Department for Business Innovation & Skills

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Government Chemist

Review 2015


“The increasing complexity of modern analytical measurements requires constant innovation”

Contents

Foreword

I am pleased to introduce this review of the referee, capability building and dissemination work of the Government Chemist function in 2015. This year was a very busy one for us in the resolution of measurement disputes relating to food law, where the number and, in particular, the complexity of cases created significant challenges for our scientific teams.

Complexity arises from both the evolving regulatory landscape and the diversity and multitude of ingredients that are now routinely used in processed products. For example, the challenges of interpreting the regulations dealing with the potential for jelly based confectionary to cause a choking hazard in young infants is described on page 23 of the review, and illustrates how the use of undefined terms such as “firm” can lead to differences in opinion around product compliance. Also, the changes introduced late in 2014 relating to the declaration requirements for the presence of allergens at the point of sale led to increased industry and enforcement focus in this space, and consequently to several disputes involving the potential presence of trace quantities of almonds in spices. These referee cases called on the full range of our capability in the identification of species by both molecular biology and protein mass spectrometry. As with all of our referee work, these capabilities needed to be deployed to forensic standards given the legal implications of our findings. Indeed this important link between forensic/measurement science and compliance across multiple markets was further outlined in Sir Mark Walport’s Government Chief Scientist Annual Report entitled “Forensic Science and Beyond: Authenticity, Provenance and Assurance” that was published late in 2015.

Our ability to respond to complex regulatory issues is supported by the capability that is developed through the Government Chemist programme and augmented by LGC’s role as a Designated Measurement Institute for chemical and bio-measurement. This includes technical expertise in areas where regulation is likely to emerge, such as nanomaterials in food. Predicting future referee requirements is never easy and again this year I am highly appreciative of the guidance I have received in this respect from the Government Chemist Programme Expert Group which includes contributors from academia, Government, enforcement laboratories, trading standards and industry.

In a year full of technical challenges, I would like to thank all those responsible for delivering our work particularly our food chemists and molecular biologists, led by Kirstin Gray and Malcolm Burns respectively. I would also like to acknowledge the considerable input of Michael Walker who oversees all of the referee cases. This report forms part of our advisory and dissemination activities which includes publication of our research, articles on our website, responding to scientific consultations, providing technical input on committees and running knowledge transfer meetings. Over the past 5 years many of these outputs have been organised or delivered by Nick Boley who will retire in 2016. I thank Nick for his contribution to the work of the Government Chemist function and I hope you enjoy reading his final annual report. As ever I welcome your feedback.

[1] https://www.gov.uk/government/uploads/system/uploads/attachment_data/file/485640/gs-15-37a-forensic-science-beyond-report.pdf

Note from the Government Chemist Programme Expert Group

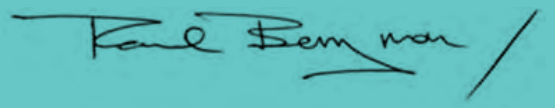
I am delighted to contribute to the 2015 Government Chemist review as Chair of the Government Chemist Programme Expert Group (GCPEG).

One of the primary functions of the GCPEG is the governance of the Government Chemist programme. The Expert Group comprises key stakeholders including regulatory and policy officials, representatives from industry, public analysts, port health authority officials, and academics. The GCPEG meets twice a year to provide independent scrutiny of referee casework, research and advice given by the Government Chemist and also reviews the quarterly progress reports.

The increasing complexity of modern measurement is a key challenge for the Government Chemist and his team. The expectation from regulators, industry and the public is that it is possible to carry out any measurement necessary to authenticate food products and detect any form of adulteration. This puts significant pressure on those responsible for developing and performing such measurements. It is clear that the Government Chemist and his team have responded magnificently to these challenges with the work carried out over the past year. The referee case involving mahaleb adulteration of cumin is an excellent example (page 20).

Work carried out under the Government Chemist programme has shown that it is possible to rise to almost any measurement challenge the modern world can throw at it. However a key challenge is to strike a balance between expenditure on state-of-the-art techniques versus the risk associated with an inconclusive result. This review demonstrates the excellent science carried out to address such measurement challenges and the confidence that gives us all in the way it underpins UK food law.

It is clear from the topics covered in this review, that the Government Chemist and his team display a high level of professionalism and skill to cover the broad range of referee cases, requests for advice and research projects that characterise the programme. I am sure all stakeholders will both enjoy and value the review.



Professor Paul Berryman
BSc, MChemA, PhD, MBA, FRSC, CSci
Chair, Government Chemist Programme Expert Group

1 Remit

The Government Chemist fulfils two functions: (a) statutory and (b) advisory, funded by the Department of Business, Innovation and Skills (BIS). Created in 1842 to help safeguard government revenue the laboratory assumed a technical appeal function in 1875 (referee analysis) linked to the food and drug Act of that year. That role continues to this day.

The Government Chemist has always striven to apply up-to-date and authoritative measurement procedures coupled with interpretative skills to act as a fair and independent arbiter to resolve disputes, to provide public protection and to contribute to effective and efficient regulatory enforcement in industrial sectors where chemical measurements are important. The need to develop measurement techniques and procedures both within our own laboratories and in collaboration with other expert organisations continues to exist. This will enable the Government Chemist to be able to respond to potential future issues as and when they arise.

Statutory function

The Government Chemist has a statutory function comprising science-based duties prescribed in seven acts of Parliament. These duties (see Box 1 on page 8) cover public protection, safety, health, value for money, and consumer choice. The resolution of scientific disputes is a cornerstone of our activities, the most important aspect of what we do, and is usually called 'referee analysis'. Our role in the resolution of disputes between regulators and businesses and is based on our independent measurements, and expert opinions. Success here often avoids recourse to legal process which reduces the burden on public finances. Many of these cases can have a significant impact on either or both parties, as well as far-reaching consequences. Credibility of the referee rests on first-class science, which is underpinned by the assignment of our home laboratory, LGC, as the the UK's designated national measurement institute (*Designated Institute*) for chemical and bio-measurement. We believe our pioneering work on allergens in spices in 2015 is a perfect exemplar of this.

Legislation covering the food, agriculture and medicinal products sectors, where the safety and protection of the consumer is of prime importance, contains equivalent provisions for the taking of official samples and subsequent analysis.

There are several routes for referral to the Government Chemist, described in more detail on page 11. For businesses, a successful appeal to the Government Chemist may avoid the effects of penalties prescribed under criminal law, potentially expensive compliance actions and, most seriously, loss of reputation and goodwill. The referral sometimes comes from the court itself, with proceedings suspended pending the outcome. When the Government Chemist's findings confirm those of the enforcement authority, the appropriate action to protect the public can, of course, proceed with increased authority. But, regardless of the outcome, the scientific outputs of the case can be disseminated to all parties and the lessons of these can hopefully be taken on board which should help reduce the possibility of recurrence.

Dissemination of referee cases also takes place through scientific publications, seminars, workshops, training events and via our website: https://www.gov.uk/government/organisations/government-chemist.

► **Section 2 of this review looks at the year's completed referee cases.**

The need for referee analysis is frequently the greatest in areas where measurements are difficult, and new methods need to be developed and/or validated. Some cases are in areas of high public or media salience, or where novel products are being introduced into the market. The Government Chemist carries out research and development (R&D) in the form of capability building projects based on horizon scanning which identifies the areas where this is most likely to occur. However, these cannot predict every possible referee case, and method development is still necessary on an ad hoc basis. The outputs of these studies are disseminated publicly; in particular, to stakeholders in the analytical community thus reducing the need for referral to the Government Chemist.

► **See Section 3 for an overview of R&D activities.**

Advisory function

The Laboratory of the Government Chemist was originally founded in 1842 with the remit to detect adulteration of tobacco on behalf of HM Customs & Excise. It continued to develop after this time to become established for nearly half the 20th Century as a free-standing central department with a broad responsibility for the investigation and analysis of a wide range of samples and problems on behalf of other government departments and authorities.

On privatisation in 1996, LGC signed an agreement with the Secretary of State for Trade and Industry which underpinned the continuity of the broader public functions by appointing the Government Chemist 'as a source of advice for HM Government and the wider analytical community on the analytical chemistry implications on matters of policy and of standards and of regulations'. This agreement continues to this day and serves to highlight the importance of chemical and biochemical measurements in underpinning the UK economy. As new technologies are developed and become more widely and routinely used, the need for such advice to be given is even greater to ensure that this happens in an appropriate manner.

The principal means of delivery of the advisory function is in the response to government calls for advice or published consultations, where there is a significant or important analytical science content. These responses provide relevant information specifically to the department, agency, European Commission Directorate-General or other public body publishing the consultation, as well as to a broad range of stakeholders who have an interest in regulatory compliance and the associated measurement aspects of this. Consultation responses are published on the Government Chemist web page, on GOV.UK. The advisory function also looks at emerging issues involving new, updated or planned regulation and related analytical measurements and addresses these by means of small targeted projects and publications, by publication through the Government Chemist blog[2] or, latterly, through the publication of stories on the Government Chemist web page[3].

► **See Section 3 for more about the wider advisory function.**

Governance

The Department for Business, Innovation and Skills (BIS) funds a programme to enable delivery of statutory casework, scientific advice and any work and research necessary for the ongoing effectiveness of the Government Chemist's functions. Within BIS, responsibility for both the Government Chemist and the wider UK National Measurement System formerly rested with the National Measurement Office (NMO), but that responsibility now lies with the Knowledge and Information Team within BIS.

BIS have put into place arrangements to ensure that the Government Chemist programme is delivered competently, and that scientific standards, impartiality, transparency and integrity are maintained. LGC has rigorous structures and procedures in place to ensure no conflicts of interest arise between work carried out under the statutory function and its commercial food analysis activities. These were further strengthened by some structural changes to LGC's operational divisions in 2014 and the system has been demonstrated to work well.

The Government Chemist Programme Expert Group (GCPEG), formerly the Government Chemist Working Group (GCWG), plays a key role in the governance of the Government Chemist programme, providing the necessary independent scrutiny of the programme. The GCPEG also offers advice to BIS regarding future priorities, which feeds into the programme formulation process. It meets twice a year to oversee and discuss the progress and quality of work delivered under the programme. The GCPEG is tasked by BIS to advise on:

- The effectiveness and impact of the programme in providing an independent, expert service to resolve disputes between food control authorities and food traders on analytical results and their interpretation
- The requirement for medium to long term Government Chemist capability building work aimed at preventing disputes arising and enabling better response to referee cases
- The progress of the current projects in meeting technical milestones and targets; and
- The formulation and prioritisation of new projects to maintain and develop the capabilities needed to discharge the GC functions (i.e. capability building, knowledge transfer, regulatory foresight and statutory analysis).

The GCPEG comprises representatives of regulatory and enforcement bodies, industry, trade associations and academia, with a broad range of backgrounds, skills and interests.

[2] http://governmentchemist.wordpress.com/
[3] https://www.gov.uk/government/organisations/government-chemist

Details of the membership of the Government Chemist Programme Expert Group are given below:

Paul Berryman

Chair of Government Chemist Programme Expert Group

Paul is owner and director of Berryman Food Science Ltd, which works closely with government and business, including with FERA and the FSA. Paul has an extensive career spanning more than 30 years in which he has worked at senior level with most of the top 100 global food companies.

Robbie Beattie

Robbie is appointed as Public Analyst, Agricultural Analyst and Food Examiner to nine local authorities in Scotland. He leads 44 laboratory staff who test a range of samples including food, water, asbestos, consumer products and environmental samples. He also leads an Environmental Assessment team. He has had a varied career spanning a range of businesses and organisations including Royal Ordinance Factory, Scottish & Newcastle Breweries, and Medicines Testing Laboratory.

Simon Branch

Simon joined RHM Technology as a Senior Analytical Chemist in 1990, where he progressed through a number of roles to become Head of Innovation and Improvement, before moving to the McCormick Corporation where he took responsibility for the Product and Process Development teams. In 2014, he moved to Goldenfry as Head of Innovation. During his career, Simon has sat on a number of committees including the RSC LGC Advisory Committee and the RSC Science and Technology Board.

Andrew Damant

Andrew leads the Scientific Methods and Laboratory Policy Team at the Food Standards Agency and is responsible for Agency policy on UK national reference laboratories and official control laboratories. Andrew is an official UK delegate on numerous international committees and also acts as advisor to various UK committees.

Lucy Foster

Lucy began her career as a government scientist at the Ministry of Agriculture, Fisheries and Food in 1998. She joined the Food Standards Agency in 2000 before moving to the Department for Food and Rural Affairs in 2009 where she is a science lead on food and farming. Lucy has considerable experience in food safety from a science and a policy perspective, including microbiological foodborne disease, food hygiene, food additives and food compositional and labelling standards.

Jonathan Griffin

Jonathan began his career as a Graduate Scientist at Kent County Council, where he carried out classical and instrumental analysis of foods, agricultural samples, water and consumer goods. He completed the Mastership of Chemical Analysis (MChemA) in 2002 and became a Public Analyst. He continues to work as Public Analyst and Technical Manager for Kent Scientific Services.

Martin Hall

Martin is the Director of Science at Campden BRI and has overall responsibility for the departments of Chemistry and Biochemistry, Microbiology, Consumer & Sensory Science and Statistics. Martin has 40 years' experience of a wide range of food related subjects with specific interests in food safety and quality, authenticity and analytical techniques.

Declan Naughton

Declan joined the Inflammation Research Group at Barts and The London School of Medicine and Dentistry, where he spent 10 years before accepting posts at Bath University and the University of Brighton. He is currently Professor of Biomolecular Sciences at Kingston University London. His research interests span food safety; nutrition; natural products; performance enhancing drugs; inflammation; drug discovery; and endocrinology.

Linda Plested

Linda started her career in food science working for the Milk Marketing Board, before joining the Watney Mann and Truman Brewers network where she undertook analytical and project work. In 2001, she became a Trading Standards Officer (TSO) for Surrey County Council Trading Standards. Linda retired in 2015 and was replaced by David Pickering, TSO for Buckinhamshire and Surrey Trading Standards. We would like to thank her for her significant contributions to the PEG over the years.

Roger Wood

Roger is an experienced food analysis specialist, who recently retired from the UK's Food Standards Agency. He holds the Mastership in Chemical Analysis, (MChemA), the statutory qualification required to practice as a Public Analyst. He has represented the UK at numerous EU methods of analysis and sampling Working Groups in the food and feed sectors over the past 35 years.

Kirsty Dawes

Kirsty is an imported food specialist, working for Suffolk Coastal Port Health Authority, based at the Port of Felixstowe. Kirsty is an Environmental Health Practitioner with a BSc in Environmental Health, and one of the few non-chemists represented on the group.

David Ferguson

David spent the first half of his career with BP Research before operating as an independent consultant for clients in the industrial, public and charity sectors in the analytical chemistry arena. During this time he worked for government as the Independent Advisor for the Government Chemist Function. He is currently semi-retired and looks after the affairs of the Analytical Chemistry Trust Fund.

John Figgins

John is a Technical Specialist for Food at BRC Global Standards.

The current Government Chemist programme

The current Government Chemist programme, covering 2014-2017, commenced in April 2014, and concludes in March 2017. The programme continues to reflect the prioritisation exercise carried out by the GC Programme Expert Group, and is similar in structure and themes to the 2011-2014 programme:

- Intelligence gathering: horizon scanning projects on the scientific implications of policy development, emerging legislation, changes to existing legislation and enforcement trends
- Capability building: innovative and relevant R&D which aims to reflect potential needs for future casework under the Government Chemist's statutory role
- Statutory activities: work carried out in relation to individual cases that are referred to the Government Chemist under his statutory function as defined in Acts of Parliament
- Knowledge transfer: improved dissemination of regulatory and analytical developments to a wide range of stakeholders, to stimulate improvement of standards of measurements, the understanding of the regulatory environment and to help industry to innovate concerning new products and processes.

This is the second Annual Review of the current GC programme. The main aim of the programme continues to focus on the statutory referee analyst function, which requires no further introduction, but there are a number of other activities and projects that are also part of the programme which support and enable the Government Chemist to discharge this function properly and effectively.

An important aspect of the programme is to ensure we are well equipped and prepared to deal with future referee cases. To this end, the programme contains a series of laboratory-based projects that build and extend our technical capabilities. These were developed through stakeholder consultation and prioritised by the GCPEG and thus take into account the perceived future direction of measurement testing in the food industry. These include projects to improve the detection of food allergens (using DNA, ELISA and mass spectrometry technologies), to improve authenticity testing of meat and fish and the detection of GMO foods (using DNA-based methods), and, for mycotoxin detection (using mass spectrometry approaches). However, the plans for delivering these projects can sometimes be overtaken by events, as will become apparent later in this Review, and the use of our breadth of expertise to develop methodology in support specific cases under the referee function takes precedence.

Other laboratory-based components of the programme ensure we maintain high standards of quality and performance in our established capabilities through participation in Proficiency Testing schemes. In addition, we also deliver some small-scale laboratory studies to apply our technical expertise to address measurement challenges faced through the introduction of new legislation for the food, agriculture and chemical industries (e.g. identification of illegally-logged timber, and differentiation of silver nanoparticles from ionic silver in effluent waters).

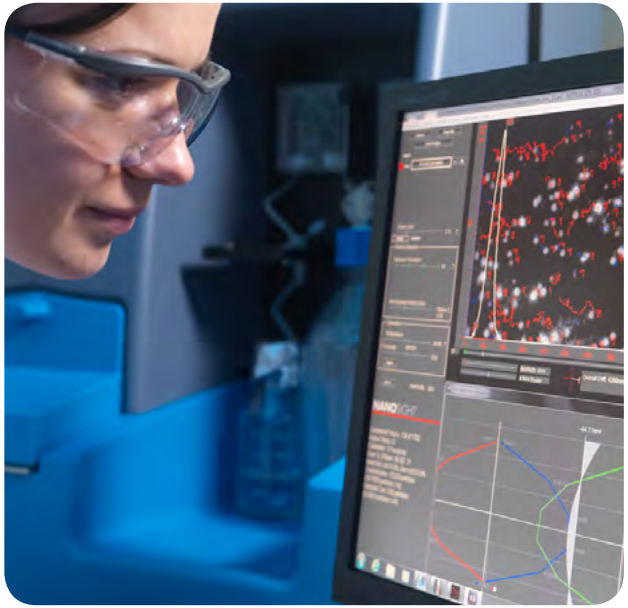
The final element of the programme ensures our relevance and impact to UK stakeholders. We undertake a broad range of horizon scanning and stakeholder engagement activities which keep us abreast of regulatory and technology developments, whilst importantly, we ensure programme outputs and advice are clearly communicated to the UK scientific audience and the Government, which includes training events, presentations at conferences and meetings, seminars and publication of articles and reports via the GC website.

People

LGC staff who directly support the Government Chemist function have clearly and independently defined roles (Figure 1). Within this framework, there are particular requirements for the management of statutory casework:

- Nominated officers, one of whom holds the requisite statutory qualification[4], have overall responsibility for case supervision, They prepare and sign government chemist certificates of analysis
- Only the Government Chemist or Deputy, once satisfied that the case has been properly completed, may countersign.

The members of staff carrying out work under the Government Chemist's statutory function must continually demonstrate their competence through participation in an extensive variety of appropriate proficiency testing schemes and collaborative studies. The diverse nature of LGC's scientific activities therefore leads to a wide range of skills and specialisms being available in-house. Many of the staff involved in delivering the programme have also carried out research and development work, which often involves programmes with international collaboration, which gives them the capability to contribute positively and efficiently to their work.



[4] All work is overseen by Michael Walker, a nominated officer holding the statutory MChemA qualification

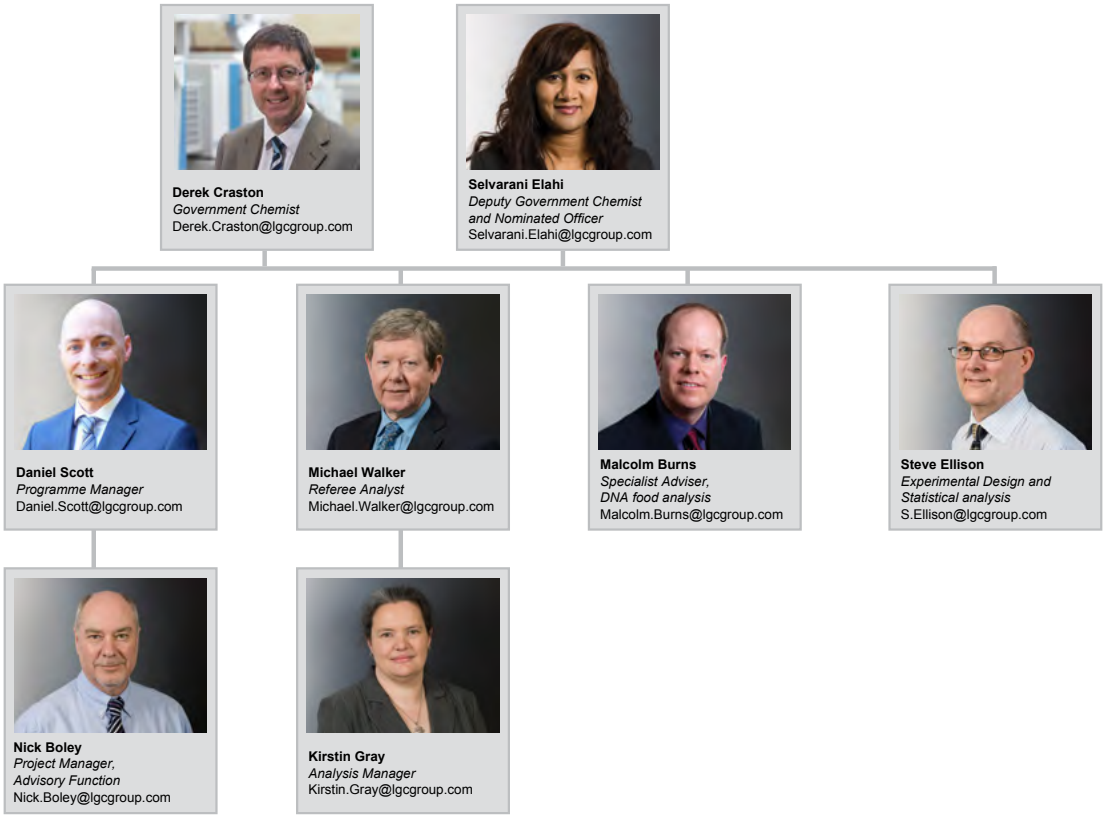
Collaboration

The Government Chemist's remit covers a very wide area of measurement science, which contains a significant number of potential challenges, not all of which can be predicted from our horizon scanning activities. Some of these challenges may lie outside our sphere of specific expertise, and the expertise or equipment needed to address them may not be readily available within the broad range of activities undertaken within LGC.

We therefore need to be mindful of the scope for collaboration with a range of potential stakeholders, who are able to complement our own expertise and activities, in order to ensure the Government Chemist function can be comprehensively discharged. This will enable our capability building research and development work to be responsive and carry the appropriate authority within the scientific measurement community. Therefore we can continue to benefit public health, safety and well-being, as well as the wider scientific community, including those UK manufacturing industries which depend on reliable and accurate analytical measurement, and how they interact with regulation.

If you would like to get involved with any aspect of our work, or for more information on our work, please contact us at Government.Chemist@lgcgroup.com or go to the website https://www.gov.uk/government/organisations/government-chemist.

Figure 1 Government Chemist organogram and contact points.



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Box 1: The Government Chemist in legislation

The duties of the Government Chemist as referee analyst are defined in or under:

Food Safety Act 1990
Food Safety (Sampling and Qualifications) Regulations 2013
Food Safety (Sampling and Qualifications) (Scotland) Regulations 2013
Food (Northern Ireland) Order 1989
Food Safety (Northern Ireland) Order 1991
Food Safety (Sampling and Qualifications) Regulations (Northern Ireland) 2013
Poultry Meat (Water Content) Regulations 1984
Natural Mineral Water, Spring Water and Bottled Drinking Water Regulations 2007[1]
Materials and Articles in Contact with Food Regulations 2012[1]
Agriculture Act 1970
The Animal Feed (Hygiene, Sampling etc. and Enforcement) (England) Regulations 20151
Genetically Modified Animal Feed Regulations 20041
Human Medicines Regulations 2012
Farm and Garden Chemicals Act 1967

The Government Chemist 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 Government Chemist are understood with reference to:

Freedom of Information Act 2000
Scotland Act 1998 (Cross-Border Public Authorities) (Specification) Order 1999
Administrative Provisions Act (Northern Ireland) 1928

[1] Enacted as separate legislation in England, Northern Ireland, Scotland and Wales

2 Science underpinning sound dispute resolution

Referee casework arises most frequently under the Food Safety Act 1990 or the Agriculture Act 1970.

Formal samples taken under statutory enforcement provisions are divided into parts for analysis on behalf of the authorities, the food and feed business operator (FBO) and, when required, the referee. During 2015, 14 cases were referred to the Government Chemist, three of which remained in progress at the time of writing and all in connection with food (see Table 1).

The Referee Function

The problems that were referred to us in 2015 included novel investigations, familiar issues and re-emerging questions. The most challenging investigations involved alleged allergens in spices, for which we had to develop completely new methods of analysis; there are more details on page 20. Familiar issues included aflatoxins, naturally occurring cancer causing contaminants; and there were also issues to resolve relating to pesticides residues, food authenticity, and residues of veterinary medicines. Two issues resurfaced after gaps of several years; illegal dyes and the choking hazards of jelly mini-cups, a fuller account of the latter is on page 21. Unusually, part way through an investigation prompted by a trader's concerns about a food labelling prosecution, the trader asked us to abandon the case. Most of the 14 cases involved multiple samples.

Table 1 Overview of Referee Cases in 2015

Origin			Basis		
Inland Authority	5	36 %	Dispute	8	57 %
Port Health Authority	6	43 %	Other*	6	43 %
Central Competent Authority (FSA)	3	21 %			

*Other includes SEO – Supplementary Expert Opinion, pursuant to Article 11(5) of Regulation 882/2004 on official controls and requests for assistance from other Government Departments or Local Authorities.

Referee casework is a demand led service which has been at the core of the Government Chemist's function since 1875. Demand reduced slightly in 2015 (see Figure 2) but increased in complexity. In guaranteeing fair scientific treatment for all by authoritative adjudication on disputes we underpin public and industry confidence in the food and feed official control system. We maintain the even-handed credibility of this referee role by stringent governance of the function and painstaking analytical rigour. Our aim is to safeguard consumers, regulators, the agrifood sector and the courts from unwitting errors in measurement science.

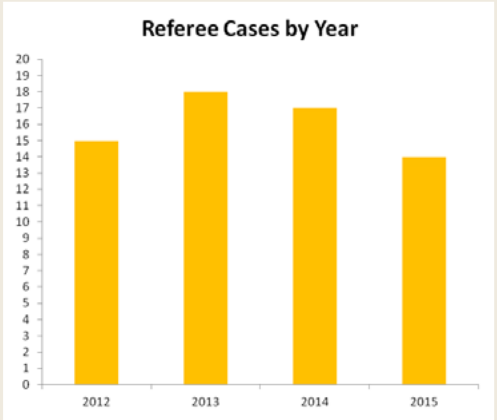


Figure 2 Referee Cases by year

There is no legal definition of the referee analyst function; however we regard it as independent expert analysis and / or interpretation to help avoid or resolve disputes. There are statutory provisions for referral of retained portions of formal [5] samples to the Government Chemist in regulations made under both the Food Safety Act 1990 and the Agriculture Act 1970.[6] The statutory conditions for referral begin with the contemplation or commencement of legal proceedings where the prosecution intends to adduce analytical evidence. The referral may be by the local authority authorised sampling officer, the prosecutor or the court. The defendant may also, subject to agreement to defray some or all of the Government Chemist's costs, request referral. It would be rare and, in fact, has never happened that the Government Chemist would require a defendant or trader to cover all the costs of a referee case, which can be substantial. If the above route is not open to a trader they may request a supplementary expert opinion (SEO) pursuant to Article 11(5) of Regulation 882/2004 on *official controls* and in defined circumstances SEO may be requested of the Government Chemist. The Government Chemist also acts as a source of advice for government and the wider analytical community on the analytical chemical implications on matters of policy, standards and regulations. Hence referee casework arises by a variety of routes and 2015 saw increased recourse to the Government Chemist as an advisor to both central and local government.

Analytical results must be interpreted in increasingly complex scientific legal and policy contexts, and in an increasingly global supply chain. When a referral is received we begin with a case meeting to examine the problems associated with the case and often instigate a literature review of the topic. Few referee cases are routine nowadays and often our analytical methods must be newly devised, implemented or modified to deal with particular problems.

Our default analytical strategy is multi-replicate analyses on multiple days. The extent of replication together with analysis of reference materials, (certified, where available), and of blanks and spiked blanks and/or sample aliquots, amounting to many of the elements of a stand-alone method validation, provide the necessary high level of analytical confidence. All significant analytical steps are witnessed by a second scientist, all data transcriptions are checked and the results are evaluated against prescribed quality criteria. The entire dataset is independently evaluated by professional statisticians for bias and outlying results and to yield a case specific measurement uncertainty if required. A certificate is drafted and reviewed by a qualified person and finally the case file is brought to the Government Chemist for peer review. If all steps are satisfactory the Government Chemist will allow the findings to be released.

Veterinary medicine residues

Veterinary medicines are used to treat sick animals or prevent disease in herds or flocks. All veterinary medicines must be authorised before they can be marketed or used on animals. Veterinary medicine residues in the food chain, including in imported food, are monitored through surveillance programmes. Safety limits are set as part of the authorisation procedures to minimise risks to health. A safety-based legal limit (Maximum Residue Limit – MRL) is calculated which represents the maximum amount of the veterinary medicine that is safely and legally permitted in food.

In 2015 we were called in when an importer disputed official results for the concentration of the veterinary medicine 'albendazole' in corned beef. The Public Analyst had certified against the consignment for excess albendazole. A laboratory acting for the importer reported data below the MRL, including a finding of the parent drug which is not included in the residue definition.

Albendazole, one of the benzimidazole anthelmintics, is authorised for use in ruminants.[7] However, owing to reported teratogenicity, there are MRLs for various tissues, e.g. in muscle and fat of 100 micrograms per kilogram (µg/kg) as the sum of albendazole sulfoxide, albendazole sulfone, and albendazole 2-aminosulfone, expressed as albendazole. Albendazole is extensively metabolized in domestic animals and humans with rapid conversion to a sulfoxide and subsequently a sulfone metabolite. Sulfoxide metabolites are responsible for the systemic biological activity of benzimidazole drugs.

The case commenced with discussions on further sampling and we received a range of samples from the detained consignment, which consisted of product from two batch production dates, for referee analysis:

- Two portions of homogenised corned beef originally analysed by the laboratories previously involved
- Two unopened cans from the original sampling exercise and
- A further 40 unopened cans chosen at random from the consignment of which four cans were separately analysed.

Hence we analysed three previously unopened cans from each of the two batch production dates in the consignment.

[5] Formal samples taken under statutory enforcement provisions are divided into parts for analysis on behalf of the authorities, the food or feed business operator (FBO) and, when required, the Government Chemist.

[6] Boley, N. 2016, Annual Statement of Statutory Scope, available at https://www.gov.uk/government/publications/government-chemist-annual-statement-of-statutory-scope-2015--2

[7] Commission Regulation (EU) No 37/2010 of 22 December 2009 on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin, Consolidated version of 17.05.2015

The analysis consisted of acetonitrile extraction, liquid/liquid partitioning and solid phase extraction clean-up followed by liquid chromatography separation and tandem mass spectrometry detection and quantification (LC-MS/MS). Isotopically labelled albendazole-D3 and albendazole sulphoxide-D3 were used as internal standards. At least two precursor ion to product ion transitions were examined for each analyte and quantification was performed against appropriate calibration curves established by a series of pre-extraction matrix standards. Method performance was monitored including by assessing calibration linearity, appropriate transition ratios, post-extraction matrix spikes and for each positive sample a further extract was fortified to estimate overspike recovery. Having established results by reference to the isotopically labelled internal standard, the data were not further adjusted by reference to the overspike recovery. The analysis of three replicates on each of three days enabled the calculation of a case specific measurement uncertainty for each positive metabolite.

Our findings did not confirm the presence of the parent drug, as only the sulfoxide metabolite was detected. We confirmed an exceedance of the MRL in both samples originally analysed and also in two previously unopened cans of the product. One of these contained over 15 times the maximum permitted amount of residue.

It was open to the importer to request analysis (at their own expense) of the remaining 36 unopened cans that had been sampled, to further characterise the consignment. However, in the event the consignment was re-exported and did not enter the UK supply chain.

We met with officers of the port to discuss the sampling and analytical issues arising from the case and will also publish a conference paper on the topic at the EuroResidue VIII conference May 2016 in the Netherlands.

Pesticides residues

As with veterinary residues, all foodstuffs intended for human or animal consumption in the EU must conform to maximum residue levels (MRLs) for pesticides in order to protect animal and human health.[8] MRLs are in general recommended by the European Food Standards Agency (EFSA) based on a risk assessment and adopted in law by the Commission. Where a MRL has not been specifically set a 'default' MRL of 0.01 mg/kg is applied. Article 18 of Regulation (EC) No 396/2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin [9] prohibits products being placed on the market as food or feed if they contain a pesticide residue exceeding the prescribed MRL.

Early in 2015 a sample of imported organic green tea was reported by the Public Analyst to contain the pesticide hexachlorobenzene (HCB) above its MRL. The importer's analysis returned a result below the MRL and the retained portion was referred to us. A published QuEChERS-based method was identified [10], evaluated and deployed.

Three replicates of the sample were analysed on each of three days by acetonitrile extraction followed by gas chromatography mass spectrometry (GC-MS). Quantification was performed by standard additions including with isotopically labelled HCB at appropriate concentrations. We found the tea to contain almost three times the maximum permitted amount of the pesticide thus confirming the Public Analyst's findings and the product did not enter the UK supply chain. We also investigated the possibility that the hexachorobenzene originated in the teabag material rather than in the tea but were able to rule that out.

[8] Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC

[9] Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin, consolidated version of 02.02.2014 available at http://eur-lex.europa.eu/Notice.do?val=400559:cs&lang=en&list=400559:cs,&pos=1&page=1&nbl=1&pgs=10&hwords

[10] Tomas Cajka, Chris Sandy, Veronika Bachanova, Lucie Drabova, Kamila Kalachova, Jana Pulkrabova, Jana Hajslova, 2012, Streamlining sample preparation and gas chromatography–tandem mass spectrometry analysis of multiple pesticide residues in tea, Analytica Chimica Acta, 743, 51-60

Meat speciation

Focus on food authenticity remained high in 2015. The FSA funded 2014-15 National Food Sampling Programme[11] included an additional element of local authority testing of lamb dishes from takeaway restaurants for meat speciation (and where appropriate for allergens and additives). There were over 60 samples considered to be non-compliant when sampled by a local authority from the restaurant and its suppliers which needed following up. The Public Analyst reported one lamb sample as satisfactory, however a product described as goat meat was reported to contain only sheep DNA. Moreover the Public Analyst also reported a minced lamb product with a substantial amount of chicken DNA, a "cooked lamb curry" with only beef DNA and a sample described as "cooked minced lamb" was found to contain chicken DNA as well as sheep DNA. Proceedings were instigated in the Magistrate's Court and the defendant supplier entered a 'not guilty' plea. Anticipating a possible analytical defence the local authority requested a referee analysis of the retained portions of the samples.

We applied both ELISA (to check the protein) and real time PCR (to identify cell nucleus DNA) to multiple replicates of the samples. The "cooked lamb curry", consisted of seven pieces of cooked meat and some sauce. We tested multiple replicates of each piece of meat (and the sauce) individually and showed that the meat was beef and not sheep meat. The "goat meat", also consisted of seven pieces of raw meat and similar detailed analysis confirmed that the meat was sheep and not goat. We found the "cooked minced lamb" to consist of a mixture of chicken and sheep meat, and the "minced lamb", consisted of a mixture of sheep and chicken meat.

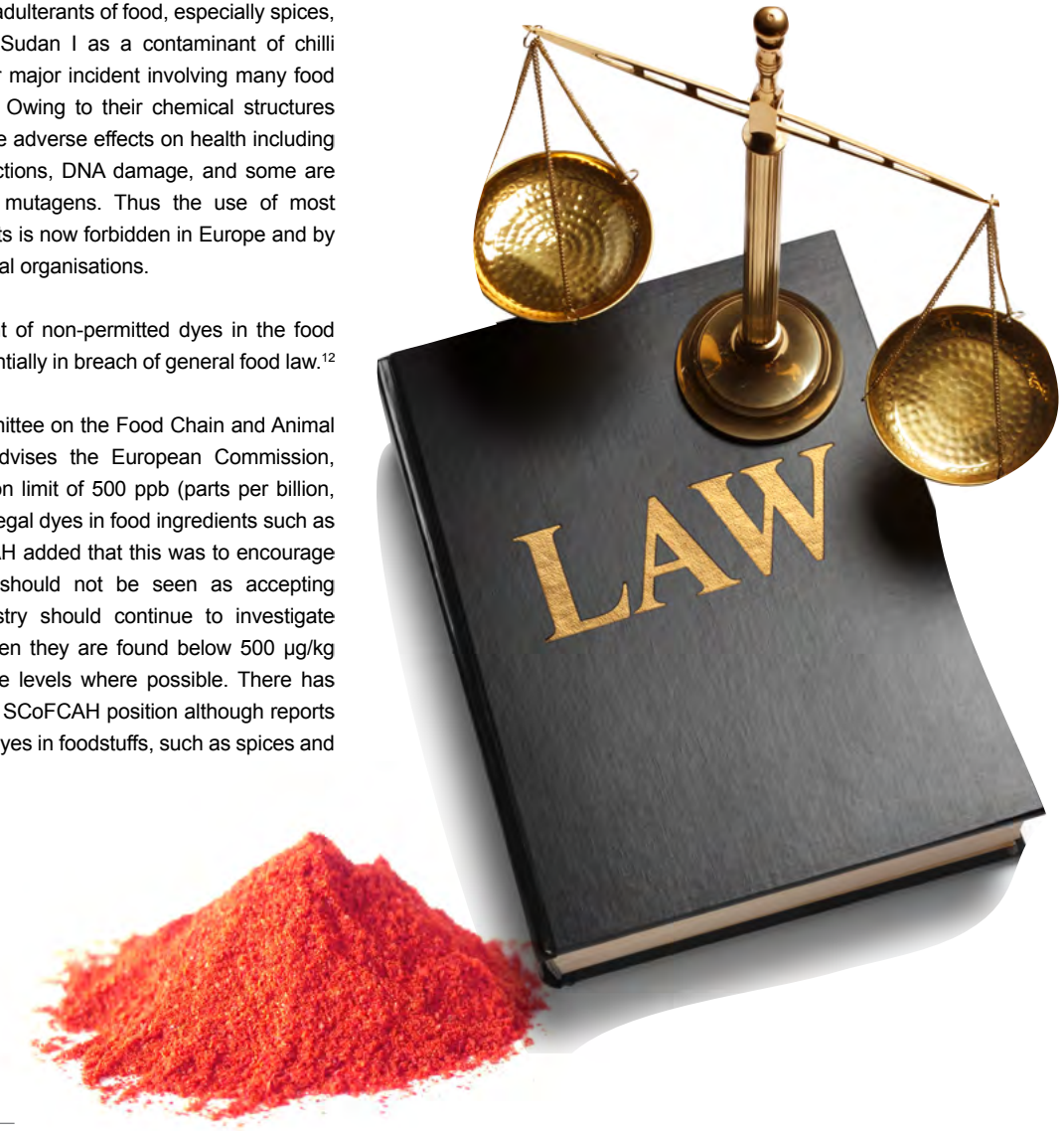
Hence we upheld all of the Public Analyst's findings and the defendant was found guilty and received a total penalty (fines and costs) of £7100.

Illegal Dyes

Synthetic dyes are notorious adulterants of food, especially spices, following the emergence of Sudan I as a contaminant of chilli powder in 2003 and a similar major incident involving many food products withdrawn in 2005. Owing to their chemical structures many synthetic dyes can have adverse effects on health including allergenic and asthmatic reactions, DNA damage, and some are suspected carcinogens and mutagens. Thus the use of most synthetic dyes in food products is now forbidden in Europe and by many national and international organisations.

Strictly speaking, any amount of non-permitted dyes in the food chain is undesirable and potentially in breach of general food law.[12]

However, the Standing Committee on the Food Chain and Animal Health, SCoFCAH, which advises the European Commission, advised in 2006 that an action limit of 500 ppb (parts per billion, µg/kg) should be applied to illegal dyes in food ingredients such as spices and palm oil. SCoFCAH added that this was to encourage a consistent approach but should not be seen as accepting adulteration. The food industry should continue to investigate sources of contamination when they are found below 500 µg/kg and take measures to reduce levels where possible. There has been no further update of the SCoFCAH position although reports of the occurrence of banned dyes in foodstuffs, such as spices and vegetable oils, persist.



[11] Food Standards Agency, 2015,Testing for the verification for the presence of undeclared meat species and allergens in lamb products from takeaway outlet, https://www.food.gov.uk/sites/default/files/lamb-takeaway-finalreport%20-Jan%202015v2.pdf (accessed 14.01.16)

[12] *Regulation (EC) 178/2002 of the European Parliament and of the Council of 28 January 2002 laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety*

In 2015 we were called upon to resolve a dispute involving findings of the illegal dye Sudan IV in palm oil. Both the Public Analyst and the importer's laboratory agreed that their samples contained Sudan IV, however the crux was how much. The Public Analyst reported slightly over and the importer's laboratory slightly under the action limit.

Our analysis consisted of acetonitrile:acetone extraction followed by liquid chromatography separation and tandem mass spectrometry detection and quantification (LC-MS/MS). Isotopically labelled Sudan IV D6 was used as an internal standard. Two precursor ion-to-product ion transitions were examined, baseline separation of Sudan IV and Sudan Red B was established and photochromic isomerism about the azo bond was eliminated by pH adjustment. Quantification was (a) by separate pre-extraction blank[13] matrix spike calibration curves and (b) by standard additions to the sample itself, in each case over appropriate concentration ranges. Method performance was monitored including by assessing calibration linearity, appropriate transition ratios, and post-extraction matrix spikes. We found Sudan IV at not less than 366 µg/kg and not more than 418 µg/kg in what appeared to be the retail bottle of palm oil we received. By examining a side panel of the bottle we concluded that Sudan IV was neither leaching out of, nor being absorbed by the container. However, we noted that the formal sample appeared to consist of three separate product bottles. Hence the results of the analysis of the referee sample, although definitive for the sample received, stand alongside those of the other parts of the sample but do not necessarily contradict them. We therefore advised the sampling officer to ask the Food Standards Agency for a risk assessment of the totality of the results for the consignment.

Aflatoxins

The remaining three cases involved aflatoxins. These are mycotoxins, secondary metabolites produced by fungi, mainly Aspergillus flavus and Aspergillus parasiticus in or on foods and animal feeding stuffs. Aflatoxins are genotoxic carcinogens capable of inducing liver cancer particularly with simultaneous hepatitis B virus infection and are among the most potent mutagens known. Stringent control measures are in place to reduce human consumption and disputes about concentrations of these toxins close to the legislative limits (low parts per billion) in imported consignments are a regular feature of referee casework. In all three cases, peanuts, figs and coated peanuts, we upheld the Public Analysts' findings and the consignments were prevented from entering the UK food chain. The work was not wholly routine. For example, in the latter case the wheat flour and coconut based coating of the peanuts formed a gel that appeared to alter the partition characteristics of the extraction solvent necessitating careful scrutiny of the recovery correction in the results calculation.

Conclusions

In reviewing the outcomes of the technical appeals that were referred to us in we find that we uphold the results of official analyses some 75 % to 85 % of occasions. By the use of sophisticated equipment, a high analytical replication rate, contextual and forensic awareness of analysis and interpretation we aim to offer consumers, industry, the courts and regulators assurance that the technical appellate function is discharged to the highest possible standards. Of necessity these measures require considerably more time and resource than routine testing however safeguard stakeholders from potentially very costly errors. We disseminate our learning from referee work via speaking engagements, our biennial conference, and publications. Hence it is a pleasure to acknowledge the assistance of colleagues in LGC and co-authors, principally Professor Duncan Thorburn Burns, who has given generously of his time and expertise in drafting the outcomes of our work for peer reviewed publication, a key measure of transparency in the discharge of the Government Chemist's responsibilities.

[13]Palm oil shown by analysis not to contain detectable amounts of Sudan IV

3 Impact

The impact of the work of the Government Chemist programme is necessarily broad and the effects can be seen in a number of ways.

We carry out horizon scanning to identify the issues where referee cases are more likely to arise, or where new regulation/legislation may lead to food business operators and local authorities requiring advice or support. We can then prioritise our resources. We are then able to plan and carry out research projects to support those areas identified in this way. These projects have benefits beyond the referee analyses carried out under the Government Chemist's statutory function, and can often impact on the wider measurement community to prevent disputes by promoting best measurement practice in emerging areas where disputes are more likely to arise. We disseminate our project outputs through knowledge transfer activities (which are detailed later in this review); a list of publications is given later in this section. The advisory function of the Government Chemist provides advice on a breadth of analytical measurement subjects, in the regulatory and legislative context, to Government, the European Commission, and the wider stakeholder community.

All these activities are aimed at predicting future regulatory issues within the areas of chemical and biochemical measurements with the objective of providing a secure base for more efficient and cost-effective regulations.

Horizon scanning

Preparedness for future problems is enhanced by our horizon scanning of the scientific implications of policy development, emerging and changing legislation, and enforcement trends. Our foresight activities, such as our reviews on legislation with a commentary on the associated scientific context, are published on our website. We collaborate with the Institute of Food Science and Technology (IFST), the Department for Environment, Food and Rural Affairs (Defra) and the Association of Public Analysts (APA) Training Committee, gaining and sharing insights on developments in the food industry and the official food and feed control system. One specific realisation of this collaboration has been in the setting up of the Defra funded Virtual Food Authenticity Network, VAN (www.foodauthenticity.uk). The VAN, project managed by Selvarani Elahi, the deputy Government Chemist, sets out to raise awareness of the tools available to check for mislabelling and food fraud and ensure that the UK has a resilient network of laboratories with fit-for-purpose testing to check for food authenticity.

Cumin and paprika - mahaleb or almond?

In late 2014 Canadian authorities reported undeclared peanut and almond in cumin and paprika. Against a previous backdrop of fraudulent substitution of almond by peanut,[14] compromised supply chains were feared. Product recalls occurred in Canada and the US. The Food Standards Agency, FSA, issued the first UK recall of ground cumin alleged to contain traces of almond protein not listed on the label, on 31 January 2015.[15] In early February FSA referred the matter to the Government Chemist, asking for a review of the analysis that had led to the recall, and a portion of the original sample was submitted. In early March 2015 it was claimed publically that another material in the supply chain, mahaleb, gives a positive reading for almond using routine test methods.[16] On 30 April 2015 the Canadian authorities rescinded product recalls of cumin and cumin-containing products previously thought to contain undeclared almond. The Canadian authorities considered it highly likely that their original results for almond were false positives due to *mahaleb*[17] however no definitive orthogonal analytical capability existed, anywhere.

Almond is a member of the large genus 'Prunus' that also includes apricot, cherry, peach and mahaleb. *Prunus mahaleb*, a species previously little known in the UK was alleged to have been handled in the cumin supply chain. The analytical chemistry and molecular biology of *Prunus* species in spices are not well represented in the scientific literature. ELISA, the usually applied technique for the detection of food allergens is known to exhibit cross reactivity to common *Prunus* species and so too did commercially available PCR DNA assays. To our knowledge there was at the time no published DNA or mass spectrometry assay specific to almond only.

The solution: Analytical strategy

A multidisciplinary investigation of the received laboratory sample was conducted, including:

- Enzyme Linked Immunosorbent Assays (ELISA)
- Searches of bioinformatic databases;
- The development and application of real-time Polymerase Chain Reaction (real-time PCR) DNA assays
- Investigation of control materials by liquid chromatography quadrupole time of flight mass spectrometry (LC-ToFMS) and protein analysis by triple quadrupole liquid chromatography - tandem mass spectrometry (LC-MS/MS) of enzymatically digested aliquots of the sample.

In addition GC-MS of almond and mahaleb volatiles and high resolution DNA PCR melt curve approaches were explored.

The referred laboratory sample was analysed using three different commercially available ELISA platforms. DNA databases were accessed for publically available sequence data on the genus Prunus to help design a real-time PCR assay specific for mahaleb DNA. The designed mahaleb-specific assay was challenged across a range of commercially sourced control materials and found to be satisfactory.

Amino acid sequences of key *Prunus* proteins were also searched and further potential peptide targets were identified by analysis of Prunus control materials, after enzymatic digestion, by LC-ToFMS. Multiple replicates of the laboratory sample and control material were exposed to trypsin digestion, extraction, followed by solid phase extraction clean-up and LC-MS/MS.

[14] M. H. Gowland and M. J. Walker, 2014, Food Allergy, a summary of 8 cases in the UK criminal and civil courts: effective last resort for vulnerable consumers?, J. Sci. Food Agric., 2015, 95, 1979-1990.

[15] Food Standards Agency, FSA, 31.01.2015, Bart Ground Cumin recalled, retrieved from https://www.food.gov.uk/news-updates/news/2015/13512/bart-ground-cumin-recalled 30 May 2015

[16] See for example: The Independent 05 March 2015 'Food firm in 'nuts-for-spices' row blames false test results' retrieved from http://www.independent.co.uk/life-style/food-and-drink/news/food-firm-in-nutsforspices-row-blames-false-test-results-10089275.html 30 May 2015

[17] Canadian Food Inspection Agency – Agence canadienne d'inspection des ailments, 30 April 2015, retrieved from http://www.inspection.gc.ca/about-the-cfia/newsroom/food-recall-warnings/complete-listing/2015-04-30/eng/1430432363404/1430432364107 30 May 2015

Results - Cumin

The cross reactivity of ELISA platforms to *Prunus* species was confirmed and quantified, and in the referred sample:

- Prunus species protein (or proteins) was detected above the limit of quantification of three ELISA platforms with statistical significance
- A specially developed real-time PCR method generated repeated responses consistent with mahaleb DNA being present, and no species examined co-amplified in the tests applied
- No peptides solely attributable to almond were detected by LC-MS/MS. Two peptides demonstrated chromatographic retention times and precursor/product MS response ratios consistent with those of control samples of mahaleb kernels. Hence chromatographic and mass spectrometric signals indicated that mahaleb protein was present in the referred sample.

In isolation, none of the applied techniques could answer, in a forensically robust manner, the question of whether almond or mahaleb or both were present in the cumin. However in combination the findings provided strong evidence of the presence of mahaleb rather than almond in the cumin.

The low sample weight and the small concentration of *Prunus* protein meant only some 20 milligrams of the mahaleb protein was available to us for examination. This, together with the pioneering nature of the investigation, made the research particularly challenging. The parties involved were kept informed of progress throughout the investigation and, when a definitive report had been approved by the Government Chemist, information was placed on our website.

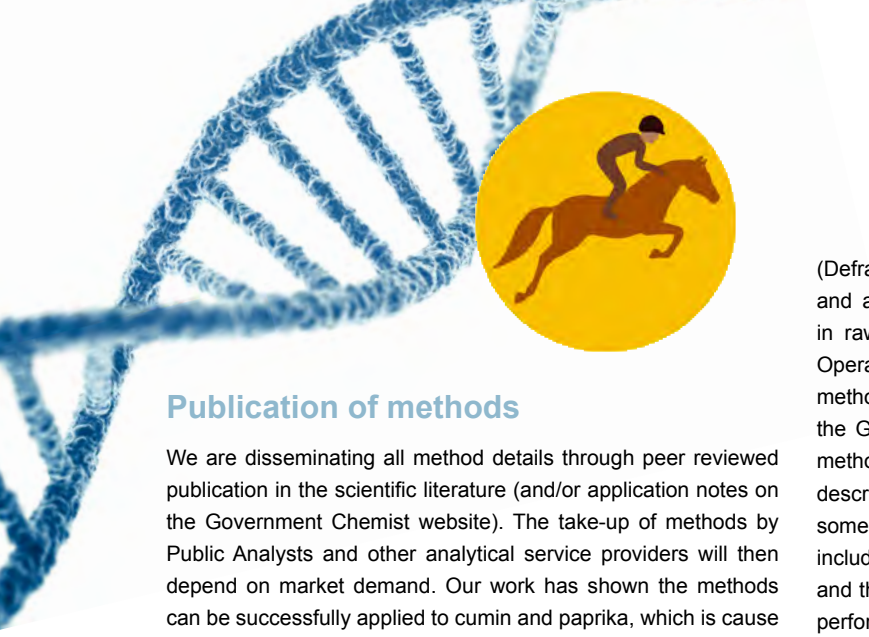
Results - Paprika

With the cumin question settled, the focus shifted to paprika, again at the request of FSA who assisted in part funding this investigation. A retained portion of a formal sample of paprika that had returned apparent positives for almond was obtained. It is understood that the product did not enter the UK food chain. ELISA and the advanced DNA and mass spectrometry techniques previously developed for the cumin case were investigated to ensure they were applicable in paprika.

We confirmed by ELISA that the referred sample contained Prunus species proteins and provided a semi-quantitative estimate of amount. The specially developed mahaleb-specific real-time PCR tests showed that mahaleb DNA was not present but, in a separate screening assay, again specially developed for the case, a profile for the sample consistent with the presence of almond DNA was found. Almond, but not mahaleb, signature peptides were confirmed by chromatographic and mass spectrometric findings. Hence in the paprika case almond was, as suspected, present rather than mahaleb.

Scope of analysis

All our results were based on randomly sourced commercial samples as controls and there are several species of apricot, many species and cultivars of plum and cherry, and many cultivars of peach. The mahaleb specific and Prunus screening DNA assays and the LC-MS/MS method have not been tested on all cultivars and species of apricot, plum, cherry or peach. It is assumed that the DNA and LC-MS/MS assays will not cross react with untested Prunus species, as confirmed by in-silico database searches, but additional experimental validation would be desirable further to reinforce this.



Publication of methods

We are disseminating all method details through peer reviewed publication in the scientific literature (and/or application notes on the Government Chemist website). The take-up of methods by Public Analysts and other analytical service providers will then depend on market demand. Our work has shown the methods can be successfully applied to cumin and paprika, which is cause for hope that they can be transferred to any food matrix. However it is always necessary to validate a method in the particular food tested to check for problems including interferences, cross reactivity, extraction problems or signal suppression.

Meat Authenticity

The Issue

Issues surrounding food authenticity and food fraud continue to be of significant interest within the European food industry, partly due to the pressures faced by producers within today's challenging financial climate and the international nature of modern food production. The 2013 EU-wide issue involving the detection of the undeclared presence of horsemeat in beef products destined for human consumption emphasised the need for the development of accurate analytical approaches for the quantitative detection of meat species in a sample.

The solution

In response to the 2013 horsemeat issue, the Department for the Environment, Food and Rural affairs (Defra) commissioned a project at LGC to develop a real-time Polymerase Chain Reaction (PCR) method for the quantitation of horse DNA (Defra project FA0135)[18]. This method was capable of detecting and accurately determining the amount of horse DNA present in raw beef samples spiked with raw horsemeat. A Standard Operating Procedure (SOP) was produced to accompany the method. Following additional method validation and funds from the Government Chemist, a peer review paper highlighting the method was published in the RSC Analyst in 2015[19]. The paper described the development of the method, as well as detailing some of the important performance characteristics of the method, including applicability to a range of samples, quantitative accuracy and the limits of sensitivity. Particular attention was drawn to the performance of the method at the 1 % w/w (raw horsemeat in raw beef) level, which was the threshold level decided upon for enforcement action during the UK and EU beef product surveys in 2013 and 2014.

A joint Defra/GC Knowledge Transfer event is described elsewhere in this review which relates to this work. Following this event, it was announced that the FSA would fund LGC to coordinate an International Collaborative Trial of the method. The Collaborative Trial is currently underway, and will characterise the inter-laboratory reproducibility of the method in order to qualify its fitness for purpose, with the eventual aim of submitting the method to the International Organization for Standardisation (ISO), for consideration as a standard methodology.

In parallel with the development and discussions surrounding best measurement practice guidance for the quantitation of horse DNA, the GC programme has also been focussing on complementary activities involving use of cutting edge technologies and molecular biology approaches for the identification and quantitation of adulterant meat species. The GC area has pioneered a preliminary approach using digital PCR for the accurate quantitation of meat samples. Digital PCR facilitates absolute single molecule identification of a target DNA strand without reference to a calibration curve, and can thus be less subject to detrimental effects typically encountered with real-time PCR, such as PCR inhibition. Many experts believe that digital PCR can give much more accurate quantitative estimates of a DNA target associated with a range of scientific disciplines, and the GC is currently looking at the applicability of this technology in relation to meat quantitation.

Additionally, the GC has investigated the use of two separate DNA targets and their effect upon quantitative meat species estimation. The weight of evidence of the published literature suggests that mitochondrial DNA targets can typically be very numerous but varied in number within a cell, giving rise to good sensitivity estimates. However, it is suggested that nuclear DNA targets, being less numerous within a cell but typically of a more stable number, could give rise to less variable quantitative estimates.

GC work has focussed on assessing the lower limits of applicability (levels of sensitivity) of using both mitochondrial and nuclear DNA targets for the successful identification of meat species. Using dilutions of DNA extracted from gravimetric preparations of horse in beef samples as a model, experiments have shown that both mitochondrial and nuclear DNA targets can be extremely sensitive, with limits of detection of around <0.01 % (w/w) and <0.1 % (w/w) respectively. Overall, mitochondrial DNA targets offered at least a 10 fold increase in sensitivity.

[18] http://randd.defra.gov.uk/Default.aspx?Menu=Menu&Module=More&Location=None&ProjectID=18741&FromSearch=Y&Publisher=1&SearchText=FA0135&SortString=ProjectCode&SortOrder=Asc&Paging=10#Description

[19] http://pubs.rsc.org/en/content/articlelanding/2015/ay/c5ay01867f#!divAbstract

In relation to the applicability of using mitochondrial and nuclear DNA targets for detection and quantitation of adulterant meat materials in samples containing offal, both targets still afforded good levels of sensitivity. Preliminary work also suggested that mitochondrial DNA targets, if used in a specific set of circumstances, can give reasonable quantitative estimates compared with nuclear DNA targets when mixtures of meat and offal are present in a sample, adding further value to what has been stated in the published literature. Results from further studies carried out in the GC programme on processed foods indicate that the accuracy of the quantitative estimates of adulterant meat species is highly dependent upon the total amount of amplifiable DNA present in a sample. Different foods can differ in their level of processing (e.g. raw meat, burgers, lasagne, corned beef, etc.) and can have differential effects upon the quantitative estimation of adulterant meat species in a sample, where highly processed foods can suffer from less accurate estimates.

Following on from the EU horsemeat crisis in 2013, it was apparent there was a greater need to develop sensitive and specific detection methods for meat ingredients, inclusive of those techniques that had quantitative potential. HM Government's Elliott Review was published in September 2014 and included recommendations and advice on improving laboratory testing capability to ensure a standardised approach for food authenticity testing, as well as engagement of innovative new technologies to help facilitate improved detection approaches. In 2015, publications appeared reporting that meat adulteration (including the findings of horse DNA) was a problem in the US food chain.

The GC programme continues to support and lead the science in this very topical area. The published paper provides evidence of LGC's strong leadership in the field of providing innovative approaches to modern food authenticity testing issues. The Knowledge Transfer event supported the skill base and testing capabilities of analytical testing laboratories within the UK control network, and contributed to realisation of the Elliott Review (q.v.) into the Integrity and Assurance of Food Supply Network recommendations on standardisation of laboratory testing.

Mini-Jelly Cups – Choking Hazard?

The issue

Jelly mini-cups first came to prominence in Europe in 2003 with instances worldwide of children and elderly people choking to death on soft slippery dome shaped jellies that were designed to be placed in the mouth in one bite[20]. A UK fatality occurred in 2003 in Bolton when an 18 month old boy died after choking on a jelly mini-cup. Owing to the risks they pose certain types of jelly mini-cup were in effect 'banned' after the 2003 incidents. In 2015 we were asked to resolve differences of opinion between two expert laboratories on the compliance of a batch of jelly confectionary.

The law

Regulation (EC) No 1333/2008 now controls the use of food additives and prohibits the use of 17 specified gel forming additives in jelly mini-cups. The crux however is how the regulations define jelly mini-cup: "... jelly confectionery of a firm consistence, contained in semi rigid mini-cups or mini-capsules, intended to be ingested in a single bite by exerting pressure on the mini-cups or mini-capsule to project the confectionery into the mouth...".

Some parts of the definition are straightforward to interpret but what constitutes "firm" or "ingested in a single bite" remain questionable although the Government Chemist published a peer reviewed paper in 2011 based on a previous jelly mini-cups referee case[22]. This paper remains the only guidance on the appraisal of such products against the relevant legislation.

[20] Jelly mini-cups were first sold in Japan in 1991
[21] Consolidated version 2008R1333 — EN — 06.11.2014 — 021.001 — 1
[22] Analytical Strategy for the Evaluation of a Specific Food Choking Risk, a Case Study on Jelly Mini-Cups, Michael J. Walker, Peter Colwell, Derek Craston, Ian P. Axford and Jack Crane, Food Analytical Methods, (online)2011, DOI: 10.1007/s12161-011-9223-3; Print, 2012, 5, 54-61

The current case

We assessed the information received and took the opportunity to review the literature since our paper was published. We were grateful to the laboratories involved for their reports on the products they received and their conclusions and we also had the opportunity to discuss the matter briefly and informally with each laboratory. We were careful to emphasise that our opinion was without prejudice to any future conclusions if a product were to be submitted as a referee sample.

It was common ground that these products contained at least two and possibly three additives (E407, carrageenan, E410, locust bean gum and E415, xanthan gum), that must not be used in jelly mini-cups. The products were asserted to be of a consistent composition but differed in important respects. On one occasion the labelled list of ingredients of the product did not include E415 (xanthan gum) whereas on others it did. The products exhibited differing firmness and solubility in artificial saliva on different occasions of testing. Some appeared to exhibit bleeding of liquid (synaeresis) while others did not.

There was also the question of the gel strength. A product made with carrageenan differs according to the type of carrageenan used, the species of seaweed from which it was obtained and even the hardness (calcium ion) content of the water with which it is made. It is possible therefore that the differences between the products tested may have resulted from differences in composition from batch to batch and/or subtle changes in the chemistry of the gels.

Serious injuries and deaths by choking on jelly mini-cups, containing carrageen and the much harder konjac, are comparatively rare but over 30 have occurred in Japan, South Korea, USA and the UK, with some victims lingering in a vegetative state. There are choking hazards associated with many other types of food. However, EU legislators have, for good reasons, drawn particular attention in food additives legislation to jelly mini-cups. And other jurisdictions (USA, Australia and South Korea) do so too. The courts of three jurisdictions have examined, in civil cases, the issues surrounding jelly mini-cup fatalities. In two jurisdictions, (USA and South Korea) the manufacturers were held liable. In a third (Japan) the manufacturer was not held liable, in part owing to particular circumstances.[23,24]

The opinion

We took the view that the matter turned on two questions: (a) did the product have a firm consistence? and (b) was the product intended to be ingested in a single bite?

In our 2011 paper we proposed that 'firm' means a choking risk (a) by being hard, so that it requires considerable force to bite into and, or (b) not being readily disrupted or brought into solution by saliva (or its simulants) in a time of two minutes. The first limb of the definition applies to jellies containing konjac; the second applies to jellies containing other gel forming additives. In our view in the most significant test in the present case, where it was carried out (2 out of 3 occasions), the product remained intact and unchanged in artificial saliva under the recommended conditions for over two minutes, even for a sample considered to be 'very soft and pliable with little structural strength'. In compression tests the products ruptured between 5 and 14 N, these values, although much lower than those reported in our 2011 paper (>32.8 N) are commensurate with compression strengths mentioned in Korean regulations on jelly mini-cups. Therefore we considered the product had a firm consistence.

Addressing the 'ingested in a single bite' question we considered the product size, weight and shape. The product partially fitted into the 'small parts cylinder' and passed through 'template E'. Items which fit completely within the 'small parts cylinder' (BS EN 71-1:2005) without the application of pressure are deemed a choking risk for children less than 3 years of age. 'Template E' of the British and European Toy Safety Standard, BS EN 71-1: 2011 addresses the risk of objects entering a child's mouth and blocking the airway at the back of the mouth and upper throat. We concluded that ingestion in a single bite was probable.

Overall we considered, on the basis of the extensive information received and the peer reviewed (forensic) papers on the topic, that the product met the definition of a jelly mini-cup. Hence it was not compliant with the provisions of Regulation 1333/2008 as implemented by the Food Additives, Flavourings, Enzymes and Extraction Solvents Regulations 2013. Accordingly we advised the batch should be remitted to the Local Authority for formal sampling. This afforded the Public Analyst and the food business the opportunity to analyse the same batch and if a dispute remained we agreed to analyse the retained portion. In the event the matter appears to have settled with the consignment not coming on the UK market. We are currently drafting an update paper on jelly mini-cups for submission for peer reviewed publication to share our learning.

The problems we are asked to tackle are often complex and difficult, balancing the protection of vulnerable consumers and being fair to businesses. But we are privileged to have the scope for in depth multidisciplinary scientific investigation and measured forensic deliberations, often building on the published work of others. We trust that publishing our own findings both in annual reviews and in the peer reviewed literature we do justice to both the difficulties and the privilege of addressing them.

[23] Kawawa, Noriko. "Jelly Mini-Cups Containing Konjac: Is a Warning Enough to Protect Vulnerable Consumers?." Australian Journal of Asian Law 13.2 (2013)
[24] Kim, Suk Shin. "The Mini-Cup Jelly Court Cases: A Comparative Analysis from a Food Ethics Perspective." Journal of Agricultural and Environmental Ethics 27.5 (2014): 735-748

Nanosilver or ionic silver – how can we tell them apart?

The issue

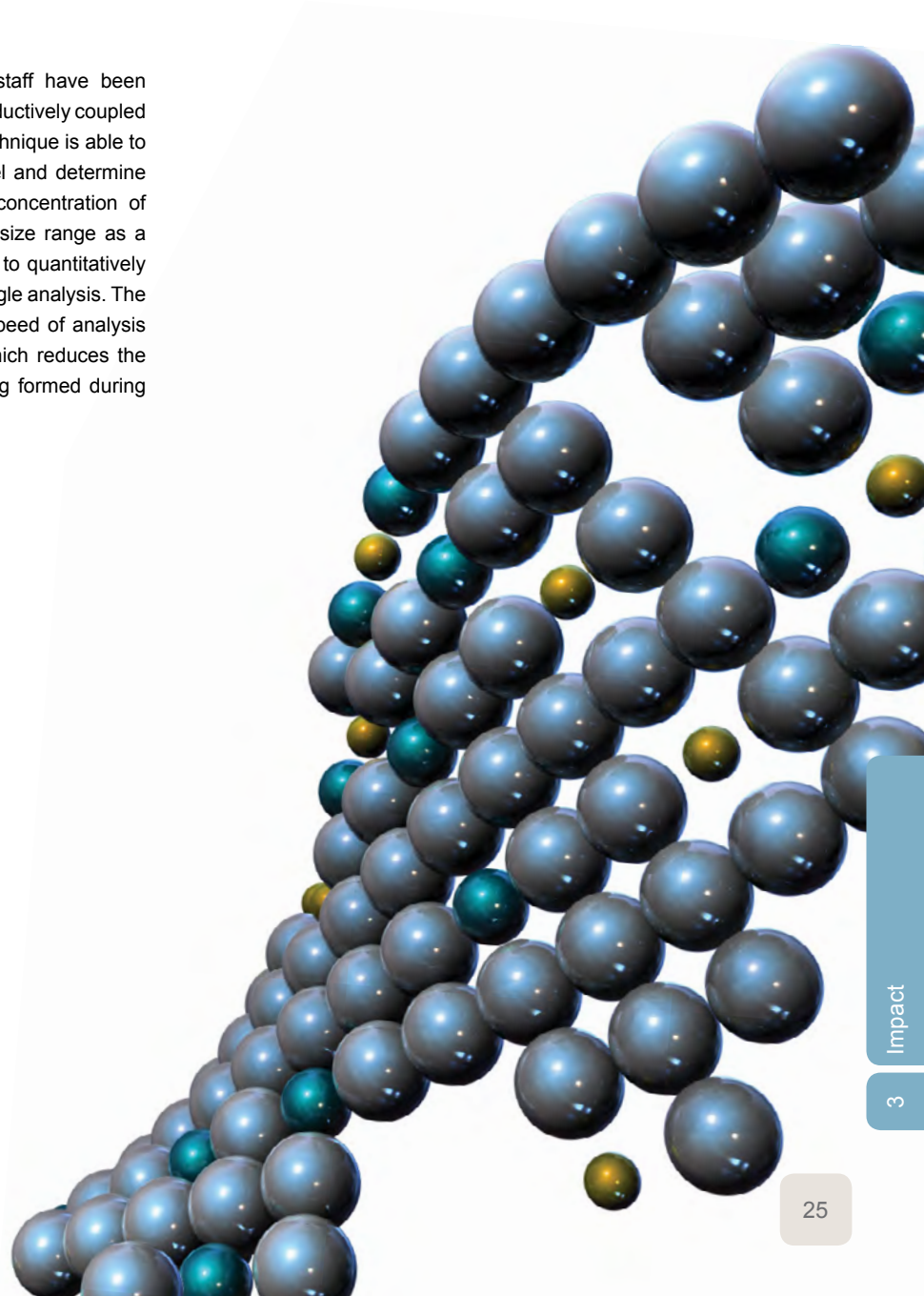
Nanotechnology, the study and use of particles of 1 to 100 nm, is a rapidly growing science with nano-materials being used in products from food packaging to healthcare to construction. Silver nanoparticles (NPs) in particular have played a major role in nanotechnology since the beginning of the 21st century and are currently widely used as antibacterial agents in textile coatings, cosmetics and disinfectant products.

There is increasing concern about the fate of silver nanoparticles following their release into the environment as it known that silver nanoparticles can convert to the ionic form (Ag^{2+}) in the environment. Some studies have shown that silver ions are significantly more toxic than silver nanoparticles so in order to better understand the potential impact on the environment and human health, there is a need for quantitative particle counting methods which comply with EU recommendations[25] and that are capable of differentiating between the ionic silver and the nanoparticle/aggregate fractions present in the environment.

Current microscopy-based techniques, although able to determine the size of the particles, are not capable of providing information about the number-based concentration of nanoparticles directly in suspension. Other techniques that are capable of both counting and sizing nanoparticles directly in suspension, such as nanoparticle tracking analysis (NTA), suffer from matrix-induced interferences, which could lead to a bias in the results. Crucially, none of these approaches are able to distinguish between the ionic silver and the nano-silver.

The solution

To address this need, Government Chemist staff have been assessing the use of single particle analysis by inductively coupled plasma mass spectrometry (spICP-MS). This technique is able to characterise metallic NPs at the ultra-trace level and determine both the size distribution and number-based concentration of NPs, i.e. the number of objects within a given size range as a fraction of the total. As such it has the potential to quantitatively determine both ionic silver and silver NPs in a single analysis. The primary advantages of this technique are the speed of analysis and the minimal sample treatment required, which reduces the possibility of artefacts such as aggregates being formed during sample manipulation.



[25] http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:275:0038:0040:en:PDF

Feasibility study to assess the potential for ICP-MS for silver nanoparticle measurement

A feasibility study was performed using a model system: a highly diluted silver suspension containing both a silver nanoparticle reference material (RM, NIST SRM8017) and ionic silver in the form of silver nitrate. No significant amount of dissolved silver was found in a suspension of the pure RM, making it ideal for use in this situation.

The diameter of the silver NPs in the mixture was determined and the size distribution of the silver NPs in the mixture was obtained using spICPMS. This suggests a nominal diameter of 71±2 nm (Figure 3), in good agreement with the nominal value of 75 nm given by NIST for the RM. Due to the high degree of dilution and low particle numbers present in the sample, this data was also used to provide information about the number-based concentration. A concentration value of 5.0×10^{17} particles/L was obtained, indicating a recovery of 95±8 % and suggesting that this approach is indeed quantitative.

The advantage of spICPMS for this application is that the single particle events for silver NPs are easily distinguished from the signal produced by ionic silver and consequently both forms of silver can be quantified in a single run, as shown in Figure 4. The maximum level of ionic silver that can be separated from silver NPs using this method, and without the need for an additional fractionation or separation techniques, was determined to be 10 ng/g.

Conclusions and future work

Initial work has shown the feasibility of spICPMS as a technique for the characterisation, in terms of both size and number-based particle concentration, of silver NPs in samples that contain low levels of ionic silver (<10 ng/g). Quantification and differentiation of the ionic silver from silver NPs can be carried out rapidly in a single analysis which takes approximately 1 minute. This would allow the behaviour of these NPs in the environment to be monitored and could support work in understanding their toxicity. In samples where the levels of ionic silver are much higher than that of the silver NPs additional separation techniques such as field flow fractionation should be considered prior to spICP-MS as they extend the range of samples that can be investigated.

Knowledge transfer

The Government Chemist seeks to benefit innovation and regulation by dissemination of knowledge gained through our work, particularly in referee analysis. This dissemination is aimed at both the analytical and regulatory communities to improve knowledge and skills through a coherent package of knowledge transfer activity which includes:

- The organisation of the Government Chemist conference (on a biennial basis)
- The publication of case studies based on actual referee analysis
- The organisation of training in collaboration with the APA Educational Trust, the Food Standards Agency and Defra;
- Proactive input to key stakeholder organisations; and
- Provision of sound advice to stakeholders.

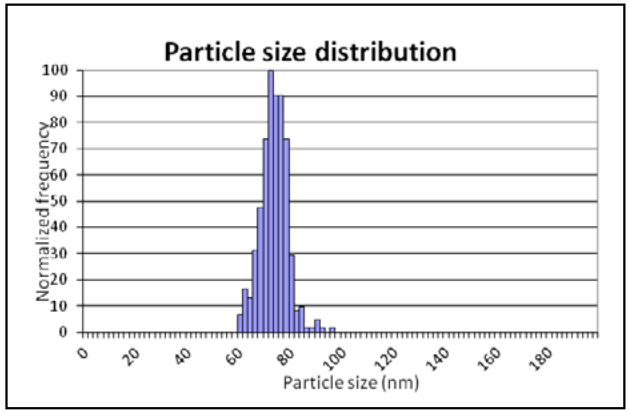


Figure 3: Particle size distribution of NIST silver NPs with 75 nm nominal size

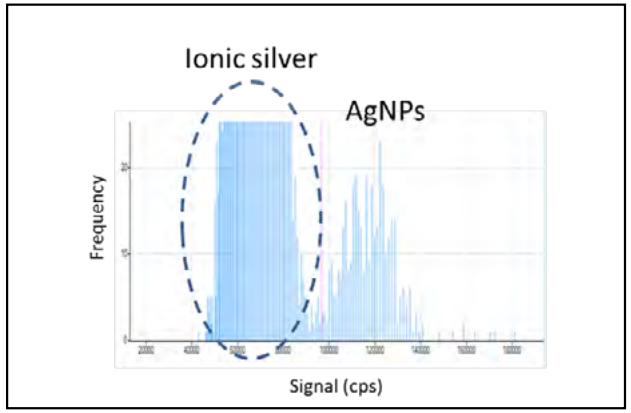


Figure 4: Signal distribution of NIST silver NPs spiked with 5 ng/g ionic silver

Government Chemist conference

The biennial Government Chemist conference is an important event in the programme calendar and plans for the next two-day event, to be held in June 2016, are well under way.

The conference promotes the fundamental role of sound measurement science in ensuring the safety and integrity of the food chain, and will be highly relevant to all stakeholders including public analysts, government officials, industry and public sector scientists, academics and other leading food experts.

The conference, “Science supporting trust in food”, will be held at the Royal Society, London, on 21st and 22nd June 2016.

The Government Chemist Website

The Government Chemist website has now been hosted on the GOV.UK platform since July 2014, with the landing page: www.gov.uk/government/organisations/government-chemist
The Government Chemist pages can also be reached from anywhere on the website by entering “Government Chemist” in the search box

Only recent and relevant corporate information, research, news and policy content have transitioned to the new GOV.UK website. Updates on Government Chemist news can be obtained by subscribing for alerts via the website.

Advice

Many different stakeholders turn to the Government Chemist for advice on a wide range of topics. We answer around 5 requests for advice per month, a level that has remained constant for the past few years. Table 4 below summarises who asked us for advice in 2015 and Table 5 describes the topics we were asked to comment on.

Table 4 Stakeholders asking the Government Chemist for advice in 2015

Origin of enquiry	Number of enquiries
Commercial	24
Official Control Laboratories	11
Press, radio, journals, etc	11
Food Standards Agency / Defra	7
Individual	4
Trading Standards / Environmental Health / Local Authority	4
Government (international)	2
Solicitor	2
Port Health Authorities	1
University Dept	1
Total	67

The largest number of enquiries related to allergens, which reflects an increasing interest and awareness following the publication of EU Regulation in this area. Other topics such as evaluation of the choking risk posed by jelly mini-cups, food authenticity and the interpretation of results, and nutrition analysis generated regular questions. In each case we gave carefully considered advice, supplying a copy of our peer reviewed research findings on the question and sometimes referring the enquirer to another source of information.

The enquirers were invariably grateful for our time and advice.

Table 5 Summary of topics we have advised on

Subject	Number of enquiries
Allergens	13
Scientific peer review	7
Food Authenticity	4
Referee analysis	4
Jelly mini-cups choking risk	3
Illegal dyes	3
Irradiation	2
Nitrogen factors	2
Aluminium	2
Tobacco / e-cigarettes	2
Oils & fats / lipids	2
Pesticides	2
Other	21
Total	67

Training

The Government Chemist acquires a great deal of expertise and knowledge through discharging the statutory function. This forms the basis of material which can be used in the provision of training for practising analysts.

In September 2015, Malcolm Burns led on a joint Defra/GC Knowledge Transfer (KT) workshop on the use of real-time PCR for food authenticity testing and quantitation of horse DNA. The event was attended by Public Analysts from across the UK, as well as representatives from both Defra and Food Standards Agency (FSA). The joint event delivered a series of audio-visual presentations regarding the principles and best measurement practice guidelines for use of the real-time PCR method for quantitation of horse DNA, and consisted of workshops on data analysis, interactive breakout discussion groups, and provision of event handouts incorporating relevant guidance material and decision trees. The event focused on best measurement practice on all aspects of using real-time PCR for food authenticity testing but with particular emphasis being placed on the application of the methodology and associated SOP for the quantitation of equine DNA isolated from raw meat products. The event concluded with a round table interactive discussion session where topics covered included the provision of advice and best practice guidelines for qPCR in food authenticity testing, as well as providing a forum for Public Analysts to identify and prioritise future training needs. The Knowledge Transfer event focussed on bringing together a number of UK Official Control Laboratories along with the Government Chemist team to discuss and share their experiences and expertise. As a direct result of this valuable Knowledge Transfer exercise, a list of requirements and recommendations were identified regarding using real-time PCR for food authenticity testing.

In cooperation with the APA Educational Trust, our joint flagship training activity is the “Analysis and Examination of Foods” an intensive annual week long residential postgraduate course. This is held at Reading University and is aimed mainly at public analyst laboratory staff working towards the MChemA the statutory qualification required to practice as a Public Analyst. In April 2015 we hosted 13 registered delegates. The delegates were from UK Public Analyst Laboratories, both private and public sector, and one was from the Food Standards Agency. Several sessions were devoted to demystifying the MChemA exam process encouraging other delegates to consider taking the qualification. Some of the lectures were given by Public Analysts currently in practice while others were from experts outside the profession. The course received excellent feedback and all delegates recorded that they enjoyed the course and considered that it met their expectations to a high degree. More information on the course is available on the Government Chemist website.

The Reading course was organised and facilitated by Michael Walker acting as APA Training Officer. Michael also recorded an entry level webinar on food authenticity for broadcast late in 2015 as part of a food webinar ‘Food eSeminar: 2. Food authenticity and traceability testing’ and participated in a ‘Romer Academy’ two-day intensive workshop on allergen management and advanced testing in Tulln, Austria.

The wider advisory function

The Government Chemist also has a role to provide advice on subjects with an analytical measurement dimension to both government (including the European Union and devolved administrations) and the wider community of stakeholders, which includes industry, academe, Non-Governmental Organisations (NGOs) and local Government. This is done by means of the provision of specific advice pertaining to aspects of measurement topics on a broad range of policy and regulatory developments, and also providing a proactive scientific and measurement-based support service to those industries where chemical measurements are an important aspect of their activities. The publication of our outputs through the Government Chemist website is an important means of disseminating such advice as well as receiving feedback.

Addressing scientific issues with stakeholders

We have continued to follow developments of both the UK Chemical Stakeholder Forum (UKCSF) and the Hazardous Substances Advisory Committee (HSAC) by attending meetings of these bodies and, where appropriate, making contributions to relevant discussions. We continue to be the de facto experts on analytical measurement issues within the HSAC, and have been frequently asked to provide an opinion on this where required. We have been invited to join a small sub-group of HSAC considering the Committee's input into the UK (Defra-led) response to the forthcoming consultation from the European Commission on the proposed definition of a nanomaterial. This is a significant breakthrough as our status in HSAC is as an observer body, and it is rare for observers to be asked to participate in sub-groups of the Committee. We have also provided comment and input to Defra's proposed Chemicals Management Strategy as a consequence of our involvement with HSAC.

Box 2: Our public consultation responses

Food Standards Agency (Wales)	Consolidation of animal feed legislation in Wales for Composition, Marketing and Use and for Hygiene, Sampling, etc. and Enforcement
Food Standards Agency (Northern Ireland)	Consolidation of animal feed legislation in Wales for Composition, Marketing and Use and for Hygiene, Sampling, etc. and Enforcement
Food Standards Agency (Northern Ireland)	Country of Origin of Certain Meats Regulations (NI) 2015
Food Standards Agency (Wales)	The Natural Mineral Water, Spring Water and Bottled Water (Wales) Regulations 2015
Food Standards Agency (Northern Ireland)	The Natural Mineral Water, Spring Water and Bottled Water (Northern Ireland) Regulations 2015
Food Standards Agency (Northern Ireland)	The Food Law Code of Practice (Northern Ireland) Review 2015

We are now active members of the Nanomaterials Environment and Health Government Group (NEHGG), the successor body to the Government Officials Strategy Group on Nanomaterials, led and chaired by Defra. We have contributed to this group by continuing to make our views very clear on the need for valid measurement methods for the determination of nanoparticles to be developed in support of the proposed definition of a nanomaterial, and highlighting developments in measurement science in this novel area.

We have continued to provide advice through our responses to official consultations (see Box 2). These consultations are carried out by the Government (including devolved administrations and agencies), standards bodies or Directorates-General of the European Union, to obtain the input of both interested and expert stakeholders on proposed new legislation or regulations, prior to enactment and are considered by legislators to be an important part of the development process for new legislation and regulation. The Government Chemist is well-placed, through the expertise within LGC in a breadth of matters in analytical science, to respond authoritatively and independently to a wide range of consultations which have chemical or bioanalytical measurement implications.

Specific questions which we addressed included:

- Ensuring that consolidations to legislation do not inadvertently change how it operates with respect to the Government Chemist's activities
- The need to ensure that appropriate sampling and testing regimes are in place to be able to monitor possible radioactive contaminants in spring water
- Highlighting the analytical measurement difficulties and opportunities in identifying the country of origin of certain meats.

Dissemination

During 2015, we have refocused the blog on the promotion of events and publications, with the stories previously carried by the blog moving to the Government Chemist website. This changed occurred in June 2015.

We gave a talk on the activities of the Government Chemist advisory function at the 59th meeting of the United Kingdom Chemical Stakeholders Forum (UKCSF) in January 2015. We also presented the GC's work on nanoparticle characterisation (see page 26) to European laboratories working in this area.

Taking our advice into new areas

We have provided advice in new areas during 2015. We were consulted by the Department for Transport regarding an application for the renewable component of end-of-life tyres. We studied the technical documentation accompanying the application and verified that the chemistry and associated calculations were correct.

We organised a seminar held in Birmingham in March 2015, which was a repeat of the successful 2013 seminar "REACH and CLP enforcement: measurement and related issues for Public Analysts and Enforcement Authorities" following a request from the Training Committee of the Association of Public Analysts. Over 50 delegates attended.

Lab-based studies

The prioritisation process undertaken by the GCPEG prior to the commencement of the 2014-2017 programme identified a small number of proposed project areas which were considered appropriate for small-scale funding. Other small-scale projects have been, and will continue to be, developed in response to issues which surface during the programme.

The following study was concluded under the 2014-2017 programme:

- A desk study looking at the current and proposed priority hazardous substances and priority substances listed in the Water Framework Directive and its daughter directives, with specific reference to the ability of environmental monitoring laboratories to measure these compounds accurately at the maximum levels laid down in the regulations. The report highlighted where gaps in measurement capability exist, and also considered the quality assurance tools available to assist environmental monitoring laboratories concerned with the effective enforcement of the Water Framework Directive and its daughter directives. The report was published on the Government Chemist website and we received positive feedback from both the Environment Agency and the European Commission DG Environment.

Other projects have been started in this programme which will be completed during 2016. These are:

- Identification of sustainable timber, which was highlighted in the 2014 Review. A desk study to ascertain whether this is feasible was completed in 2014 and laboratory work was commenced. This has concentrated on various species of oak, specifically the extraction and subsequent analysis of oak DNA in order to differentiate species. The extraction step has proved very challenging, but some good progress has been made.

  DNA has been extracted from non-dried timber using a commercial kit-based method. Quantity and quality were evaluated spectrophotometrically and integrity with use of gel electrophoresis, and results obtained indicate that the DNA was of low concentration, associated with co-purifying organic compounds, and subject to a high degree of shearing. Subsequent sequence analysis enabled the positive identification of samples to the species level with reference to the NCBI GenBank and BOLD databases. The DNA extraction method has subsequently been refined with improvements to both yield and quality being observed. Endpoint PCR has been repeated and the resulting amplicons will be sent for DNA sequencing in order to evaluate a sequencing approach for the purpose of timber identification. The success of this project to date has demonstrated that there is a basis for the identification of timber species, which could potentially be applied to the identification of "illegal" timber sources.

- Work has also been carried out on the development of a method to differentiate the ionic, more toxic, form of silver from the nanoparticulate form in the environment. This small project has demonstrated that silver nanoparticles can be measured separately from silver ions so that environmental monitoring laboratories can get a much more accurate picture of the ionic silver load in effluent streams and water treatment plants in the UK. More detail on this study is given on page 26.

[26] http://governmentchemist.wordpress.com/


Agilent 1

Chemical nomenclature

The Government Chemist was represented by Kevin Thurlow on IUPAC's Advisory Committee to Chemical Nomenclature and Structure Representation Division (VIII) since its inception in 2002 until his recent retirement. Kevin has also represented the Government Chemist on the RSC's "Committee on Standards in Nomenclature, Terminology, Units and Symbols" (CSN) from 1991 - 2014.

The former committee is "virtual". Members are invited to comment on draft proposals and documents and to participate in drafting of new or revised recommendations for chemical nomenclature.

The RSC committee meets once a year. Most members are concerned with education (both school and university), but there are representatives of BSI and scientific societies. Government Chemist input is appreciated as it brings in an industrial and regulatory focus, more practical than theoretical, which is otherwise absent from the committee.

The LGC Forensic Drugs team assists the Ministry of Justice in the preparation of amendments to legislation. Our input to this consisted of supplying accurate chemical names and descriptions so that legislation could continue to deal with "legal highs". It is important that the correct chemicals or families of chemicals are banned, whilst allowing harmless chemicals, or legitimate medicines to be items of trade. Many of the "legal highs" are chemically very similar to legitimate products, so care needs to be taken to ensure compounds with structural similarities to a banned product are not inadvertently caught under this developing area of legislation.

Substance identity is very important in compliance with legislation, particularly relating to chemical safety. REACH requires accurate naming of chemicals so that correct procedures can be followed to use chemicals safely, or to deal with problems efficiently if they occur. Kevin has also assisted with nomenclature for other legislation, and delivered a talk on the importance of nomenclature to chemical safety.

It is also important to use correct names in publications to aid communication. A paper reporting high-class research can be rendered worthless if it is not clear which chemicals are involved.

SCA Committees

The Government Chemist is also represented on the Steering Committee of the Standing Committee of Analysts (SCA). The SCA, sponsored by the Environment Agency, comprises a series of working groups who provide authoritative guidance on methods of sampling and analysis for determining the quality of environmental matrices. Guidance is published as Blue Books within the series "Methods for the Examination of Waters and Associated materials".

During the year Gary Bird continued as Chairman and Co-ordinator of the Radiochemical Methods Working Group (WG9) of the SCA and started work on reviewing the current Blue Book radiochemical methods, including extending the scope of Blue Book 94 to cover measurement of radon gas.

Publications

Publishing peer reviewed papers is integral to our work enabling transparency to the analytical community. The following were published in 2015:

Busby E and Burns M, Method Verification of the LOD Associated with PCR Approaches for the Detection of Horse Meat, JAPA, 2014, 42, 1-17

Dunn PJH, Webb EC, Honch NV, Eriksson G, Liden K and Evershed RP, Compound-specific amino acid isotopic proxies for detecting freshwater resource consumption, Journal of Archaeological Science, 2015, 63, 104-116

Nixon G, Burns M and Wilkes T, Development of a real-time PCR approach for the relative quantitation of horse DNA, RSC Analytical Methods, 2015, published on website, DOI: 10.1039/c5ay01867f

Wilkes T, Bushell C, Nixon G, Waltho A, Alroichdi A and Burns M, Application of spectral imaging to pasta authenticity testing, International Journal of Food Science and Technology, 2015, (awaiting publication)

Hugget J, Whale A and Garson J, Digital PCR and its potential application to microbiology, 2015, Molecular Microbiology: Diagnostic Principles and Practice 3rd Edition (accepted for publication)

Holcombe G, Topping J and Walker M, A peanut quality control material to improve allergen analysis – How difficult can it be?, Clinical and Translational Allergy, 2015, 5 (Suppl 3), 116

Hill S, Taylor A, Day MP, Marshall J, Patriarca M and White M, Atomic spectrometry update: review of advances in the analysis of clinical and biological materials, foods and beverages, Journal of Analytical Atomic Spectrometry, 2015, Published on website, http://pubs.rsc.org/en/content/articlelanding/2015/ja/c5ja90001h#!divAbstract

Walker M, Thorburn Burns D, Elliott C, Gowland MH and Mills C, Is food allergen analysis flawed? Health and supply chain risks and a proposed framework to address urgent analytical needs, Analyst, Nov 2015.

Walker M, Holcombe G, House D, Topping J and Mills C, 2015, A peanut quality control material to improve allergen analysis – How difficult can it be?, Clinical and Translational Allergy, 2015, 5 (Suppl 3):P116

Gray K, Walker M, Burn M, Mazur M, Niedzwiedzka K, Lizka K and Thorburn Burns D, Screening for the presence of a range of commonly used illegal dyes in food and spices, JAPA, 2015, (awaiting publication)

Burns M, Measurement issues associated with quantitative molecular biology analysis of complex food matrices for the detection of food fraud, Analyst, 2015, DOI: 10.1039/C5AN01457C

Glossary

See the International Vocabulary of Metrology[27] for the current definitions of terms used in measurement science

AFM	Atomic force microscopy, a very high-resolution type of scanning probe microscopy
AMWG	Authenticity of Methods Working Group (Defra)
APA	Association of Public Analysts
BIS	Department for Business, Innovation and Skills
Defra	Department for Environment, Food and Rural Affairs
DfT	Department for Transport
DH	Department of Health
DNA	Deoxyribonucleic acid
ECHA	European Chemicals Agency
EFSA	European Food Safety Authority
ELISA	Enzyme-linked Immunosorbent Assay
EU-RL	European Union Reference Laboratory
FBO	Food or feed business operator
FSA	Food Standards Agency
FCM	Food Contact Material
FFF	Field Flow Fractionation
GC-MS/MS	Gas chromatography-tandem mass spectrometry
GCPEG	Government Chemist Programme Expert Group
GMO	Genetically Modified Organism
HSAC	Hazardous Substances Advisory Committee. Expert committee pro-
IUPAC	International Union of Pure and Applied Chemistry
LC-ToFMS	liquid chromatography quadrupole time of flight mass spectrometry
LC-MS/MS	Liquid chromatography-tandem mass spectrometry
MChemA	Mastership in Chemical Analysis – this Royal Society of Chemistry qualification is required for appointment as a Public Analyst or as an
MRL	Maximum Residue Level
N	Newton, SI unit of force
NEHGG	Defra-led Nanomaterials Environmental and Health Government Group.
NMI	National Measurement Institute
NMRO	National Measurement and Regulatory Office
Official Food Analyst	A person qualified under the Food Safety (Sampling and
PCR	Polymerase chain reaction, a technique used to amplify DNA sequences so that they can be identified
Port Health Authority	Special type of local authority created to ease administration at seaports where the port area is covered by more than one local
Public Analyst	Analytical scientist appointed under statute by UK local authorities to
Quantitative analysis	Measurement, with results expressed as a number and a unit, of the
REACH	Regulation (EC) No 1907/2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals, as amended
RSC	Royal Society of Chemistry
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 Government Chemist under acts of Parliament to provide impartial analysis in the resolution of disputes relating to the enforcement of regulation

[27] International Bureau of Weights and Measures, International vocabulary of metrology – basic and general concepts and associated terms (VIM), Third Edition, JCGM 200:2008, 2008, www.bipm/org/utils/common/documents /jcgm/JCGM_200_2008.pdf

Glossary

SCA	The Environment Agency's Standing Committee of Analysts
SCoFCAH	Standing Committee on the Food Chain and Animal Health
SEO	Supplementary expert opinion in the context of Regulation (EC) No 882/2004 on official controls, Article 11(5)
SOP	Standard Operating Procedure – a standard, validated method for analytical measurements
Sp-ICPMS	Single particle inductively-coupled plasma-mass spectrometry
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
UKCSF	United Kingdom Chemical Stakeholders Forum
WFD	European Union Water Framework Directive

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