition and the potential of quantitative mapping.

These techniques for fuller sample characterisation are likely to be in demand for drug discovery and development, and for imaging in clinical and forensic contexts. They could also help resolve a variety of issues regularly encountered by the GC, including food adulteration or accidental contamination with traces of allergenic products.

Our science base

Figure 5: Peter Stokes configures the DESI source for imaging mass spectrometry

Advancing protein measurement science

Through a six-way collaboration funded by TSB's Technology Programme, LGC has exploited and built upon a hydrogen-deuterium exchange technique to determine how physical and chemical stress factors affect the shape and spatial conformation of proteins. When protein molecules are dissolved in deuterium-enriched water, hydrogen atoms that are accessible to the solvent are more readily exchanged by the heavier isotope. After a prescribed period, the exchange reaction is quenched by pH and temperature reduction, the protein is digested under carefully controlled conditions with a suitable enzyme, and the masses of the resulting fragments are determined by Fourier transform ion cyclotron resonance mass spectrometry (FTICRMS; Figure 1). The masses of the fragments will have been increased whenever deuterium has replaced accessible hydrogen. As well as providing information about conformation under the test conditions, the exchange technique can be applied widely to characterise protein-protein, protein-ligand and protein-DNA binding constants.

Working with cells

Biopharmaceutical products (biologics) manufactured using cells are already available, but are prone to minor chemical variations, occurring after the primary structure has been translated from the genetic code, because of the intrinsic variability of living cell systems. This can have significant clinical consequences and also determine whether large scale production from a chosen cell source will be achievable. Cell metabolism determines these post-translational modifications and ultimately, the yield of the desired product. Given enough data, metabolic processes in cell cultures can be directed toward safe and efficacious healthcare products - biopharmaceuticals as well as advanced therapies - chemically tailored to match the clinical need. Renewables markets also stand to benefit from metabolically engineered, sustainable feedstocks and fuels.

The National Measurement System's Innovation R&D programme has been developing an integrated approach for measuring the metabolic response of cells to changes in culture parameters such as temperature, pH and substrate availability aiming to guide and optimise bioprocessing pathways. Pilot-scale bioreactor facilities (Figure 6) have been used to test the benefit of combining a suite of measurement technologies including mass spectroscopy, protein microarrays and flow cytometry. When employed in concert, these techniques tracked key biomarkers in the culture samples more robustly. Novel data analysis techniques can feed back into instructions for the adjustment of cell culture parameters so as to favour synthesis of the desired bioproduct. The software platform is readily extensible to online process monitoring.

Figure 6: New bioreactor facilities at LGC will ease the translation of laboratory-scale research into industrial bioprocessing applications

Our platform capability in cell sciences could potentially underpin the development and validation of analytical techniques based on novel measurement principles to complement our current arsenal.

Our science base

At the forefront of innovation

The National Measurement System's Measurement for Innovators (MfI) programme promotes a fast track model for innovation by exploiting the assets of the UK's four National Measurement Institutes (NMIs): NPL, NEL, NWML and LGC. Companies and other bodies with a promising concept or prototype, but needing help with analytical science issues, can simply submit an application to LGC for processing with the least possible burden on the applicant. All the NMIs welcome applications in their respective fields of expertise.

Three forms of support are available:

- Joint Industry Projects (JIPs) - focused collaborations lasting up to a year, for which DIUS can offer to match the contributions made by the industrial partners, whether in cash or in kind;
- Consultancies - expert advice freely available to SMEs for up to four days on measurement issues relating to a specific product or service;
- Secondments - time-limited postings to or from an NMI which aim to exploit synergies, build relationships and share knowledge in the pursuit of agreed project objectives. Companies, universities and many other UK organisations are eligible.

As examples, LGC completed collaborations supporting nutritional science and pharmaceutical safety studies (see inset).

Improving dietary selenium intake

Selenium is of shared interest to food scientists, clinical researchers and metrologists because it has nutritional and potential health benefits over a limited dietary intake range, but certain chemical forms can be toxic at higher levels. LGC worked with a company specialising in the nutritional improvement of food crops to build on the current fragmentary understanding of the chemical forms of selenium present in different foods. Selenium's chemical form can affect its availability for uptake and metabolism by the body. The characterisation studies at LGC helped guide the development of more nutritious food products.

Predicting drug toxicity

The need for a reduction in animal-based testing and speedier development of new pharmaceutical compounds, coupled with the implementation of REACH, keeps up the pressure to explore alternative cell-based systems that could prove to be useful indicators of the toxic effects of chemicals. LGC completed a joint project with a company that had developed versatile software to analyse the expression of a key set of genes in human cell cultures exposed to chemicals. Two LGC statisticians reviewed practical aspects and advised on efficiency improvements to the experimental design for validation of the software predictions. The advice helped improve the robustness of a system intended to distinguish various mechanisms by which tested substances can exert toxic effects on the body, relate the dose to the biological response, and indicate what could be done to reduce the toxicity of the chemicals.

5 Our wider remit

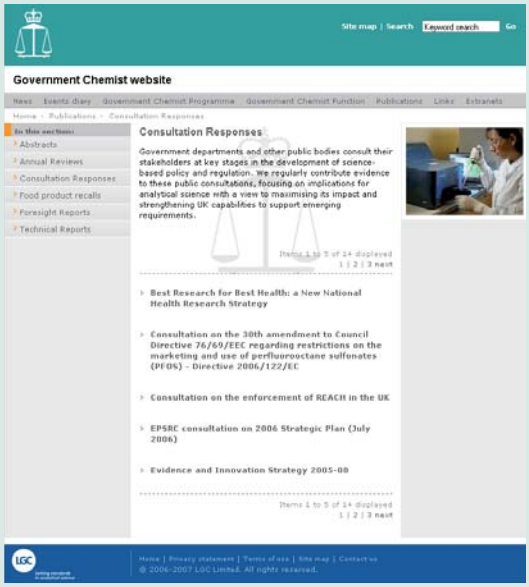
We advise Government and our wider community on the dependencies between analytical science, policy, standards and regulation. In 2007 we made an increased contribution to consultation through public and official channels. As the EU's major new chemical regulation, REACH, entered into force, we developed advice on analytical issues relating to substance identity, and convened key stakeholders to develop practical compliance approaches; we are engaging with Chemistry Innovation to help emerging technologies play their part.

Analytical science, policy, standards and regulation

The Government Chemist responded to requests for science-based advice (Box 3) on:

- Policy development relating to home affairs, healthcare, and materials and articles in contact with food;
- Standards for the quality of environmental waters, and EU-led discussions aiming to increase the contribution of standardisation to innovation;
- Regulation in draft with implications for the food chain, and for the risk assessment, marketing and use of chemicals.

Our task is often to apply the established principles of sound analytical science to a new context. Recurring themes include: minimising the uncertainties inherent in sampling procedures; the innovation-friendly specification of flexible performance requirements, rather than fixed technologies; the need to set regulatory limits that reflect current measurement capabilities as well as policy goals; developing consensus on the way analytical science can validly contribute to a wider evidence base; and exploiting global synergies. For further information, see www.governmentchemist.org.uk



Our wider remit

Box 3: Regulatory and scientific consultations to which the GC responded in 2007

Defra	Consultation on the enforcement of REACH in the UK
Defra	Consultation on the 30th amendment to Council Directive 76/69/EEC regarding restrictions on the marketing and use of perfluorooctane sulfonates (PFOS)
DIUS	Commission discussion paper on standards and innovation
FSA	EU Regulation 882/2004 on official controls - draft European Commission guidelines on annual reports on implementation of national control plans
FSA	Regulation (EC) No 882/2004 on official controls - implementing rules for import controls for 'high-risk' feed and food of non-animal origin
FSA	The draft Official Feed and Food Controls Regulations 2007
FSA	The Materials and Articles in Contact with Food Regulations 2007
FSA	The Plastic Materials and Articles in Contact with Food (Lid Gaskets) Regulations 2007
FSA	Proposal of the European Commission for a Regulation on recycled plastic for use in contact with food
MHRA	Challenges and priorities for the next five years
OSI	Review of science in the Home Office
WFD-UKTAG	Proposals for environmental quality standards for Annex VIII substances under the Water Framework Directive

The analytical toolbox

As industrial chemicals may be complex reaction products, or vary widely depending on the source, under REACH the first task for manufacturers and importers is to gather evidence of a substance's identity and present it in accordance with technical guidance from the European Chemicals Agency. We carried out cases studies with affected businesses and SMEs to develop advice on analytical issues affecting REACH substance identity.

On 11 October, we gathered over thirty experts in industrial, scientific and regulatory affairs for this year's flagship event, *REACH - the Analytical Toolbox.*

REACH submissions must demonstrate the analytical chain of evidence - both the methods used and the results obtained. Much of the required science is challenging, but fortunately, the technical guidance provides considerable scope for innovation. We reviewed the latest official guidelines and focused on developing practical compliance approaches.

Our industrial case studies attracted widespread enthusiasm, and helped participants from different backgrounds to identify common issues. Requirements for data on impurities and minor constituents were clearly challenging. There were concerns about the availability of data for some imported products, and the potential need to monitor the specifications of starting materials. Chemical nomenclature and 3D structure were key issues for complex bioproducts such as carbohydrates. In many cases, data on similar substances could be pooled to help assess risk, provided that there is chemical evidence for the way they are related.

We then presented on the underpinning conditions for sound analytical science in support of REACH: method validation, quality assurance, and the uptake of innovation, such as microarrays for toxicogenomic studies, to ease compliance.

The Analytical Toolbox reinforced a core network that is clearly proactive about the challenges REACH presents for the chemical industry and its supply chains, involving the characterisation and risk assessment of an estimated 30 000 substances. We believe the participants will cascade effectively to the wider UK community facing the new requirements.

Our wider remit

Underpinning Chemistry Innovation

The Chemistry Innovation Knowledge Transfer Network (CIKTN)[9] , established in 2006 to drive innovation performance across the UK chemistry-using industries, now has a front page focus on Measurement and Standards for Emerging Technologies (MSET).

MSET is a web-enabled channel providing rapid access to measurement information and advice focused on the needs of the parent knowledge transfer network (KTN). The aim is to help companies, and indeed all technology users, understand how measurement can impact on and improve products and services. The MSET concept had been road-tested with other KTNs including BioProcessUK, Health Technologies, and Sensors. We are developing Chemistry Innovation MSET for the benefit of the whole chemistry-using community, with proactive participation from the parent KTN and the National Physical Laboratory alongside expert input from RSC's Analytical Division.

The Chemistry Innovation MSET website[10] features case studies designed to stimulate further networking and collaboration, covering topics ranging from illegal dyes to confidence in genetic measurements. There are interactive tools implementing best practice for web-enabled communications, include online news and event listings, discussion boards and even an Ask an Expert facility providing direct access to consultancy that would otherwise attract a substantial premium. We incorporated a Standards Centre as a single access point for the normative guidelines, reference materials and harmonised technical standards that underpin sound analytical science, while our MSET Learning Hub helps address the skills gap in chemical sciences.

Our approach to MSET for Chemistry Innovation recognises that sound analytical measurement underpins the two key goals for developers of novel technologies: establishing a market and achieving regulatory acceptance. By encouraging the adoption of online communication technologies that can enable wider outreach, MSET promises to be a valuable tool for the Government Chemist and wider National Measurement System in fostering sound science.

Figure 7: David Carter, gas chromatography specialist, benchmarking innovative monitoring tests for environmental pollutants

[9]http://innovation.globalwatchonline.com
[10]www.mset.org.uk/chemistry

Publications

Belli M, Ellison SLR, Fajgelj A, Kuselman I, Sansone U, Wegscheider W. Implementation of proficiency testing schemes for a limited number of participants. Accreditation and Quality Assurance 2007;12:391-8.

Burns MJ, Valdivia H. A procedural approach for the identification of sources of uncertainty associated with GM quantification and real-time quantitative PCR measurements. European Food Research and Technology 2007;226:7-18.

Burns MJ, Valdivia H. Modelling the limit of detection in real-time quantitative PCR. European Food Research and Technology 2007;226:1513-24.

Corbisier P, Broothaerts W, Gioria S, Schimmel H, Burns MJ et al. Toward metrological traceability for DNA fragment ratios in GM quantification. 1. Effect of DNA extraction methods on the quantitative determination of Bt176 corn by real-time PCR. Journal of Agricultural and Food Chemistry 2007;55:3249-57.

Cowen S, Ellison SLR. Handling undetected and low-level components in purity determination. Accreditation and Quality Assurance 2007;12:323-8.

Domann P, Pardos-Pardos AC, Fernandes D et al. Separation-based glycoprofiling approaches using fluorescent labels. Proteomics 2007;7:70-6.

Entwisle J, Garrett J, Elahi S, Roper P. Investigations into the effect of selected storage conditions on the alcohol content and original gravity of beers samples. Food Control 2007;19:461-4.

Flasza M, Kemp P, Shering D, Qiao J, Marshall D et al. Development and manufacture of an investigational human living dermal equivalent (ICX-SKN). Regenerative Medicine 2007;2:903-18.

French DJ, Jones D, McDowell D, Thomson JA, Debenham P. Analysis of multiple single nucleotide polymorphisms closely positioned in the ovine *PRNP* gene using linear fluorescent probes and melting curve analysis. BMC Infectious Diseases 2007;7:90.

Goenaga Infante H. HPLC-ICP-MS for preliminary identification and determination of methyl-selenium metabolites of relevance to health in pharmaceutical supplements. Agilent Handbook of Hyphenated ICP-MS Applications 2007;August.

Goenaga Infante H, Joel SP, Warburton E, Hopley CJ, Hearn R, Jüliger S. Investigation of the selenium species distribution in a human B-cell lymphoma line by HPLC- and GC-ICP-MS in combination with HPLC-ESIMS/MS and GC-TOFMS after incubation with methylseleninic acid. Journal of Analytical Atomic Spectrometry 2007;22:888-96.

Goenaga Infante H, Sturgeon R, Turner J, Hearn R, Sargent M et al. Total selenium and selenomethionine in pharmaceutical yeast tablets: assessment of the state of the art of measurement capabilities through international intercomparison CCQM-P86. Analytical and Bioanalytical Chemistry 2007;390:629-42.

Henke L, Muche M, Blaauw A et al. Validation of a "new" short tandem repeat (STR) fluorescent multiplex system and report of population genetic data. Clinical Laboratory 2007;53:477-82.

Hughes P, Marshall D, Reid Y, Parkes H, Cohava G. The costs of using unauthenticated, over-passaged cell lines: how much more data do we need? Biotechniques 2007;43:575-86.

Jüliger S, Goenaga Infante H, Lister TA, Fitzgibbon J, Joel SP. Chemosensitization of B-cell lymphomas by methylseleninic acid involves nuclear factor-kB inhibition and the rapid generation of other selenium species. Cancer Research 2007;67:10984-92.

Kuselman I, Belli M, Ellison SLR, Fajgelj A, Sansone U, Wegscheider W. Comparability and compatibility of proficiency testing results in schemes with a limited number of participants. Accreditation and Quality Assurance 2007;12:563-7.

Linsinger T, Andrzejuk W, Bau A, Charoud-Got J, de Vos P, Emteborg H, Hearn R et al. Production of three certified reference materials for the sulfur content in gasoline (petrol). Energy Fuels 2007;21:2240-4.

Mackay LG, Burke D, Lui F-H, Sousou N, Vamathevan VV, Cuthbertson J, Mussell C, Myors RB. A high-accuracy method of analysis of 19-norandrosterone in human urine as utilised for the international laboratory intercomparison CCQM-P68. Accreditation and Quality Assurance 2007;12:475-82.

Mackay L, Burke D, Welch M, Tai S, Mussell C, O'Connor G et al. An international intercomparison for 19-norandrosterone in human urine: the Comité Consultatif pour la Quantité de Matière (CCQM) Pilot Study CCQM-P68. Accreditation and Quality Assurance 2007;12:459-64.

Publications

Mehta S, Keer JT. Performance characteristics of host-cell DNA quantification methods. BioProcess International 2007;5:44-58.

Mussell C, Wolff-Briche CSJ, Hopley CJ, O'Connor G. Analysis of 19-norandrosterone in human urine by gas-chromatography-isotope-dilution mass spectrometry: method adopted by LGC for participation in the Comité Consultatif pour la Quantité de Matière (CCQM) Pilot Study P68. Accreditation and Quality Assurance 2007;12:469-74.

Pang S, Qureshi F, Shanahan D, Harris N. Investigation of the use of rolling circle amplification for the detection of GM food. European Food Research and Technology 2007;225:59-66.

Rogival D, Van Campenhout K, Goenaga Infante H, Hearn R et al. Induction and metal speciation of metallothionein in wood mice (*Apodemus sylvaticus*) along a metal pollution gradient. Environmental Toxicology and Chemistry 2007;26:506-14.

Shanahan D, Stokes P, Burns MJ, Harris N. A novel method for the detection of Roundup Ready soya using liquid chromatography-electrospray ionization mass spectrometry. European Food Research and Technology 2007;225:483-91.

Trapmann S, Burns MJ, Broll H, MacArthur R, Wood R, Zel J. Guidance document on measurement uncertainty for GMO testing laboratories. European Commission Directorate-General Joint Research Centre Institute for Reference Materials and Measurements, reference EUR 22756 EN, 2007.

Turner J. 7500ce ICP-MS: Isotope dilution applications using custom databases. Agilent ICP-MS Journal 2007;30:6.

Turner J, Evans L, Hearn R, Taylor A. The use of collision/reaction cell ICP-MS with isotope dilution to provide assigned values for Zn, Cu and Se in serum samples for a proficiency testing scheme. Agilent Application Note 5989-7429EN, 2007.

Vickers PJ, Braybrook J, Lawrance P, Gray K. Detecting tartrate additives in foods: evaluating the use of capillary electrophoresis. Journal of Food Composition and Analysis 2007;20:252-6.

Walker M, Elahi S. IFST President chairs successful Government Chemist's dissemination seminar. Journal of the Institute of Food Science and Technology 2007;21:14.

Walker M, Elahi S. The facts never lie. Chartered Institute of Environmental Health, Environmental Health Practitioner, 6 July 2007.

Warburton E, Goenaga Infante H. Methane mixed plasma - improved sensitivity of inductively coupled plasma mass spectrometry detection for selenium speciation analysis of wheat-based food. Journal of Analytical Atomic Spectrometry 2007;22:370-6.

Warburton E, Norris P, Goenaga Infante H. Comparison of the capabilities of accelerated solvent extraction and sonication as extraction techniques for the quantification of kavalactones in *Piper methysticum* (Kava) roots by high performance liquid chromatography with ultra violet detection. Phytochemical Analysis 2007;18:98-102.

Wilkes T, Laux H, Foy CA. Microarray data quality - a review of current developments. OMICS 2007;11:1-13.

Zeine C. Impurities in drug products. Pharmazeutische Industrie 2007;69:624-8.

Glossary

APA	Association of Public Analysts
biomarker	substance used as an indicator of the identity, exposure history or health status of a biological system
bioprocessing	use of micro-organisms, biological cells or their components to produce substances such as pharmaceuticals and speciality chemicals
bioreactor	vessel in which a biological system produces useful substances or degrades harmful ones
capillary electrophoresis	family of high performance techniques that use narrow-bore fused-silica capillaries to separate charged substances in an electric field
CCQM	Consultative Committee for Amount of Substance (international body for metrology in chemistry)
CEN	Comité Européen de Normalisation (European Committee for Standardization)
CIKTN	Chemistry Innovation Knowledge Transfer Network
Defra	Department for Environment, Food and Rural Affairs
deuterium	heavier form (isotope) of hydrogen, clearly distinguishable by mass spectrometry
DIUS	Department for Innovation, Universities and Skills
ECHA	European Chemicals Agency
ELISA	enzyme-linked immunosorbent assay (a type of immunoassay)
elution gradient	continuous, progressive change in the composition of the fluid pumped onto a chromatographic column
FAPAS	Food Analysis Performance Assessment Scheme
flow cytometry	counting and characterisation of individual cells or particles which are presented to the measuring instrument in a stream of fluid
FSA	Food Standards Agency
FTICRMS	Fourier transform ion cyclotron resonance mass spectrometry
GC	Government Chemist
GC-MS	gas chromatography-mass spectrometry
gene expression	production of a characteristic biomolecule (RNA) from the genomic sequence, which may be followed by translation into specific proteins
genome	one full set of an organism's DNA, containing one copy of each gene
HMRC	Her Majesty's Revenue & Customs
hydrometer	compares the density of a liquid, such as an alcoholic drink, with that of water, based on the depth at which the instrument floats (displacement principle)
ICP-MS	inductively coupled plasma mass spectrometry - used for elemental analysis
immunoaffinity clean-up	isolates a substance by means of its reaction with antibodies
immunoassay	measures or detects a substance by means of its reaction with antibodies
LACORS	Local Authorities Coordinators of Regulatory Services

Glossary

LC	liquid chromatography
LC-fluorescence	substances are separated by LC, then determined by the fluorescence they emit while exposed to light of a suitable wavelength
LC-MS/MS	liquid chromatography-tandem mass spectrometry
ligand	in bioscience, a substance, whether naturally present or deliberately introduced, that interacts with a biomolecule to produce a biological effect
MALDI	matrix-assisted laser desorption/ionisation: a sample is mixed with a chemical matrix, from which its molecules are gently desorbed and ionised by a pulsed laser. The technique is suitable for introducing large, fragile molecules into a mass spectrometer
MALDI-ToF/ToF	mass spectrometer with a MALDI source and two ToF stages, allowing ion fragmentation for the study of complex samples
MHRA	Medicines and Healthcare products Regulatory Agency
microarray	compact array of biomolecular probes - typically either DNA or protein - which can be used to acquire data simultaneously on the composition of a sample
MS	mass spectrometry
multicollector ICP-MS	ICP-MS with an array of detectors that allows simultaneous detection of different elements and isotopes, thus performing ratio measurements at high precision
NEL	National Engineering Laboratory
NMI	National Measurement Institute
NMS	UK National Measurement System
NPL	National Physical Laboratory
NWML	National Weights and Measures Laboratory
OSI	former Office of Science and Innovation
PCR	polymerase chain reaction, a technique used to amplify DNA sequences so that they can be analysed
plasmid	small, double-stranded, self-replicating circle of DNA; occurring in nature, they may be tailored or built up artificially for bioscience applications
primary structure	chemical structure of a protein when viewed as a linear molecule
proteomics	study of the protein complement of cells, emphasising interactions and overall organisation
Public Analyst	analytical scientist appointed under statute by UK local authorities to provide scientific advice for the enforcement of many Acts of Parliament
QToF	quadrupole time-of-flight mass spectrometer
QUID	quantitative ingredient declarations
REACH	Regulation (EC) No 1907/2006 Concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals
real-time PCR	determination of DNA sequences as they are generated by PCR amplification

Glossary

referee analysis	impartial analysis by the GC to help resolve disputes relating to test results obtained on behalf of two independent parties
referee function	duty of the GC under Acts of Parliament to provide impartial analysis in the resolution of disputes relating to the enforcement of regulation
RSC	Royal Society of Chemistry
SME	small and medium-sized enterprise
tandem mass spectrometry	use of linked mass spectrometers; molecules of interest can be broken up after the first stage to allow more detailed characterisation by analysing their fragments in the second
titration	measurement of a dissolved substance by adding known volumes of a liquid reagent of known strength until the end point of a known reaction can be observed, e.g. as a colour change
titrimetric	relating to measurement by titration
ToF	time of flight: the time taken for particles such as ions to travel a fixed distance is measured, and used to identify them
toxicogenomics	investigation of the toxic mechanism and potency of substances based on the way they affect the activity of genomic DNA
TSB	Technology Strategy Board
USP	United States Pharmacopeia
volumetric	relating to measurement by volume
WFD-UKTAG	UK Technical Advisory Group supporting implementation of the Water Framework Directive (2000/60/EC)

Glossary