GOVERNMENT CHEMIST

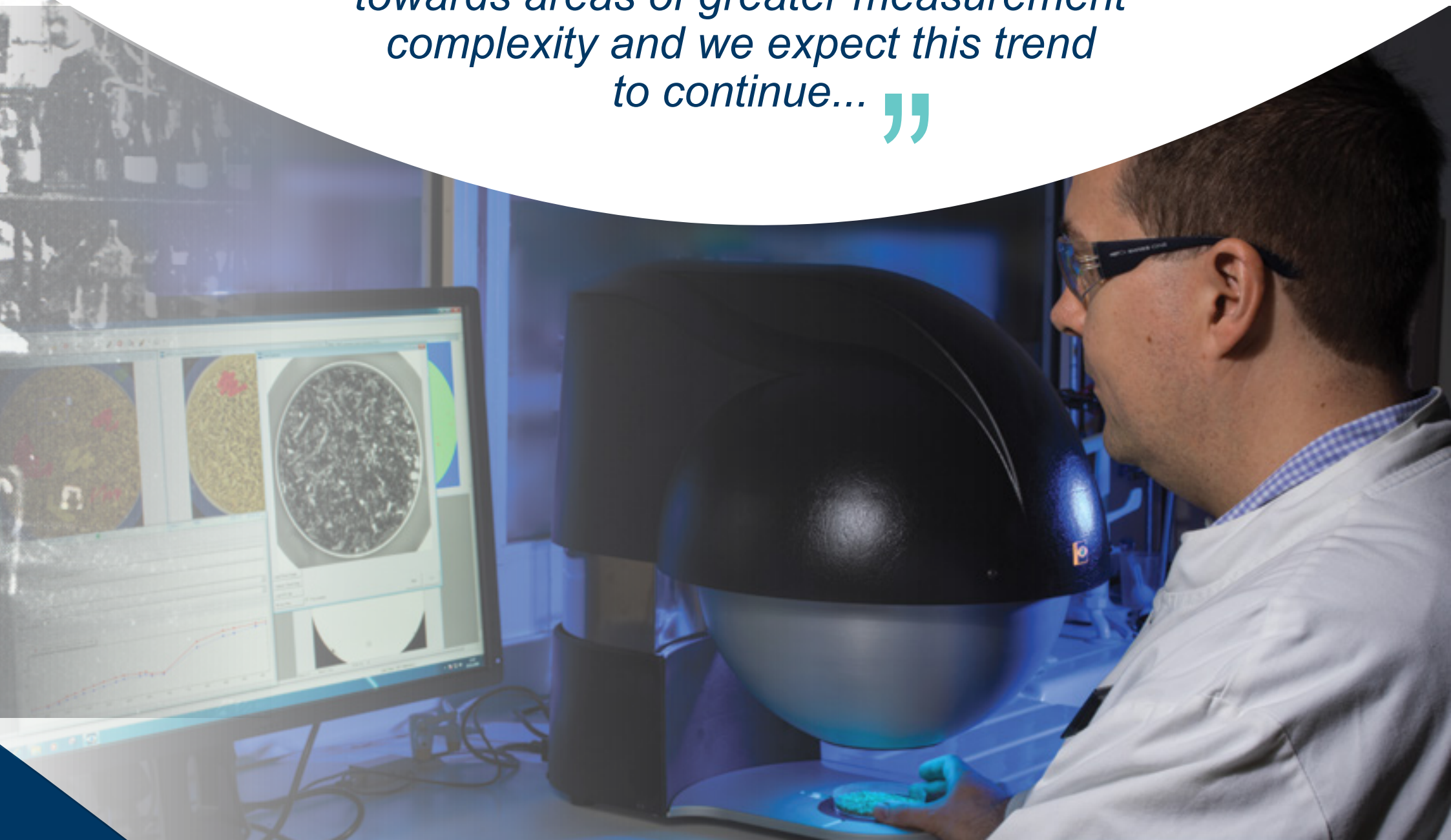
Review 2017

Department for Business, Energy & Industrial Strategy

FUNDED BY BEIS

CONTENTS

“...in the last ten years we have seen a shift towards areas of greater measurement complexity and we expect this trend to continue...”



Foreword by Derek Craston, Government Chemist

This is my tenth and last Government Chemist Review as I hand over to my successor, Julian Braybrook, at the end of May 2018. The role of Government Chemist is a fascinating one, due to the breadth of its coverage and the nature of the science required to fulfil the statutory and advisory functions. Since 2008 we have seen a shift in our work towards areas of greater measurement complexity. I expect this trend to continue in response to new advances in measurement science and regulation and due to a shifting landscape in topics like authenticity, and in areas of public and political concern. I look forward to following these developments through future Government Chemist Reviews.

Looking to the future, we have already initiated a number of scientific projects that utilise advanced measurement tools to address unmet regulatory needs. Some of these projects are described in this review. For example, regulation is generally enforced through targeted methods that look for specific elements, molecules, biologicals or known sequences of DNA. The maturing of measurement technologies in Next Generation Sequencing, proteomics and metabolomics are enabling their robust use in applied markets. Therefore, there is an opportunity to employ wide based screening approaches that can detect a varied range of areas of potential non-compliance as well as flagging deviations from the norm that might warrant further investigation. Similarly we have started looking at screening approaches that can work in the field with a view to understanding their potential, and the requirements for calibration and validation that might allow their use in frontline enforcement. In the longer term, as these tools evolve, reduce in price, and improve in ease of use, reliability and performance, the regulatory system might need to adapt to potential use by consumers in the home, restaurant or supermarket.

Of course, a principal objective of this review is to report on the casework of the Government Chemist as defined in the related statutes of law. In 2017 we supported disputed measurements in areas of familiarity, like aflatoxins and nitrofuran contamination, as well as newer subjects such as allergens and controlled level additives (sulphites). Casework numbers increased this year somewhat on 2016 and remained demanding in their delivery and interpretation. It is also worth noting that in a number of cases we were able to resolve disputes through discussions of the data and regulation with the relevant parties, thereby further reducing the associated cost to the public purse.

As I come to the end of my tenure I would like to finish with a note of thanks to the experienced team that has delivered the work of the Government Chemist over the last few years. Specifically I would like to acknowledge the significant contribution of Michael Walker who has supervised the casework and disseminated its outputs with care, enthusiasm and clarity for more than a decade. I would also like to acknowledge the work of Selvarani Elahi who has deputised in my absence; of Kirstin Gray who has managed a large proportion of the laboratory work; of Malcolm Burns who has led our efforts in molecular biology; and of Simon Cowen who has managed the team that has provided statistical rigour to our work. Finally I once again thank our sponsoring Government Department (Department for Business, Energy and Industrial Strategy) and the Programme Expert Group who have ably supported our work over the last twelve months. If you have any feedback on the review then please feel free to send it to me directly.

Derek Craston
BSc, PhD, Hon.DPhil, FRSC
Government Chemist

Note from Paul Berryman, Chair of the Government Chemist Programme Expert Group

It is my pleasure to contribute to the 2017 Government Chemist Review as Chair of the Government Chemist Programme Expert Group (GCPEG). I would like to start my contribution by thanking Andrew Damant for deputising for me in the meetings during 2017.

The GCPEG is responsible for the governance of the Government Chemist programme. This expert group comprises key stakeholders representing regulation and policy makers, industry, public analyst laboratories, port health authority, and academia. We meet twice a year to provide independent oversight of the ongoing referee casework programme, research projects and advice given by the Government Chemist, and also review the quarterly progress reports.

The three year programme running from 2014-2017 concluded successfully in March, having met all the objectives for the individual projects. As well as discharging the referee and advisory function, the Government Chemist team developed capabilities in the allergenic protein characterisation area, reviewed DNA approaches for food analysis and made significant progress in adopting multispectral imaging technologies to combat food fraud. Some of the themes of the completed programme will continue in the 2017-2020 programme, and you can read about this in the Impact section of this review.

During 2017 the Government Chemist team undertook a wide range of referee cases, some of them familiar, such as detection of mycotoxins in nuts and nitrofurans in shellfish, but also some novel cases such as *Mitragyna speciosa* (kratom). The scientific approach underpinning the resolution of these cases is detailed in Section 2, 'Using our scientific expertise to resolve disputes'.

In each review we reflect on the long standing role that the Government Chemist has played in the UK, and the way it has strived to continue to offer a valuable service to stakeholders. During the year in review LGC, the organisation hosting the Government Chemist role, celebrated 175 years of existence. And while it is tempting to look back and revisit pivotal moments, it is the future that the Government Chemist, his team and the Programme Expert Group look towards to identify and prepare for forthcoming challenges.

Preparing for exiting the EU, the consumer as an analyst, and evolving consumer behaviours are some of the challenges looming on the near and far horizons. To this end, we prioritised a number of projects to build capabilities in key areas such as portable technologies, exploitation of Nuclear Magnetic Resonance (NMR) and PCR technologies for food authenticity purposes, and advanced mass spectrometry techniques for mycotoxins.

This review demonstrates the excellent science carried out to address these measurement challenges and how it underpins UK food law. The Government Chemist and his team endeavour to make well timed use of available expertise to continue to provide a valued service to the UK public. I hope you enjoy this review.

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

1 WHAT WE DO

The Government Chemist role was created to help in the protection of the public from fraud, malpractice and harm. In 1875, the laboratory was appointed as ‘referee analyst’, a role linked to the Sale of Food and Drugs Act of that year. The role continues to this day.

The Government Chemist has always used 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 respond to future issues as and when they arise.

The Government Chemist fulfils statutory and advisory functions, both of which are funded by the Department for Business, Energy and Industrial Strategy (BEIS).

Our 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 10) 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 is in the resolution of disputes between regulators and businesses and is based on our independent measurements, interpretations and expert opinions. A successful resolution often avoids recourse to legal process which reduces the burden on public finances. Many of these cases are important and can have a significant impact on either or both parties, as well as potential consequences for industry and regulation in general. Our credibility as the referee, and our ability to develop new capability for future challenges, rest on first-class science which is underpinned by the assignment of our home laboratory, LGC, as the National Measurement Laboratory and Designated Institute for chemical and bio-measurement.

There are several routes for referral to the Government Chemist. The main route is the Food Safety (Sampling and Qualifications) (England) Regulations 2013 (and their equivalents in Scotland, Wales and Northern Ireland), which are invoked for many of the dispute resolution activities we undertake. These regulations state that all formal test samples are divided into three parts by an authorised officer. The enforcement authority and Food Business Operator (FBO) – ‘the trader’ – each receive one of these samples to perform independent analyses, while the third part of the sample is retained in case there is a dispute requiring the Government Chemist to act as referee.

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.

FBOs may also, in some circumstances, request a referral to the Government Chemist without having their own portion of the sample analysed. This procedure is known as ‘second (formerly supplementary) expert opinion’ (SEO) and is described on our

website.[1] 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. Lastly, the referral sometimes comes from the court itself, with proceedings suspended pending the outcome.

The Government Chemist also acts as a source of advice for government and the wider analytical community and in some instances we are asked by government to resolve a dispute when a formal sample has not been taken.

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 are disseminated to all parties so that lessons are shared, which if taken on board should help reduce the possibility of recurrence.

Dissemination of referee cases also takes place through scientific publications, the Government Chemist conference, seminars, workshops, training events and via our website.[2]

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

The need for referee analysis is often greatest in areas where measurements are difficult, where novel products are being introduced into the market, or where there is high public and media interest, for example allergen detection. New methods need to be developed and validated to accommodate that need. 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. The outputs of these studies are disseminated publicly and stakeholders, particularly in the analytical community, have access to new developments which can help them in their statutory work and hence prevent referrals to the Government Chemist. However, these cannot predict every possible referee case, and method development and validation is still necessary on an ad hoc basis.

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

Fulfilling our advisory function

The long history of the Government Chemist function and its involvement in regular and wide-ranging dispute cases means that the team is well placed to provide advice on analytical science implications on matters of policy, standards and regulations. Hence, when LGC was privatised in 1996, an agreement was signed with the Secretary of State for Trade and Industry which supported the continuity of the Government Chemist's public functions, including the provision of advice. This agreement continues to this day and serves to highlight the importance of chemical and bio-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 adequately is even greater.

The advisory function is fulfilled in the main by responding 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. Consultation responses are published on the Government Chemist website.

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 also published on the Government Chemist website.

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

Governance of the Government Chemist role

BEIS funds the Government Chemist programme to enable delivery of statutory casework, scientific advice and any R&D work necessary for the ongoing effectiveness of the Government Chemist's functions. Within BEIS, responsibility for both the Government Chemist and the wider UK National Measurement System lies with the International, Science and Innovation Directorate.

BEIS 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 internal structures and procedures in place to ensure no conflicts of interest arise between work carried out under the statutory function and its commercial activities. The GCPEG 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 BEIS regarding future priorities, which feeds into the programme strategy and formulation process. It meets twice a year to oversee and discuss the delivery, planning and quality of the programme, and also has oversight of the scientific standards of the programme. The GCPEG is tasked by BEIS 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 progress of the current projects in meeting technical milestones and targets;
- The formulation and prioritisation of new projects to maintain and develop the capabilities needed to discharge the Government Chemist 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.

[1] https://www.gov.uk/guidance/submit-a-supplementary-expert-opinion-sample
[2] https://www.gov.uk/governmentchemist

The GCPEG membership for 2017 is given below.

Paul Berryman (Chair)

Paul is the Director of Berryman Food Science Ltd, which works closely with government and businesses, including the Department for International Trade (DIT), Innovate UK and SGS Ltd. He is a visiting Professor at the University of Reading. 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. An Expert Witness and former Public Analyst, he holds the MChemA, an MBA and a PhD in Science Strategy. He was also CEO and Research Director at Leatherhead Food Research Ltd.

Robbie Beattie

Robbie is appointed as Public Analyst, Agricultural Analyst and Food Examiner to nine local authorities in Scotland. He leads 48 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. He is currently a senior manager with The City of Edinburgh Council where he manages a portfolio of income generating assets.

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 Royal Society of Chemistry (RSC) LGC advisory committee and the RSC Science and Technology Board.

Andrew Damant

Andrew leads the Surveillance, Methods and Laboratory Policy Team at the Food Standards Agency and is responsible for the Agency's surveillance strategy, 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.

Kirsty Dawes

Kirsty is a specialist in imported food, 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 in 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 RSC Analytical Chemistry Trust Fund.

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 Defra in 2009. 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.

Jonathon Griffin

Jonathon 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 MChemA in 2002 and became a Public Analyst. He continues to work as Public Analyst and Technical Manager for Kent Scientific Services. Jonathan became President of the Association of Public Analysts (APA) in 2015, representing them in discussions with central and local government bodies and chairing the Council of the Association.

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. He is currently the Interim Associate Dean for Research for the Faculty of Science, Engineering and Computing.

David Pickering

David is the Trading Standards Manager for the Buckinghamshire and Surrey Trading Standards Service. David qualified as a Trading Standards Officer in 1989 and has been part of and managed teams dealing with food, animal feed and animal health throughout that time. He has been the Chartered Trading Standards Institute Lead Officer for food for over 16 years and represents the profession on numerous groups including the national Food Standards Focus group. He has a law degree and a Master of Laws (LLM) in European Law.

Sophie Rollinson

Sophie is the Food Science lead in Defra's Food and Farming Directorate and manages the Department's Food Authenticity Research Programme. She has worked as a scientist in government since 2003 in the areas of food standards and labelling, and microbiological food safety at Defra and the Food Standards Agency.

Roger Wood OBE

Roger, after being appointed as Chief Chemist at a Public Analyst and Consulting Chemist practice, moved to the then Ministry of Agriculture, Fisheries and Food and completed his MChemA, both in 1974. Roger is an experienced food analysis specialist, who recently retired from the Food Standards Agency. 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 and has been Chair of a number of international food analysis working groups.

Government Chemist programme: priorities and progress

In the first quarter of this year, the Government Chemist programme (2014-2017) was successfully completed with all objectives met and outputs fully delivered. At the time of producing this review, the current Government Chemist programme (2017-2020) is nearing completion of its first year and there has been a good start, with work being delivered across all projects.

There have been a series of highlights with regards to referee casework, the assessment of novel methods, extension of our dissemination activities via collaboration and greater stakeholder engagement, which are all covered in this review.

Our capability building research utilises a broad range of expertise which will 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. In the 2017-2020 Government Chemist programme, projects to develop further our capabilities to ensure food safety and authenticity were commenced. In particular, novel molecular methods including Next Generation Sequencing (NGS) are being evaluated and high accuracy digital PCR is being further developed and applied. We are continuing to exploit cutting edge separation science and mass spectrometry technologies for the analysis of protein allergens and mycotoxins, building on work completed in previous programmes.

The Government Chemist horizon scanning activities identified NMR as an approach to be developed to underpin our capability in food authenticity and therefore we have started a capability building project in this space. This work will place us in a good position to respond to potential issues that require the wider analysis of metabolites such as profiling methods to determine exogenous sugars in honey. Honey authenticity remains a very topical global issue as shown by outcomes of the EU Coordinated Control Plan on honey which indicated that 38% of the 2,264 honey samples examined in the EU were non-compliant with authenticity criteria.[3]

Horizon scanning by the GCPEG also identified the need for the Government Chemist to review rapid and point-of-use technologies which may generate referee work in the medium and long term future. Two key exemplar disruptive technologies – multispectral imaging (MSI) and ambient ionisation coupled to mass spectrometry (MS) – are being assessed in the current programme. Scoping of a report to provide details and recommendations on what would be required to develop MSI as a point-of-use test device has been initiated, and initial work on ambient MS approaches has identified a number of potential applications in the rapid determination of food authenticity and adulteration including oils, honey and whisky.

The 2017-2020 programme includes additional stakeholder engagement which reflects the increased call for Government Chemist scientists to provide expert opinion and to lead or contribute to stakeholder led committees. This contribution is invaluable in disseminating the work of the Government Chemist programme. We seek to maintain this meaningful dialogue with stakeholders and regulators in areas which link measurement and regulation so that new policy, standards and legislation are based on sound measurement science.

Our stakeholder engagement highlights this year include:

- Malcolm Burns organised and co-chaired a workshop on DNA extraction for GMOs in collaboration with the EU Reference Laboratory for GM Food and Feed (EU-RL GMFF);
- Michael Walker made several contributions at a workshop in Vienna on allergen management and advanced testing organised by Romer Labs;
- Selvarani Elahi gave a lunchtime lecture at the European Commission's (EC) Joint Research Centre (JRC) in Geel, Belgium, titled 'Dispute Resolution & Fighting Food Fraud – UK Style'

The dates for the next Government Chemist conference have been set for 13-14 June 2018 at the BMA (British Medical Association) House, London.

In developing the Government Chemist programme for 2017-2020, a comprehensive horizon scanning and stakeholder consultation process was executed which resulted in the revision of the Government Chemist strategy document. Whilst the key strategic aims of the Government Chemist remained the same, the UK's exit from the European Union was identified as a new factor on the horizon that will likely affect enforcement, standards and regulatory compliance in the future. The policy paper[4] 'Legislating for the United Kingdom's withdrawal from the European Union' was updated in May 2017 with more detailed text on EU legislation, including treaties, that will be converted into domestic law on the day the UK leaves the EU, subject to the exceptions set out in the paper. There is no explicit reference to food or feed but, for example, legislation may refer to the involvement of an EU institution or be predicated on UK membership of, or access to, an EU regime or system. Once the UK has left the EU, this legislation will no longer work and steps must be taken to ensure that the domestic statute book continues to function. Moreover, EU Directives require domestic implementation which would fall away if the European Communities Act under which much of the conversion has been done, was simply repealed. Thus it is proposed to create a power to correct the statute book where necessary over time. Some legislation will necessarily need to await the conclusion of negotiations with the EU. Developments in UK policy continue to be closely monitored via the Government Chemist horizon scanning activities.

The Government Chemist continues to receive positive feedback on the quarterly food and feed legislation reviews that are published on the Government Chemist website and they remain the most downloaded of all the Government Chemist publications. This is an example of feedback received:

[3] https://ec.europa.eu/food/sites/food/files/safety/docs/oc_control-progs_honey_jrc-tech-report_2016.pdf
[4] Department for Exiting the European Union, https://www.gov.uk/government/publications/the-repeal-bill-white-paper/legislating-for-the-united-kingdoms-withdrawal-from-the-european-union

"I just wanted to reiterate how valuable the Food and feed law: Compendium of UK food and feed legislation quarterly reports are for me. They provide a very useful structured overview of changes and developments, but most importantly they provide context and rationale for changes and development in legislation – parochially for the UK, but also for the wider global food and feed industry".

People

LGC staff who directly support the Government Chemist function have clear 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 for Public Analysts,[5] 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 also carry out R&D work, often involving international collaboration, which gives them the capability to contribute positively and efficiently to their work.

Areas of collaboration with stakeholders

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 knowledge or equipment needed to address them may not be readily available within the broad range of activities undertaken at LGC. We are therefore alert to the possibility 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.

During 2017, it was recognised by stakeholders that internationally accepted definitions are important and would facilitate global action to tackle challenges related to food authenticity and food fraud. Thus three new initiatives to standardise terms used to describe food authenticity and related terms were initiated:

- The Codex Committee on Food Import and Export Inspection and Certification Systems (CCFICS) and the Codex Alimentarius Commission agreed to establish an electronic working group (eWG) to take forward new work on clarifying definitions for key terms relating to food integrity and food authenticity. The eWG is being chaired by the Islamic Republic of Iran and co-chaired by Canada and the European Union. The Government Chemist is inputting into the eWG via the UK competent authorities for food authenticity (Department for Environment, Food and Rural Affairs (Defra) and Food Standards Agency (FSA)).
- Two European Committee for Standardisation (CEN) initiatives:
  - Development of a new CEN working agreement (CWA) to standardise terms and definitions relating to food and feed authenticity. Norway is coordinating the development of the CWA, as part of the EU funded Authent-net project, with the aim of publishing the final document during 2018. Government Chemist staff inputted into the kick-off meeting that was held in May, attended a meeting at CEN in June and are members of the electronic workspace on which the document is being developed.
  - The CEN Technical Board approved the creation of a Coordination Group on Food Authenticity (FACG) in order to coordinate between different Technical Committees in the field of food and feed. Government Chemist staff attended the kick-off meeting in Brussels in June at which it was agreed that the aim of the Group is the standardisation of methods for testing the authenticity of food and feed. Government Chemist staff will continue to participate in this group. Developments at CEN can be followed on the CEN website.[6]

The Government Chemist team met representatives of the International Atomic Energy Agency (IAEA) to give a demonstration of an ambient transportable MS system currently being assessed for food authenticity and quality applications under the Government Chemist programme. IAEA seek to build capacity in developing countries using transportable technologies. A follow-up meeting will take place in 2018 to discuss possible collaboration opportunities on transportable technologies.

The Government Chemist is collaborating with Manchester University in allergen research where we benefit from access to the latest developments in this very topical and complex area. Following recognition of problematic current test methods for food allergens and the lack of adequate reference materials to promote method validation, LGC successfully responded to a call for further research by FSA. Leading a consortium of Manchester University, Institute of Biotechnology and Romer Labs we will work on several related topics. These include a systematic review of allergen analytical targets to create an open access repository of reliable markers and, guided by stakeholders, preparation of a reference material kit containing (a) a food matrix shown to be devoid of the target allergens, (b) a food matrix incurred with five priority allergens and (c) the raw material allergens. The kits will be checked for homogeneity, stability and concentrations of allergens in the incurred material, and released for sale with supporting documentation. We will disseminate knowledge gained to encourage use of the reference material to achieve tangible improvements in allergen analysis,

The Government Chemist team has successfully negotiated a joint, cross government (BEIS, FSA, Food Standards Scotland (FSS) and Defra) three year knowledge transfer project that will

[5] All work is overseen by Michael Walker, a nominated officer holding the statutory MChemA qualification
[6] https://standards.cen.eu/

deliver knowledge from government programmes to stakeholders to help enforce current regulations and prevent future disputes. By pooling funds from each of the four government departments, knowledge transfer events can be planned and coordinated according to priorities received directly from the stakeholder community delivering greater impact.

The programme for 2017-18 has been agreed and is as follows:

i. Workshop on Allergen Detection in Spices

This workshop will provide advice, guidance and best practice on the adoption of a multidisciplinary approach for the detection of allergenic proteins in spices, which could be implemented if incidents similar to those seen with the almond in paprika, and mahaleb in cumin cases were to occur in the future.

ii. e-Seminar: Advanced DNA Techniques: An introduction to dPCR

Digital PCR (dPCR) is an advanced molecular biology method that can provide absolute single molecule detection without reference to a calibration curve. Costs for dPCR are decreasing, making instrumentation and methods more affordable. This e-seminar will provide an introduction to dPCR, covering the advantages, limitations and scope of the technique, as well as available instrumentation and best measurement practice guidance.

iii. e-Seminar: Advanced DNA Assays: An introduction to advanced qPCR assay design and optimisation

Efficient primer and probe design are upstream elements which are instrumental in the success of any quantitative real-time PCR (qPCR) experiment. Equally, being able to effectively optimise a qPCR experiment in-house can maximise the likelihood of robust, specific and sensitive assays being produced. This e-seminar will provide an introduction to real-time PCR, covering different dye and probe based systems, primer and probe design and purchase, and guidance on qPCR assay optimisation.

The e-Seminars will be published on the Food Authenticity Network's[7] training pages.

For more information on our work, please contact us at government.chemist@lgcgroup.com or go to the website https://www.gov.uk/governmentchemist.

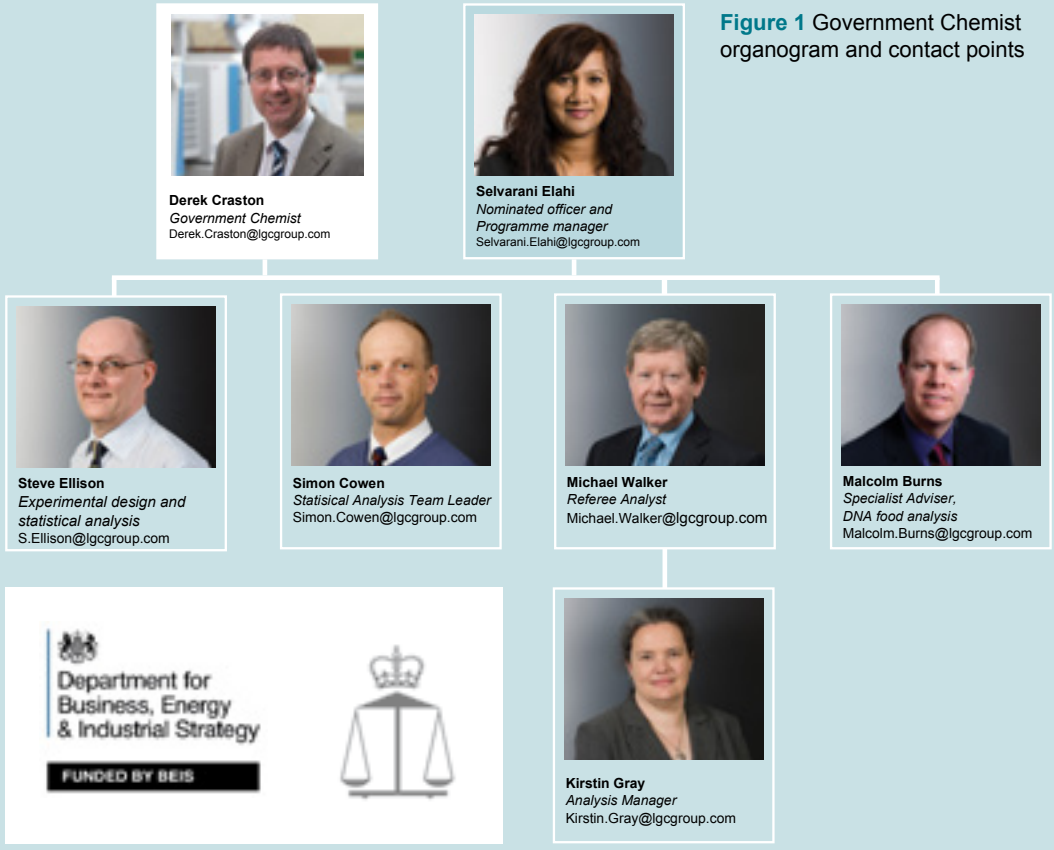


Figure 1 Government Chemist organogram and contact points

[7] http://www.foodauthenticity.uk/, a free on-line resource developed by UK government to help bring together those involved in food authenticity testing. The network's aim is 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.

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 2015[1]
Genetically Modified Animal Feed Regulations 2004[1]
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 USING OUR SCIENTIFIC EXPERTISE TO RESOLVE DISPUTES

Referee cases – resolving disputes in the UK official control system for food and feed – is a demand led service which has been at the core of the Government Chemist's function since 1875. Publishing the outcomes in our annual reviews and in more detail in peer reviewed scientific papers contributes to avoiding similar disputes in the future. Referee cases arise most frequently under the Food Safety Act 1990 and sometimes under the Agriculture Act 1970.

Overview of referee cases in 2017

During 2017 demand increased from 2016 (see Figure 1). Two cases from 2016 were resolved and eleven cases were newly referred to the Government Chemist, all in connection with food. Table 1 gives further information. The absence of referrals from inland authorities and on animal feed, although overall numbers are small, may reflect reduced sampling activity rather than the absence of potential for disputes to arise. Most of the problems referred to us and concluded in 2017 were familiar – mycotoxin contaminants, food additives, genetically modified food and jelly mini-cup choking hazards – but still complex in their measurement requirements. One new question originally posed to us in 2016, the status of the powdered leaf of *Mitragyna speciosa* also known as kratom, was concluded in the courts in 2017 and can now be reported.

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.

Table 1 Overview of referee cases in 2017

Origin			Basis		
Inland Authority	0	0 %	Dispute	9	82 %
Port Health Authority	11	100 %	Other*	2	18 %

*** Other includes SEO – Second Expert Opinion, pursuant to Article 35 of Regulation 2017/625 on official controls, and requests for assistance from other Government Departments or Local Authorities.**

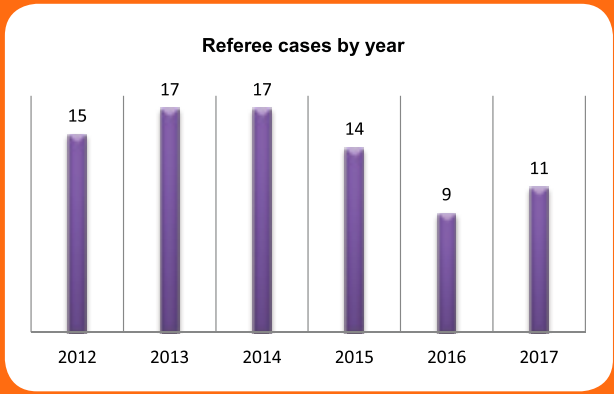


Figure 2 Referee cases by year

There is no legal definition of the referee analyst function. We regard it as independent expert analysis, including interpretation if necessary, to help avoid or resolve disputes. There are statutory provisions for referral of retained portions of formal[8] samples to the Government Chemist in regulations made under both the Food Safety Act 1990 and the Agriculture Act 1970.[9]

The statutory conditions for referral begin with the contemplation or commencement of legal proceedings where the prosecution intends to offer 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. If the above route is not open to a trader they may request a SEO pursuant to Article 35 of Regulation 2017/625 on official controls and in defined circumstances SEO may be requested of the Government Chemist.

Hence referee casework arises by a variety of routes. In some instances we are asked to resolve a dispute when a formal sample has not been taken. These we deal with on a case by case basis either accepting a portion of the original informal sample or offering to comment on any apparently conflicting analytical results from the informal sample and other relevant data. However where it seems best to do so we advise a further formal sampling exercise which ensures all parties receive properly sampled and divided parts of the same batch of food or feed.

Analytical results must be interpreted in an increasingly global supply chain and often in complex scientific legal and policy contexts. When a referral is received we begin with a case meeting to examine the problems associated with the case and often instigate a brief literature review of the topic. Since few referee cases are routine our analytical methods may need to be investigated and modified to deal with particular problems. Where possible, orthogonal confirmation of the outcomes is applied.

Our default analytical strategy is multi-replicate analyses on multiple days. The extent of replication, together with analysis of reference materials (certified, where available), blanks and spiked blanks and/or sample aliquots, practically amounts to a stand-alone method validation, and provides the necessary high level of analytical confidence. All significant analytical steps are witnessed by a second scientist, all data transcriptions and calculations are checked with the results 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 (or Deputy) for peer review. If all steps are satisfactory the Government Chemist (or Deputy) will allow the findings to be released.

Thus the analysis of the sample referred to the Government Chemist is much more than simply a routine repeat test and the resource expended on each case is considerably more than would be available to an official control or trade laboratory at first instance. This is necessary for a number of reasons: (a) the results and opinion produced by the Government Chemist must be definitive and bear detailed scrutiny, sometimes at national and international level; (b) referrals are usually on matters close to a legislative limit where analytical confidence in our data must be of the highest standard; and (c) the problems we seek to resolve occur where the science, the law or both are uncertain or complicated.

Mycotoxins

Mycotoxins are naturally occurring secondary metabolites produced by moulds. Given their toxicity, and the propensity for some to cause cancer, stringent controls are in place to reduce human consumption.

The occurrence of mould in any food is patchy and consignments can be large. Thus there are statutory requirements for multiple increments, sometimes up to 100, to be sampled and thoroughly mixed before analysis. Samples are then prepared by high speed slurrying with water to aid homogenisation. We have investigated the effectiveness of the sampling and slurrying protocol for the determination of aflatoxins in a consignment of groundnuts (peanuts) in shell. Following six replicate sampling exercises each laboratory set of samples (enforcement, defence and reference) was analysed in our laboratory for aflatoxins in a manner suitable for detailed statistical interpretation. The results demonstrated that the protocol is effective and that when properly followed the results for the three laboratory samples derived from the sampling exercise should be equivalent. The study was published in the open access Journal of the Association of Public Analysts.[10]

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 2017 there were disputes about the concentrations of aflatoxins in two separate imported consignments of peanuts. Aflatoxins, mainly produced by the moulds *Aspergillus flavus* and *A. parasiticus*, are genotoxic carcinogens capable of inducing liver cancer – particularly with simultaneous hepatitis B virus infection – and are among the most potent mutagens known. There are many forms of the aflatoxin molecule but four are common and regulated – B_1, B_2, G_1 and G_2. There are limits set for aflatoxin B_1 and the sum of all four. The analytical method for the determination of aflatoxins that has stood the test of time is acetonitrile/water extraction, dilution in phosphate-buffered saline and immunoaffinity column clean-up, followed by liquid chromatography with post column derivatisation and fluorescence detection. We also seek orthogonal confirmation of the molecular identity and presence of aflatoxin B_1 by liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) of sample extracts, together with solvent standards, and pre-extraction and post-extraction matrix spikes.

For both the aflatoxin cases we upheld the Public Analysts' findings and the consignments were prevented from entering the UK food chain, safeguarding consumers from exposure to cancer-causing toxins. Figures 3 and 4 show the results, which must take into account analytical recovery, the slurry ratio and measurement uncertainty. The lower bound of the 95 % confidence interval is the datum to compare against the statutory limit. When a consignment is large two samples must be taken and each split into three parts. Hence

[8] 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.

[9] 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

[10] Walker, M., Colwell, P., Cowen, S., Ellison, S.L.R., Gray, K., Elahi, S., Farnell, P., Slack, P. and Burns, D.T., Aflatoxins in groundnuts – assessment of the effectiveness of EU sampling and UK enforcement sample preparation procedures, J Assoc. Public Anal., 2017, 45, 1-22

in Figures 3 and 4 each laboratory has reported on two samples from the same consignment and the consignment is non-compliant if any one or both of the samples exceed the limit. Thus, for example in Figure 3, the results labelled PA 1, and GC 1, as well as both the importer's results are compliant whereas results PA 2 and GC 2 demonstrate non-compliance.

Key to data labels in Figures 3 and 4

PA 1 and PA 2 are the results provided by the Public Analyst, FBO 1 and FBO 2 are the results provided to the Food Business Owner and GC 1 and GC 2 are the results of the referee case. When a consignment is large two samples must be taken and the consignment is non-compliant if (as here) any one or both of the samples exceed the limit after taking recovery, measurement uncertainty and the slurry ratio into account.

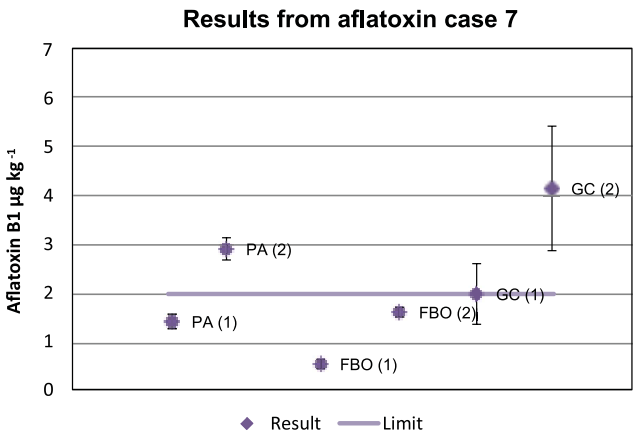


Figure 3 Results of aflatoxin case 7

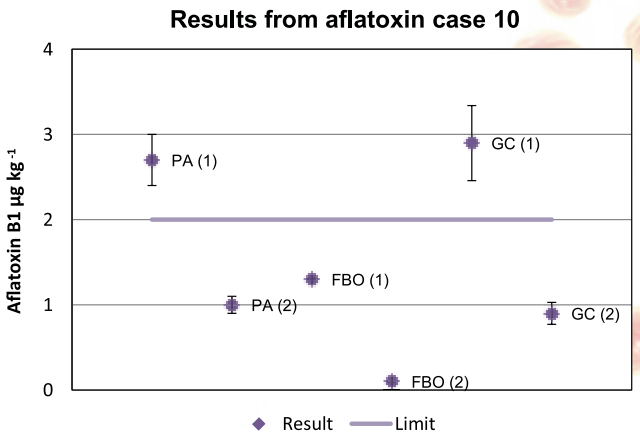


Figure 4 Results of aflatoxin case 10

Allergens and sulphites

Food allergy is a major public health concern. There are well-documented detriments to the quality of life for allergic consumers and their families, and about ten food-related anaphylaxis deaths (and potentially more near misses) every year in the UK. There are also a significant number of food allergen-related incidents and food recalls with a concomitant impact on food businesses. During 2017 we made further substantial contributions in this area with published work on allergen risk management[11,12] methods for allergen analysis,[13] the proteomics of almond and mahaleb in cumin and paprika,[14] and an overview of the complementary approaches we deployed in those cases.[15]

The focus on sulphites, the only non-protein allergen group, described in the 2016 review continued during 2017. Sulphites are a very useful group of additives with antioxidant and antimicrobial properties. They inhibit a wide range of browning reactions in food and are widely used in the food industry to preserve food quality and appearance.[16] However in the 1980s, reports emerged implicating sulphites as initiators of asthmatic reactions in small subsets of the population, on occasions with fatal outcomes, and there have been numerous reports of sensitivity or intolerance reactions in humans exposed to sulphited foods and beverages. Risk management for sulphite sensitive consumers depends on the labelling disclosure required in the Food Information Regulation (Article 21 and Annex II of Regulation 1169/2011). For the general population an acceptable daily intake of sulphites in view of their ubiquity of use, destruction of thiamine and other vitamins and the potential to disguise decay in food is managed by a permitted list of foods and compliance with maximum permitted limits.

Sulphited foods in imports are regularly monitored at the EU level on foot of Commission Regulation 669/2009 which requires increased sectoral scrutiny when evidence of threats to the food chain appears.

[11] Walker, M.J., Chapter on 'Food allergy: managing food allergens', Analysis of Food Toxins and Toxicants Analysis of Food Toxins and Toxicants (Yiu-Chung Wong, Richard J Lewis), 2017, 711-742, ISBN: 978-1-118-99272-2N

[12] Walker, M.J., Gowland, M.H. and Points, J., Managing food allergens in the UK retail supply chain, J AOAC Int., 2018, 101,1,14-55, DOI:10.5740/jaoacint.17-0385, (Epub Dec 2017)

[13] Groves, K., Cryar, A., Walker, M. and Quaglia, M., Assessment of recovery of milk protein allergens from processed food for mass spectrometry quantification, J AOAC Int, 2018, 101,1,152-161, DOI: 10.5740/jaoacint.17-0214, (Epub Dec 2017)

[14] Inman S.E., Groves, K., McCullough, B., Quaglia, M. and Hopley, C., Development of a LC-MS method for the discrimination between trace level Prunus contaminants of spices, Food Chem., 2017, 245, 289-296, DOI: 10.1016/j.foodchem.2017.10.101

[15] Walker, M.J., Burns, M., Quaglia, M., Nixon, G., Hopley, C.J., Gray, K.M., Moore, V., Singh, M. and Cowen, S., Almond or mahaleb? Orthogonal allergen analysis during a live incident investigation by ELISA, molecular biology, and protein mass spectrometry, J AOAC Int, 2018, 101, 1, 162-169, DOI: 10.5740/jaoacint.17-0405, (Epub Dec 2017)

[16] Wedzicha, B.L., Chemistry of sulphiting agents in food, Food Additives & Contaminants, 1992, 9, 5, 449-459

This gave rise, in late 2016 and 2017, to four disputes on the sulphite content of imported dried apricots where the maximum permitted limit is 2000 mg kg^{-1} (as SO_2). Analysis for sulphites in food at this level is relatively straightforward with reference methods based on the Monier-Williams procedure first published in 1927. In this method, acidification of the sample dispersed in water in a multiple necked reaction flask drives the sulphite equilibrium to sulphurous acid. Gaseous SO_2 is entrained in nitrogen gas bubbled through the boiling liquid under reflux conditions. The SO_2 is trapped in neutralised hydrogen peroxide, forming sulphuric acid which is determined volumetrically against standardised sodium hydroxide solution.

Figure 5 shows the results obtained by the Public Analyst, the laboratory acting for the importer and the Government Chemist's findings. In both case 1417-37 and case 1417-38 the results from the laboratories acting for the FBO were upheld. In cases 1720-8 and 1720-9 the results from the PA laboratories were upheld. The relatively large dispersion of the results appeared to us to be primarily driven by apricot to apricot variation in the sulphite concentration since it was common practice to chop the sample coarsely for homogenisation since blending risked thermal loss of SO_2. In 2017 we decided to explore slurrying the sample with a dilute solution of formaldehyde to achieve much better homogenisation and to 'fix' the sulphite present by formation of the much more stable hydroxymethanesulphonate adduct. This appears to have considerably reduced the dispersion of the results. A medium term study is under way which, if successful, will result in a recommendation to Sampling Officers and Public Analysts to apply the formaldehyde slurrying approach prior to splitting the sample into the official control, FBO and Government Chemist portions.

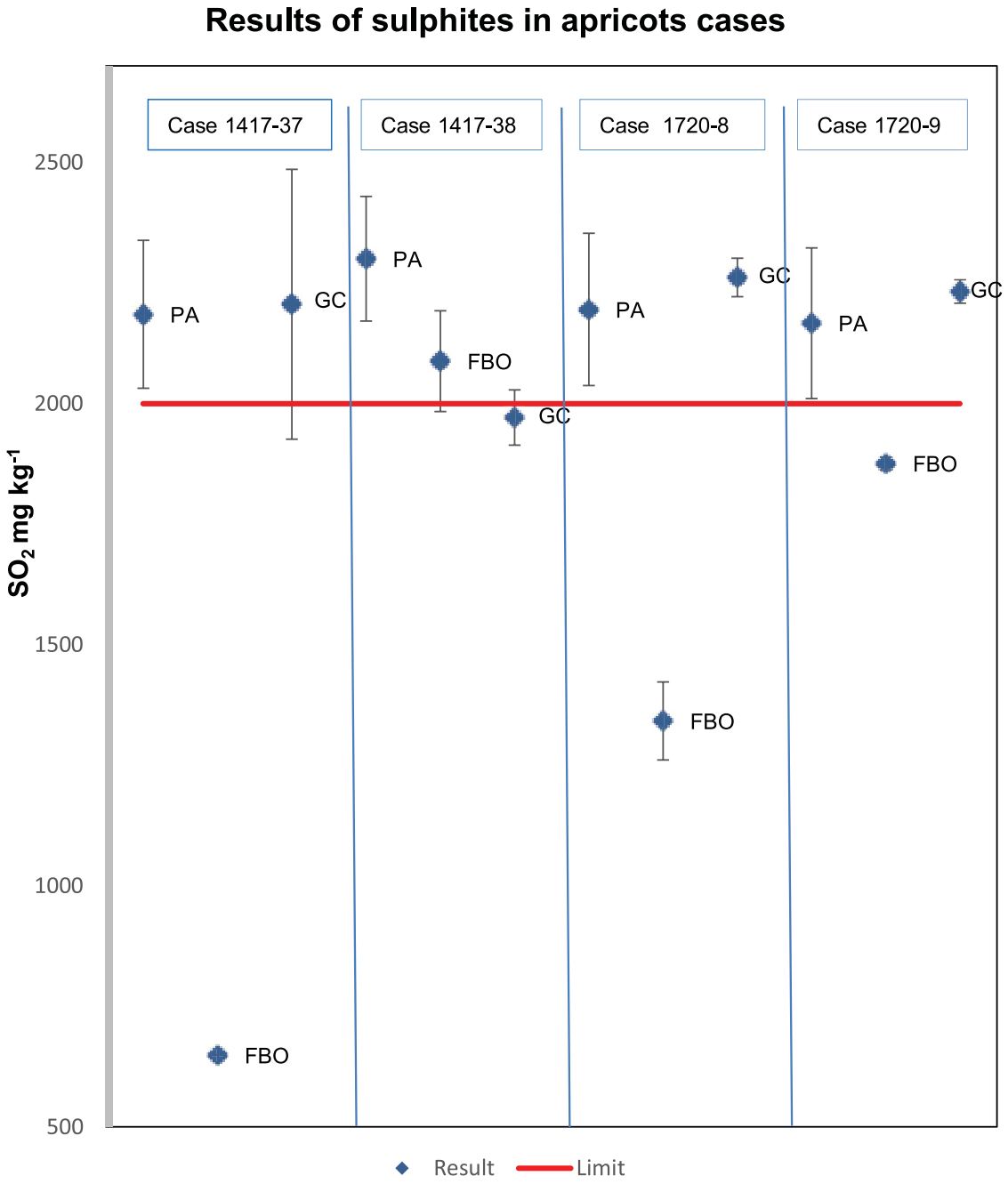


Figure 5 Sulphites in dried apricots cases – data from the Public Analyst (PA), the laboratory acting for the importer (FBO) and the Government Chemist's findings (GC) with measurement uncertainty where given by the laboratory concerned

Veterinary residues – nitrofurans

Veterinary medicines are used to treat sick animals or prevent disease in herds, flocks or aquaculture. All veterinary medicines must be assessed for safety before they can be marketed or used on animals. Where they are authorised for use limits are set (Maximum Residue Limit – MRL) and some are prohibited owing to safety concerns. The nitrofuran antibiotics fall into the latter category and are banned from use in food-producing animals in most jurisdictions. However, they are still authorised and popular for human medicine and for the treatment of non-food animals, and are widely manufactured and sold worldwide.

The five most common veterinary nitrofurans are furaltadone, furazolidone, nifursol, nitrofurantoin and nitrofurazone. Nitrofurans are rapidly metabolised in the animal and residues of the parent molecules can no longer be detected within days, if not hours, of administration. By contrast, protein-bound metabolites of four of the five most common veterinary nitrofurans have been identified which are stable for many weeks.

Test methods are therefore based upon these protein-bound metabolites, which themselves are toxicologically suspect. A further complication is that semicarbazide, the metabolite of nitrofurazone, occurs naturally in the shell of shrimps and prawns. It may also arise adventitiously from the flour treatment agent azodicarbonamide, blowing agents in plastic seals (e.g. around the lids of glass jars), the herbicide triazophos, or the action of bleach on proteins. Thus conclusive proof of administration of nitrofurazone to shrimps and prawns depends on finding tissue-bound semicarbazide in the excised core flesh of the animal. Not surprisingly this presents difficulties in sampling, homogenisation and division of samples into parts. We have discussed these issues in detail with Public Analysts and through the peer reviewed literature.[17]

Two nitrofurazone cases arose in 2017 in which the Public Analyst reported against imported shrimp consignments for the presence of semicarbazide and laboratories acting for the importer did not find this marker metabolite. Our own findings confirmed that the excised core flesh did not contain detectable amounts of semicarbazide. We published a general article explaining the control of veterinary residues in food and the difficulties that can arise in their analysis in the Institute of Food Science and Technology (IFST) quarterly journal 'Food Science & Technology'.[18]

Kratom

An interesting query that developed into court attendance arose in relation to the status of the powdered leaf of *Mitragyna speciosa* also known as kratom. Kratom, only relatively recently known in the UK, was included by the World Health Organization (WHO) among new psychoactive substances (NPS) in 2012. Recent online surveys suggest kratom is one of the most widely offered NPS on the internet. It has dose-dependent effects, producing stimulation at low doses and predominantly opioid-like effects at higher doses. Commercially available kratom products in Western countries include raw leaves, powdered dried leaves in capsules or tablets, and concentrated extracts (gum). Although there are contradictory opinions about the extent to which kratom is smoked, it is typically brewed into tea or consumed with another liquid.

The active chemical compounds isolated from kratom include over 40 structurally related alkaloids of which mitragynine is the most important. Mitragynine is largely responsible for kratom's reported analgesic effect owing to its potent opioid agonist property, however it is structurally different from morphine and other opioids. Although exhibiting dose-dependent effects, kratom has an erratic pharmacology making it difficult to define a specific dose threshold. Some beneficial effects such as analgesia have been recorded, and the potential for new therapeutic agents therefrom has been recognised but caveated with possible serious adverse effects. On ingestion mood altering effects are soon apparent, and serious conditions demonstrated after repeated administration include elevated blood pressure, nephrotoxic effects, impaired cognition and behaviour, dependence and liver failure. Fatalities where kratom was implicated along with poly-drug use have been reported.

We were called upon to assist a Magistrates' Court and subsequently a Crown Court in proceedings where an importer challenged the seizure of a consignment of kratom by a Port Health Authority. One of the questions asked was in relation to the classification of kratom as a food prior to the coming into force of the Psychoactive Substances Act 2016 on 26 May 2016. We viewed kratom leaf as a 'novel food' as defined in Regulation (EU) 2015/2283. A novel food is one that is placed on the market and that has not hitherto been used for human consumption to a significant degree within the UK or EU prior to 1997. Novel foods must not present a danger for the consumer and their sale is not permitted without assessment and authorisation. No such assessment and authorisation has taken place for kratom and in our view authorisation would not be granted, on grounds of food safety, if an application was made. On both occasions the courts accepted our evidence. The matter was further aired in the High Court which dismissed applications for a Judicial Review of the lower courts' findings.

[17] Points, J., Burns, D.T., Walker, M.J., Forensic issues in the analysis of trace nitrofuran veterinary residues in food of animal origin, Food Control, 2014, 50, 92-103

[18] Walker M. and Gray, K., Veterinary residues in food, FS&T, 2017, 31, 23-27

Jelly confectionery – a choking hazard?

Foreign body aspiration continues to be a common paediatric problem with food a major cause. Although many choking episodes resolve spontaneously, when they don't the consequences can be severe – from immediate death to brain injury owing to hypoxia. Jelly confectionery known as jelly cups, or jelly mini-cups, first came to prominence in 2001[19] 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 (Figure 6).

The Government Chemist has been involved at intervals since then in assisting the authorities and businesses to interpret legislation that seeks to control such choking risks. Briefly, food additive law bans the use of a range of gel-forming compounds in such products to avoid the possibility of 'plugging' the airway. Disputes arise, not about the presence of the additives, but about the definition of the product in which they are banned. The definition reads *"…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…"*. Although at first sight this seems straightforward it contains several elements that pose difficulties. What does "firm consistence" mean? And how can we interpret "intended to be ingested in a single bite…"? No further guidance has been issued by regulators and our paper of 2012[20] remains the only published advice on how to test a product against the definition.

In 2017 two further imported consignments of jelly confectionery were impounded on foot of adverse Public Analyst's reports and the retained samples referred to us. In each case we upheld the adverse opinions and the consignments did not enter the UK. Continued dialogue after the cases led us to advise the importers on the applicable tests and their interpretation, and that representative samples of any consignment destined for the UK should be forwarded in advance of shipping for testing in the UK by a laboratory familiar with the tests we described. To avoid a conflict of interest we declined requests to screen imports ourselves but referred the importers to the Association of Public Analysts' (APA) website to find a suitable laboratory. Further, to disseminate good practice on the topic, we published an article summarising our procedures and advice in in the IFST quarterly journal 'Food Science & Technology. [21]

Figure 6 Jelly mini-cups

[19] http://www.telegraph.co.uk/news/uknews/1365379/Sweet-alert-after-16-choke-to-death.html

[20] Walker, M.J., Colwell, P., Craston, D., Axford, I.P. and Crane, J., Analytical Strategy for the Evaluation of a Specific Food Choking Risk, a Case Study on Jelly Mini-Cups , Food Analytical Methods, 2012, 5, 54-61

[21] Walker M. and Gray, K., Safer sweets – choking risks from jelly confectionery and technical appeals to the Government Chemist in this area, FS&T, 2017, 31, 40-43

Genetically Modified Organisms (GMOs)

EU law[22] prohibits the placing on the market of genetically modified (GM) food or feed unless it is officially authorised, and provides for its labelling and supervision. Authorisation is only granted after demonstration that the GM food or feed does not have adverse effects on health or the environment and that it does not mislead the consumer. In addition the GM food must not differ from the food it is intended to replace to such an extent that its normal consumption would be nutritionally disadvantageous.

In 2017 we dealt with two referee cases involving rice products from China. There are no genetically modified rice products authorised in the European Union[23] but, from 2006 onwards, some rice products originating in or consigned from China, were discovered to be contaminated with the genetically modified rice Bt 63. Officially known as Bt Shanyou 63, this is a type of rice that has been incorporated with genes from the soil bacterium *Bacillus thuringiensis*. These newly introduced genes encode for insecticidal proteins known as Bt proteins which are toxic to rice pests and therefore make the crop resistant. The Chinese authorities took steps to control the presence of GM rice, but GM varieties, such as Bt 63 and others, continued to be found. As a consequence, the EU requires rice imports from China to be accompanied by an analytical report demonstrating the absence of GM rice.

From December 2011 all rice imports from China have been subject to inspection, sampling and analysis. Owing to the lack of detail of the full DNA sequences of genetically modified rice varieties available in China, a screening approach is adopted for certain generic genetic elements. GM plants are generally produced by inserting a transgenic sequence that encodes for a desired trait into the host genome. The trait sequence is typically bounded by regulatory promoter and terminator sequences, some of the most common being the 35S promoter (P35S) derived from Cauliflower Mosaic Virus (CaMV) and the nopaline synthase terminator (TNOS) derived from *Agrobacterium tumefaciens*. Thus P35S and TNOS are useful screening targets. Further screening targets are the genes encoding for the *Bacillus thuringiensis* endotoxin CrylAb/Ac.

The most common chemistries used to produce a signal (Cq) after amplification by PCR include the use of specific fluorescent probes (Taqman®) or DNA binding fluorescent dyes (e.g., SYBR® Green I). The commonly used fluorescent SYBR® Green dye binds to the minor groove of DNA, but may also bind to nonspecific PCR products and primer dimers and therefore is not sufficiently specific. Melting curve analysis, performed by observing the change in fluorescence as the double stranded DNA dissociates, allows for the distinction of nonspecific fragments from specific PCR products.[24] In the SYBR® Green assay the target is considered detected, according to EU-RL GMFF guidance, when *paired duplicate* extractions both give a signal for detectable amplification (Cq) accompanied by a melting temperature (T_m) that is within 1.5 °C of the T_m of the positive controls.

Generally, multiple replicates of the samples, alongside positive and negative controls, are analysed on multiple days. A real-time PCR assay for a rice taxon-specific phospholipase D (PLD)[25] is used for the detection of GM rice. Two real-time PCR instruments from separate manufacturers are deployed and interpretation of results is based both on instrument default automatic threshold settings and expert judgement of amplification curves and melting temperature plots. Where required and applicable, confirmatory procedures such as those of the GMO National Reference Laboratories of Germany[26] are applied. The Government Chemist team developed and published an in-house plasmid control for CaMV to aid detection of GM Rice Lines in 2013.[27]

The Government Chemist benefits from the synergy between our molecular biology team, and the UK National Reference Laboratory for GMOs, both hosted by LGC and led by Malcolm Burns. Additional synergy is brought about by access to the European Network of GMO Laboratories (ENGL) – 95 national enforcement laboratories from all EU Member States plus Norway, Switzerland and Turkey. Best practice is discussed within ENGL with referee casework contributing to advancing knowledge throughout the membership.

In the first GMO case in 2017 the Public Analyst reported the presence of the P35S promoter sequence. This result was initially challenged by the laboratory acting for the importer. However, we were able to resolve the issue by posing a series of questions to the laboratories involved after which the importer's laboratory reversed their opinion and the consignment was prevented from entering the UK.

In the second case the Public Analyst reported the presence of both the P35S promoter and the CrylAb/Ac sequence. The laboratory acting for the importer reported none of the marker sequences detected. However the Government Chemist's findings demonstrated the presence in the sample of the 35S promoter sequence and the TNOS terminator sequence, although the CrylAb/Ac sequence was not found. Accordingly the consignment was prevented from entering the UK. We believe that the amount of GM material present in each case was likely to be very low and towards the limit of detection of the assays. This trace level detection is borne out in the second case by the variability with which the target DNA molecules were detected, despite excellent repeatability of the negative and positive controls. Such variability, which may have had contributions from instrument sensitivity, is characteristic of stochastic (random) variability of targets at very low concentrations.

[22] Regulation (EC) No 1829/2003 of the European Parliament and of the Council of 22 September 2003 on genetically modified food and feed https://eur-lex.europa.eu/legal-content/EN/ALL/?uri=CELEX%3A32003R1829

[23] Commission Decision 2011/884/EU Recital 8.

[24] Broeders, S.R.M., de Keersmaecker S.C.J., and Roosens, N.H.C., How to Deal with the Upcoming Challenges in GMO Detection in food and feed, Journal of Biomedicine and Biotechnology, 2012, Article ID 402418

[25] Mbella, M., et al., SYBR® Green qPCR methods for detection of endogenous reference genes in commodity crops: a step ahead in combinatory screening for Genetically Modified Crops in food and feed products, Eur. Food Res. Technol., 2011, 232:485-496

[26] Bundesamt für Verbraucherschutz und Lebensmittelsicherheit, Guideline detection of genetically modified rice, 2012

[27] Burns, M., Nixon, G., Walker, M., Busby, E., Development of an in-house Plasmid Control for Cauliflower Mosaic Virus (CaMV) for the detection of Genetically Modified (GM) Chinese rice lines, J Assoc Public Analysts (Online), 2013, 41, 45-52

Ongoing areas of concern and interest

Whilst there might not be referee cases in every area of concern every year, the Government Chemist keeps a watchful eye, and up-to-date scientific capabilities, on areas where cases or enquiries could arise in the near future. Additionally, the Government Chemist is called upon to provide advice for government and the wider analytical community. During 2017, we continued activity in the food authenticity area and provided advice on tolerances for alcohol declarations.

Food authenticity – honey and chondroitin

Food authenticity – food sold which is of the nature, substance or quality demanded by the purchaser and accurately matches its description or labelling – is important to consumers, industry and regulators. Mis-description or mislabelling of food is illegal, potentially harmful, penalises the honest trader, and undermines consumer choice and value for money. When driven by financial gain it is 'food fraud', and when complex, causing serious harm, or involving organised criminals it is 'food crime'. Authenticity is primarily determined by documentation, traceability and audit, although this can be difficult and time consuming especially with imported food. In many instances verification of the composition, origin and processing of food can only be accomplished by analytical means. This is, however, often challenging and has harnessed state-of-the-art methods in genomics, metabolomics, spectroscopic and stable isotopic and trace element measurement to achieve its aim.

Food authenticity has been a constant feature of Government Chemist work from the inception of the function, and in 2016 we were asked to look into the authenticity of honey. Recognising the work on honey authenticity underway at other institutions, e.g. the Joint Research Centre (JRC) Geel, and by the New Zealand authorities in relation to a definition of Manuka honey, we maintained active interest in this area by contact with both institutions. The Government Chemist horizon scanning activities and the GCPEG identified LGC's expertise in NMR as important for building Government Chemist capability in food authenticity. As they have done in previous years, our stakeholder government departments are making use of the advisory role of the Government Chemist. We received a request from the FSA and Defra for the Government Chemist to undertake work to investigate differing views on the use of NMR profiling methods to determine exogenous sugars in honey. This work is further explained in the Impact section.

Chondroitin is an over-the-counter food supplement often available in combination with glucosamine. It is sold widely for a number of uses that range from supplements to medication for animals and humans. A Cochrane Review[28] of randomized trials found chondroitin (alone or in combination with glucosamine) better than a placebo in improving pain in participants with osteoarthritis in short-term studies. The benefit was small to moderate and more high-quality studies are needed but the combination of some efficacy and low risk may explain its popularity.

The Government Chemist has had a long interest in the analysis of supplements containing chondroitin stemming from work carried out by a Public Analyst in 2005 that suggested some samples of supplements were deficient of the declared amounts of chondroitin. However as a natural polymer isolated from parts of either land animals, birds or fish, routine analytical methods for chondroitin tend to be relatively nonspecific and an orthogonal approach is required to achieve the goal of affirming identity (including source) and purity.[29]

The Government Chemist team therefore collated all the work we have done on chondroitin and called in Professor Duncan Thorburn Burns of the Institute for Global Food Security, Queen's University Belfast, to review the matter. This resulted in a paper in the prestigious Journal of AOAC International making key recommendations for forensically robust analysis for chondroitin.[30]

[28] Singh J.A., Noorbaloochi S., MacDonald R., Maxwell L.J., Chondroitin for osteoarthritis, Cochrane Database of Systematic Reviews 2015, 1, Art. No.: CD005614. DOI: 10.1002/14651858.CD005614.pub2

[29] Hildreth, J. and Betz, J.M., Role of accurate methodology in demonstrating the safety and efficacy of Chondroitin Sulfate, J AOAC Int., 99(2), 332-332

[30] Burns, D.T., Walker, M.J. and Mussell, C., Chondroitin Sulfate: a critical review of generic and specific problems in its characterization and determination – an exemplar of a material with an Unknown or Variable Composition (UVCB), J AOAC International, 2018, 101, 196-202

Liqueurs – analytical tolerances applied to alcohol declarations

An enquiry was received in August 2017 on what tolerances we would apply to alcohol label declarations in a referee case. Alcoholic drinks above a certain strength must bear a declaration of their alcoholic strength. The declaration must be accurate within certain tolerances. Two options are available, either (a) a tolerance of 1.5 % (absolute) which applies to alcoholic drinks containing macerated fruit or parts of plants, or (b) a tolerance of 0.3 % (absolute) which applies to other beverages containing more than 1.2 % by volume of alcohol. The drinks in question are made by steeping macerated fruit in alcohol for several months. The insoluble material is then filtered off so that the drinks do not contain any visible insoluble macerated fruit.

Initially the Public Analyst assumed the product as sold contained fruit solids and applied a tolerance of 1.5 % by volume but, since realising that it was a filtered product, has been applying a 0.3% by volume tolerance. The producer maintained that a 1.5 % by volume tolerance is applicable because the drink is made using macerated fruit and still contains the soluble constituents of macerated fruit when sold. After careful consideration and consultation with stakeholders we published[31] our opinion that for the reasons given, a tolerance of 0.3 % vol. absolute is appropriate for a filtered liqueur.

Conclusions

Interesting and varied referee casework has again characterised the year under review. Demand increased compared to 2016, and referee cases continued to run at levels typical of our long term average. The absence of referrals from inland authorities and on animal feed was a new feature. This may reflect reduced sampling activity rather than the absence of potential for disputes to arise. Most of the problems referred to us were familiar but one new question, originally posed to us in 2016, relating to the status of the powdered leaf of *Mitragyna speciosa* also known as kratom, was concluded in the courts in 2017 and has now been reported.

Our aim remains to safeguard businesses, regulators and the courts from potentially very costly unwitting errors in measurement science, or the interpretation of scientific data.

We aspire to discharge the Government Chemist's duties to the highest possible standards including the use of sophisticated equipment, a high analytical replication rate, contextual and forensic awareness and statistical assessment of our datasets. Of necessity, these measures require considerably more time and resource than routine testing. However, the 2017 cases showed that on occasion, simply by asking the right questions, rapid resolution of apparent disputes is possible.

We disseminate our learning from referee work via speaking engagements, our biennial conference, our website and publications. It is a pleasure to acknowledge the assistance of colleagues in LGC, particularly Ian Axford in the kratom case, and co-authors within LGC and externally. In particular Professor Duncan Thorburn Burns of the Institute for Global Food Security, Queen's University Belfast, continues to give generously of his time and experience in publishing the outcomes of our work in the scientific literature. Publication is a key measure of transparency in the discharge of the Government Chemist's responsibilities and we are grateful to Norman Michie MChemA, editor of the open access Journal of the Association of Public Analysts where much of our output appears.

[31] https://www.gov.uk/government/publications/published-opinion-on-analytical-tolerances-for-alcohol-declarations

3 IMPACT OF OUR WORK

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 activities to identify the areas 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 the resources required to plan and carry out research projects to support those identified areas.

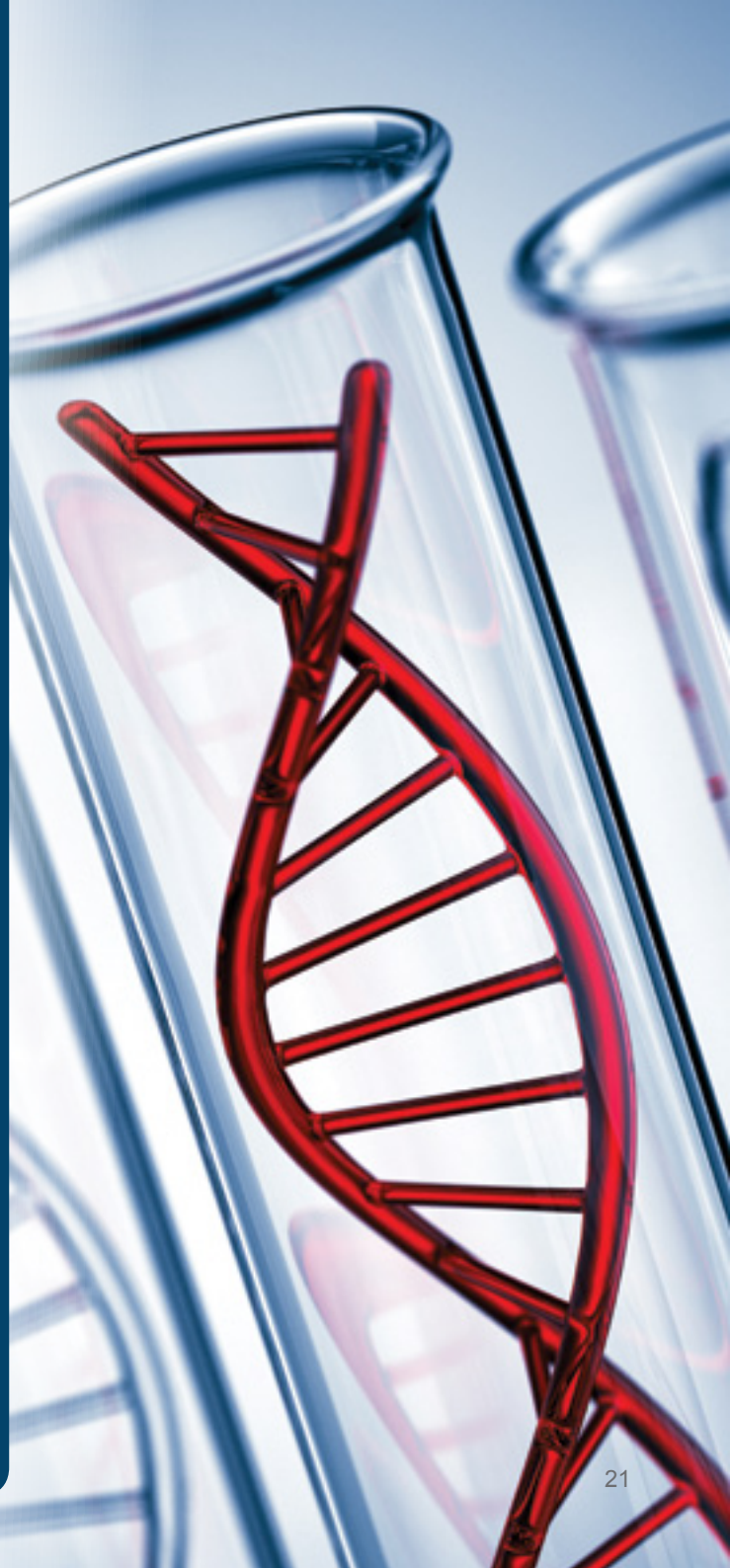
These projects have benefits beyond the referee analyses carried out under the Government Chemist's statutory function. The projects can often impact on the wider measurement community by promoting best measurement practice in the scientific areas where disputes are more likely to arise.

We disseminate our project outputs through knowledge transfer activities and publications (both of which are detailed later in this review). The advisory function of the Government Chemist provides advice on a breadth of analytical measurement subjects within a regulatory and legislative context, to government, the European Commission, and the wider stakeholder community.

All these activities are aimed at translating current capabilities into timely support and advice, and 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.

Building new capabilities

During 2017, and as a consequence of horizon scanning activities, projects to develop further our capabilities to ensure food safety and authenticity were commenced. Our capability building research utilises a broad range of expertise which will 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.



Next Generation Sequencing for food analysis

Bio-surveillance is an obligatory requirement for the monitoring of food for human consumption, which frequently comprises complex mixtures of processed biological material. In many cases, the origin of such products is unclear, and food fraud, associated health risks and violation of ethical/religious principles, are major associated concerns. DNA sequencing techniques allow accurate reading of an organism's genetic code to provide identification of a target species (e.g. fish or meat product) with confidence. Whilst conventional sequencing (Sanger) provides a simple approach for sequencing food samples containing single genetic profiles (e.g. a single target species), it cannot efficiently identify multiple species in the same test. However, Next Generation Sequencing (NGS) facilitates the detection and identification of complex genetic profiles derived from multiple species within a complex and mixed food sample.

NGS has the potential to revolutionise traditional sequencing approaches for species determination (meat, fish, plant, allergens etc.) of ingredients in food. It can be used for metabarcoding approaches (using a multiplex approach to identify multiple animal/plant species simultaneously within the one mixed sample using a panel of DNA markers) as well as examination of the metagenome (the population of bacteria that naturally occur on or within a food sample, e.g. both for food quality/safety and UK Protected Designation of Origin of foods). Where traditional DNA approaches are unsuitable for identification of multiple species in a challenging mixed food sample, NGS may provide a practical solution for target organism identification, even providing enough information to allow identification at the level of breeds for certain species. NGS may also facilitate an effective and cost-efficient route to helping identify products of new synthetic biology.

One of the projects under the current Government Chemist programme seeks to develop and maintain demonstrable competency in the identification of species in food samples using NGS through involvement in laboratory-based work, sharing of best measurement practice with other expert UK stakeholders, and providing a review of the current NGS methodologies available with guidance and clear recommendations on how to implement NGS for food testing in an analytical laboratory.

As part of this project, the Molecular Biology team at LGC participated in an international validation trial of an NGS method for plant and animal species identification, based on metabarcoding, which was part of the European Commission's Seventh Framework Programme (FP7) DECATHLON project. The work resulted in a pioneering paper,[32] one of the first to describe the use of NGS metabarcoding for plant and animal species identification.

Additionally, the Government Chemist is liaising with other stakeholders in the field of NGS to share and harmonise best practice measurement guidance so that results can be interpreted with confidence. Stakeholders include food producers and retailers, as well as those involved in the authentication of herbal health and medicinal products on a national and international scale.

Untargeted and portable technologies

The ability to analyse multiple food samples and species simultaneously using one analytical instrument is driven forwards by cost-efficiency and delivery requirements; hence the need to build capability in rapid, untargeted multi-analyte methods. Furthermore, future technological advances in analytical testing may enable the consumer to be their own analyst, disrupting the current lab-based referee paradigm. There is subsequently a need to build foundations to deal with potential referee work as a result of this possible future shift. Two key exemplar technologies are multispectral imaging (MSI) and ambient ionisation coupled to mass spectrometry (MS).

MSI is a true untargeted, multi-analyte technology, offering the benefits of a non-destructive approach, integrated analysis, and potential for quantitative testing. MSI can augment and streamline pre-existing analytical approaches for Government Chemist referee analysis, providing a more cost effective use of public funds.

Preliminary work using MSI has provided evidence for its potential across a range of food authenticity, adulteration and quality testing situations, including allergen detection, meat speciation, offal in meats and analysis of grains, rice, leaf material, and herbs and spices. The use of MSI as a rapid screening tool is currently being evaluated and compared to established analytical approaches in terms of cost effectiveness, turnaround times and efficacy of results, using data on real-time PCR, microscopy and ELISA from previous referee cases.

The Government Chemist team continues to engage with stakeholders in the field of rapid, untargeted multi-analyte approaches, including UK competent authorities, other governmental funding bodies, food manufacturers and retailers, the herbs and spices trade, UK official control labs, and national experts in plant taxonomy and classification. Through this interaction, the development of best practice guidance for the application of the approach and interpretation of obtained data continues to progress.

MS coupled to ambient ionisation allows the simultaneous analysis of a wide dynamic range of multiple compounds, with limited or no sample preparation and an analysis time of seconds. The development of transportable MS instrumentation allows the data to be collected outside the laboratory environment, and is applicable to multiple areas of concern in the food testing arena. The data can be used for rapid assessment before more costly and involved analysis of the samples is carried out.

Initial work has identified a number of potential applications in the rapid determination of food authenticity and adulteration, including oils, honey and whisky. An initial evaluation of oils shows good potential to discriminate feed stock, and also to identify refined/extra virgin olive oil. Further work on data acquisition and analysis will be developed during 2018 in consultation with stakeholders.

[32] Arulandhu, A.J., et al., Development and validation of a multi-locus DNA metabarcoding method to identify endangered species in complex samples, GigaScience, 2017, 6, 1-18, DOI: 10.1093/gigascience/gix080

A request has been made to the EU funded OLEUM[33] project – which has the overall objective of better guaranteeing olive oil quality and authenticity by empowering detection and fostering prevention of fraud – for samples that could be used to test whether the transportable MS unit being assessed can detect adulteration of olive oil.

NMR for food authentication

One of the projects under the current programme aims to develop the skills and capabilities of the Government Chemist to apply NMR based approaches to support the regulation of food products, and to disseminate the obtained knowledge to UK stakeholders in industry, regulation and enforcement.

Honey has been chosen as the initial focus of attention for NMR methodology due to ongoing concerns regarding the legitimacy of current testing methods which include ^{1}H NMR screening. Honey was ranked 6th in the top 10 of fraudulent food products in 2011,[34] with analytical testing indicating in excess of 20% of European honey samples to be suspicious or fraudulent.

The types of potential fraud employed in honey production cover two main issues – those associated with production (sugar syrup addition, filtration, water content and feeding of bees) and those associated with labelling (geographical, botanical or organic provenance).

Each of these types of fraudulent activity can lead to changes to the profile of honey's components. This may allow the fraud to be detected but only if an extensive and well curated database is held that is able to cope with geographic and seasonal variation. NMR has the benefit of providing a rich source of information on the organic composition of the material studied, providing a more holistic picture of its composition than chromatography or mass spectrometry based approaches. It is also able to provide simple direct quantitation of key components. A substantial international proprietary database of 400MHz NMR spectra has been compiled and is used commercially as the basis for routine analytical services available from a number of commercial testing laboratories. However the integrity of this database and the validity of its use in litigation for food fraud is currently unclear.

Honey primarily consists of a complex mixture of at least 20 different sugars, with many additional, minor components including amino acids and other organic compounds. We have been developing more sophisticated NMR experiments than those used in commercial testing to assess the strengths and weaknesses of the standard testing methodology. These alternative NMR tools will help validate the current methodology, allowing a better understanding of the key measurands and what they represent. Quantitative Heteronuclear Single Quantum Coherence (HSQC) spectroscopy methods, for example, are showing excellent resolution of minor sugars that cannot be resolved under current commercial NMR testing. Through this work we will be able to provide expert opinion into the EU initiative to improve both routine honey screening and the development of more sophisticated testing regimes that can be applied to other food authenticity challenges.

[33] http://www.oleumproject.eu/

[34] Spink, J. and Moyer, D.C., Defining the public health threat of food fraud, J Food Sci., 76(9), R 157-63

Advanced mass spectrometry methods for mycotoxin screening

Mycotoxins are carcinogenic secondary metabolites produced by certain fungi species which appear in food as a result of fungal contamination in the field or during storage. Disputes about their concentrations are a frequent source of referee cases. Current mycotoxin methods used for official control purposes appear not to detect masked mycotoxins (conjugated or non-extractable mycotoxins) which, evidence now suggests, can be converted to the toxic form in the mammalian gut.

It is anticipated that this evidence might prompt the setting of regulatory limits for such 'masked' mycotoxins. In preparation for such eventuality, the Government Chemist team is building on previously developed mass spectrometry capabilities to increase the scope of current methods based on QuEChERS (Quick Easy Cheap Effective Rugged Safe) extraction followed by LC-MS analysis, to include mycotoxins and masked mycotoxins. QuEChERS method is a streamlined approach that was developed to make it easier and less expensive for analytical chemists to examine pesticide residues in food.

During this project, multiple analytical LC-MS platforms will be reviewed to select a best fit approach, which will then be optimised and validated to generate a generic method to complement current bespoke individual mycotoxin methods. Knowledge gathered during the project will be used to provide sound advice to Public Analysts and trade laboratories and better prepare for future referee cases.

In its initial phase, the project team has completed an evaluation of the QuEChERS method for a number of mycotoxins and three distinct matrices. The initial data has been very promising with good recovery of the mycotoxins (non-masked) under study. It has been determined that dependant on target mycotoxins and matrix, minor modifications of the QuEChERS method is required, and that a workflow based on targets/matrix can be developed to aid analysts in the use of the method.

The project has also identified that new software solutions will be required to realise fully the potential of the screening method for masked mycotoxins and consequently, options will be evaluated for discussion and potential acquisition.

Sharing and transferring knowledge

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, FSA, FSS and Defra;
- Proactive input to key stakeholder organisations; and
- Provision of sound advice to stakeholders.

Government Chemist conference

The Government Chemist conference is a biennial event. The last conference took place in 2016 and was reported in the last review. At the time of writing, plans are underway to organise the 2018 Government Chemist conference around the topic of 'Food chain resilience in a changing world'. The conference will offer perspectives from industry, regulators, regulation enforcers, scientists and academics on ensuring the safety and authenticity of food, against an ever evolving regulatory and trading landscape.

Case studies: allergens in spices

It is now well known that the 2015 incident in the UK of cumin alleged to be contaminated with almond, a risk for people with almond allergy, was caused by the Prunus species, *P. mahaleb*. In our investigations of the subsequent referee cases we developed two novel PCR assays, one specific for *P. mahaleb* and a melt curve analysis method capable of identifying common Prunus DNA. Peptides unique to almond and mahaleb were identified permitting identification of the proteins by liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS). We developed criteria for peptide identification to forensic standards. This work enables a staged approach to be taken to any future incident thought to involve Prunus species and provides a template for the investigation of similar incidents. The work was well received. Comments at the post incident review of the recalls applauded the availability of the Government Chemist for interdisciplinary research of this nature. The work led to five peer reviewed papers,[35-39] which were all published in 2017, several international speaking engagements, and the jointly funded knowledge transfer event described in the Areas of collaboration with stakeholders section.

[35] Walker, M., Burns, D., Elliott, C., Gowland H., and Mills, C., Flawed food allergen analysis – health and supply chain risks and a proposed framework to address urgent analytical needs, Analyst, 2016, 141, 24-35

[36] Burns, M., Walker, M., Wilkes, T., Hall, L., Gray, K. and Nixon, G., Development of a Real-Time PCR approach for the specific detection of *Prunus mahaleb*, Food and Nutrition Sciences, 2016, 7, 703-710, http://dx.doi.org/10.4236/fns.2016.78071

[37] Nixon, G., Hall, L., Wilkes, T., Walker, M. and Burns, M., Novel approach to the rapid differentiation of common Prunus allergen species by PCR product melt analysis, Food and Nutrition Sciences, 2016, 7, 920-926, http://dx.doi.org/10.4236/fns.2016.710091

[38] Inman S.E., Groves, K., McCullough, B., Quaglia, M. and Hopley, C., Development of a LC-MS method for the discrimination between trace level Prunus contaminants of spices, Food Chem., 2017, 245, 289-296, DOI: 10.1016/j.foodchem.2017.10.101

[39] Walker, M.J., Burns, M., Quaglia, M., Nixon, G., Hopley, C.J., Gray, K.M., Moore, V., Singh, M. and Cowen, S., Almond or mahaleb? Orthogonal allergen analysis during a live incident investigation by ELISA, molecular biology, and protein mass spectrometry, J AOAC Int., 2018, 101, 1, 162-169, DOI: 10.5740/jaoacint.17-0405, (Epub Dec 2017)

The Government Chemist website

The Government Chemist website is hosted on the GOV.UK platform with the landing page www.gov.uk/governmentchemist. The Government Chemist pages can also be reached from anywhere on the site by entering 'Government Chemist' in the search box. Updates on Government Chemist news can be obtained by subscribing for alerts via the website.

During 2017, 27 articles including news and reports were published on the Government Chemist webpages, which have been viewed in approximately 31000 unique visits. The most frequently accessed documents are the quarterly updates on food and feed legislation, the Government Chemist Review and articles about training events.

Advice

Many stakeholders turn to the Government Chemist for advice on a wide range of topics. We answer on average four requests for advice per month, a level that has remained constant for the past few years. During 2017 we received 40 request for advice. Figure 7 summarises the origin of the enquiries. Figure 8 describes the topics we were asked to comment on. There is a wide spread of enquiries across many topics, with allergens, food safety and authenticity being amongst the most common. The "Other" category included enquiries on trace elements, sampling and sample preparation.

In each case we gave carefully considered advice, supplying a copy of our peer reviewed research findings on the question where applicable and sometimes referring the enquirer to another source of information.

The enquirers were invariably grateful for our time and advice.

Origin of enquiries

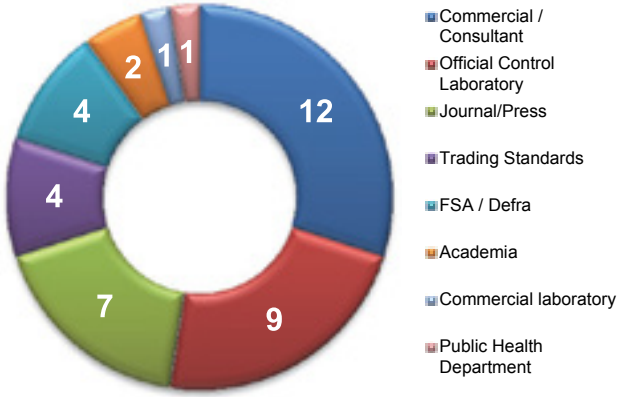


Figure 7 Distribution of enquiries by origin

Enquiries by subject

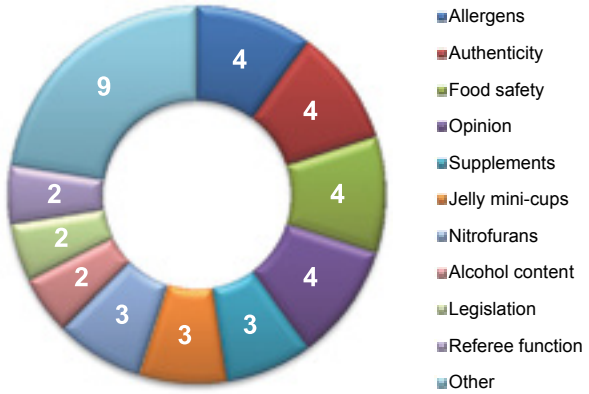


Figure 8 Distribution of enquiries by subject

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.

Analysis and Examination of Food postgraduate course (joint APA and Government Chemist event)

This postgraduate course on the analysis of food had the focus and sense of enjoyment that characterises a good summer school. The sunshine and verdant Reading campus also helped. The commitment of the delegates to long days of intense learning, the dedication and expertise of the lecturers and good organisation made it the success it was.

Thirteen delegates, all experienced scientists in their own right, benefitted from the course. Mainly from UK Public Analyst Laboratories, the intake had an international flavour with two scientists from Gibraltar and one from the Hong Kong Special Administrative Region, China. The course, over a two year cycle, offers a distinctive learning experience, validated by active practitioners in the APA Training Committee, with unique features:

- A vibrant mix of lectures, laboratory practical sessions and interactive exercises;
- Wide range of experts, not available together elsewhere;
- Up-to-date teaching of safety (chemical and microbiological), authenticity, analysis and the law of food, water, feeding-stuffs and fertilisers;
- RSC 'approved training' status;
- Professional networking with peer group and leading experts, National Reference Laboratories, senior academic researchers and policy officials;
- Alignment with the MChemA syllabus;
- Practical and relevant training in microscopy, mycology and microbiology;
- Interactive exercises including 'expert witness' role play mentored by experienced court going scientists;
- Support in the form of delegate packs, pre-course material and in the training section of the APA website[40] regularly updated by the APA Training Committee.

The course was supported by the Governemnt Chemist programme, FSA and RSC Analytical Chemistry Trust Fund and was organised by Michael Walker. Feedback was wholly positive. The following comments capture the tone of the feedback.

"Very good lecturers, entertaining, relevant and informative"

"Always an eye-opener and thoroughly enjoyable"

"Improved my confidence with a microscope"

"Brilliant way of learning, very informative, enjoyable and well planned"

"Interesting in understanding wider implications of our work"

[40] http://www.publicanalyst.com/training/

Publications

Publishing peer reviewed papers is integral to our work enabling transparency to the analytical community. A list of papers published in 2017 is presented below.

Arulandhu, A.J., et al., Development and validation of a multi-locus DNA metabarcoding method to identify endangered species in complex samples, GigaScience, 2017, 6, 1-18, DOI: 10.1093/gigascience/gix080

Burns, D.T., Tweed L. and Walker, M.J., Review of analytical strategies to estimate geographic origin, species authenticity and adulteration by dilution, Food Anal. Method., 2017, 10, 7, 2302-2310, DOI: 10.1007/s12161-016-0756-3

Elahi, S., 'I-KANN-25: A case study from India – using teaching kitchens in urban slums for nutrition education and population health', Complete Nutrition (CN) Magazines, March 2017

Ellison, S.L.R., Ramsey, M.H., Lawrance, P., Stuart, B., Minguez, J. and Walker, M.J., Is measurement uncertainty from sampling related to analyte concentration?, Anal. Methods, 2017, 9, 5989-5996, DOI: 10.1039/c7ay00752c

Groves, K., Cryar, A., Walker, M. and Quaglia, M., Assessment of recovery of milk protein allergens from processed food for mass spectrometry quantification, J AOAC Int., 2018, 101, 1, 152-161, DOI: 10.5740/jaoacint.17-0214, (Epub Dec 2017)

Inman S.E., Groves, K., McCullough, B., Quaglia, M. and Hopley, C., Development of a LC-MS method for the discrimination between trace level Prunus contaminants of spices, Food Chem., 2017, 245, 289-296, DOI: 10.1016/j.foodchem.2017.10.101

Walker, M.J., Tackling food supplement fraud, Technology Networks (Editorial), March 2017

Walker, M.J., Health and nutrition claims – guidance, regulation and self-regulation, Nutrition Bulletin, 2017, 42, 69-79

Walker, M.J., Chapter on 'Food allergy: managing food allergens', Analysis of food toxins and toxicants (Yiu-Chung Wong, Richard J Lewis), 2017, 711-742, ISBN: 978-1-118-99272-2N

Walker, M., Colwell, P., Cowen, S., Ellison, S.L.R., Gray, K., Elahi, S., Farnell, P., Slack, P. and Burns, D.T., Aflatoxins in groundnuts – assessment of the effectiveness of EU sampling and UK enforcement sample preparation procedures, J Assoc. Public Anal., 2017, 45, 1-22

Walker M. and Gray, K., Veterinary residues in food, FS&T, 2017, 31, 23-27

Walker M. and Gray, K., Safer sweets – choking risks from jelly confectionery and technical appeals to the Government Chemist in this area, FS&T, 2017, 31, 40-43

Walker, M.J., Gowland, M.H. and Points, J., Managing food allergens in the UK retail supply chain, J AOAC Int., 2018, 101, 1, 14-55, DOI:10.5740/jaoacint.17-0385, (Epub Dec 2017)

Walker, M.J., Burns, M., Quaglia, M., Nixon, G., Hopley, C.J., Gray, K.M., Moore, V., Singh, M. and Cowen, S., Almond or mahaleb? Orthogonal allergen analysis during a live incident investigation by ELISA, molecular biology, and protein mass spectrometry, J AOAC Int., 2018, 101, 1, 162-169, DOI: 10.5740/jaoacint.17-0405, (Epub Dec 2017)

Wilkes, T., Hall, L. and Burns, M., A brief review of current bioinformatic decision support system (DSS) tools for screening for GMOs in the EU using PCR based approaches, J Assoc. Public Anal., 2017, 45, 023-040

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, academia, 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

Government Chemist staff sit on a number of important committees where they seek to input into and influence the development of new legislation, standards and policy to ensure that they are based on sound measurement science and are fit-for-purpose. These include the IFST Scientific Committee, the Association of Public Analysts Training Committee, the European Network of GMO Laboratories (ENGL) Steering Committee, and the Food Authenticity Network Management Committee.

We have also 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 these committees and are asked to provide an opinion on measurement related issues as they arise. Specifically, the Government Chemist inputted into discussions on the appropriateness of the EU Nanoform definition and highlighted the fact that there are still significant measurement issues with the characterisation of nanomaterials in their pure form, let alone in the environment, as there are very few reference materials or reference methods available. Government Chemist staff sent information on the characterisation of nanomaterials to the committee to illustrate the measurement challenges in this rapidly evolving area. We are 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 also participate in the Nanomaterials Environment and Health Industry Group (NEHIG) which is an industry led group assessing the risk to the environment and human health from the use of nanomaterials. Meetings are convened as deemed necessary and sometimes in conjunction with NEHGG.

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 (LGC) continued as Chairman and Co-ordinator of the Radiochemical Methods Working Group (WG9) of the SCA.

The Government Chemist was represented at a joint workshop, the first of its kind, organised by the Committees on Toxicity (COT), Mutagenicity (COM) and Carcinogenicity (COC) of Chemicals in Food, Consumer Products and the Environment on whether epigenetics should be included in chemical risk assessment. The focus of the meeting was to discuss the feasibility of epigenetic alterations in monitoring exposure to xenobiotics and the possibility of interpreting the changes for incorporation in chemical risk assessment. The meeting concluded that:

- A better definition of the issue is needed;
- Caution is advised with regards to classifying chemicals according to the way they regulate gene expression via epigenetic changes;
- Epigenetics data should be considered on a case-by-case basis, depending on what additional information is available and may provide new/supporting evidence to confirm biological plausibility;
- Given that epigenetic changes are also a basic biological response, it was difficult to see how it would be possible to build epigenetic effects into chemical regulation frameworks;
- Current approaches to chemical risk assessment are effective at protecting human health.

Knowledge to date indicates that there is no chemical that exerts toxicity by a purely epigenetic mechanism and that other markers of toxicity provide appropriately protective risk assessments.

Responding to official consultations

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.

Box 2 Government Chemist public consultation responses

Department	Consultation	Details of consultation
Food Standards Scotland	Shellfish Review: Draft guidance on shellfish toxin controls for the scallop sector	This consultation provided an opportunity to comment on draft guidance which had been developed by FSS in order to provide greater clarity on the food safety controls that are expected to apply to the scallop (pectinidae) sector, with particular reference to shellfish toxins. The consultation covered aspects that could potentially impact sampling and analysis of scallops for shellfish toxins so a response from the Government Chemist was submitted.
Food Standards Agency (NI)	The proposed Materials and Articles in Contact with Food (Amendment) Regulations (NI) 2017	The proposed (Amendment) Regulations will provide for the enforcement, in Northern Ireland, of Commission Regulation (EU) No. 10/2011 as amended by Commission Regulation (EU) No. 2016/1416, by amending the Materials and Articles in Contact with Food Regulations (Northern Ireland) 2012. This consultation sought comments from industry, district councils, consumers and other interested stakeholders on the proposed (Amendment) Regulations. These regulations make specific mention of the use of nanomaterials so a response from the Government Chemist was submitted.
Food Standards Agency (Wales)	The proposed Materials and Articles in Contact with Food (Amendment) (Wales) Regulations 2017	The proposed (Amendment) Regulations will provide for the enforcement, in Wales, of Commission Regulation (EU) No. 10/2011 as amended by Commission Regulation (EU) No. 2016/1416, by amending the Materials and Articles in Contact with Food Regulations (Wales) 2012. This consultation sought comments from industry, district councils, consumers and other interested stakeholders on the proposed (Amendment) Regulations. These regulations make specific mention of the use of nanomaterials so a response from the Government Chemist was submitted.
Food Standards Scotland	Consultation on a food surveillance strategy for Scotland	FSS posed 17 questions requesting input to help them shape a food surveillance strategy for Scotland. The Government Chemist gave a comprehensive response to this consultation giving multiple links to other global organisations and initiatives that FSS could collaborate with or learn from in order to develop its surveillance strategy. On submission of his response, the Government Chemist was thanked by a Senior Scientific Advisor in the Food Protection Science and Surveillance Branch of FSS.
Food Standards Agency (England)	FSA's post implementation review of the Food Safety (Sampling & Qualifications) (England) Regulations 2013 (SI 264)	The post implementation review requested responses to five specific questions: i. Have the Regulations achieved their original objectives? ii. Are the objectives still valid/relevant? iii. Is this Regulation the best option to achieve its objectives, can you think of other ways of achieving this, e.g. non-regulatory, less regulation? iv. Can the regulation be improved? v. Have there been any unintended consequences brought about by the Regulations? The Government Chemist provided a detailed response in line with his opinion that the regulations have achieved their original objectives, that they remain valid and relevant, and that there hadn't been any unintended consequences that the Government Chemist was aware of.
Food Standards Agency (England) **Food Standards Agency (NI)** **Food Standards Scotland** **Food Standards Agency (Wales)**	The Animal feed (Basic Safety Standards) Regulations 2018: • England • NI • Scotland • Wales	The consultations were on draft regulations which are aimed at transposing into national law, the provisions of a revised EU Directive to: • Prohibit the intentional addition of radioactive substances in the production of animal feedingstuffs; • Prohibit the import or export of animal feedingstuffs to which radioactive substances have been intentionally added during production; • Provide the accompanying enforcement powers to deal with non-compliance. Government Chemist staff consulted colleagues in the IAEA (radiation experts) and the APA (animal feed experts) to assess the likelihood of deliberate addition of radioactive substances in animal feedingstuffs. The Government Chemist responded to the consultations agreeing that the likelihood of deliberate addition is negligible compared with the risk from nuclear accidents such as Chernobyl and Fukushima, control of which is available by alternative food and feed law, but highlighted several issues that need to be considered in relation to naturally occurring radioactivity in animals and the use of security devices that use radiation for inspection at ports of entry.

Disseminating the Government Chemist function

Michael Walker provided a major contribution to the MoniQA Association symposium on 'Food fraud prevention and effective food allergen management' in Bari, Italy. He gave the opening lecture on food fraud covering the 2013 horse meat issue and a second lecture on 'What do we need to measure, how should it be reported and how low can we go?'

Selvarani Elahi, gave a lunchtime lecture at the European Commission's Joint Research Centre (EC JRC) titled 'Dispute resolution and fighting food fraud – UK style' in which she gave an overview of the history of LGC, the home of the Government Chemist. She explained the role of the Government Chemist and highlighted three case studies, showing the value the function brings to food testing related disputes. Selvarani also detailed the benefits of joining the Food Authenticity Network.

Malcom Burns helped develop, organise, co-chair and deliver a DNA extraction workshop with the EU-RL-GMFF at the European Commission's Joint Research Centre. This workshop was a three-day event which was attended by over 30 experts representing 19 EU member states and other countries as far afield as Mexico, Ecuador and Brazil. The ENGL Chair and Head of Unit of Food and Feed Compliance, Professor Hendrik Emons, personally thanked Malcolm for the model approach to organising such an interactive workshop, citing all of the positive feedback he had received from participants.

Michael Walker was invited to speak at a two-day intensive workshop on allergen management and advanced testing at the 'Romer Academy' in Austria. Michael delivered two sessions 'Food allergy – challenges and developments' and 'EU-Regulation 1169/2011 on allergen labelling for non-prepacked food' and took part in panel discussions in this industry seminar that attracted delegates from food manufacturers and analytical service laboratories in Austria, Bavaria, Germany, Italy, Latvia and Lithuania.

Selvarani Elahi attended the 3rd Need for Nutrition Education/ Innovation Programme (NNEdPro) International Summit on Medical Nutrition Education and Research – 'Research to Practice'. This conference was targeted at medical and healthcare professionals to address global priorities in nutrition education and implementation programs. Selvarani gave a presentation highlighting the work of LGC and the achievements of the virtual Food Authenticity Network and presented the concept of creating a similar international knowledge network in nutrition in conjunction with the Chair and founder of NNEdPro (Professor Sumantra Ray).

Work carried out by the Government Chemist is frequently disseminated through the Food Authenticity Network website. The network now has nearly 800 members from 40 countries and over 1100 Twitter followers (@FAuthenticity). Discussions are in progress to transform the network from a UK government funded initiative into an industry led global network that can help in the fight against food fraud; leading to an increase in consumer confidence and public trust in the integrity of the food chain.

Addressing measurement issues

Five studies were completed during the 2014-2017 Government Chemist programme which addressed a wide range of measurement issues impacting regulation and enforcement in the chemical and environmental sectors:

- Illegal timber profiling;
- Analysis of nanosilver and ionic silver in environmental waters;
- Water Framework Directive (WFD) measurement issues;
- Brominated flame retardants in water;
- Analysis of diesel fuel, containing biodiesel.

The outputs of these studies have been reported in previous reviews.

To ensure the 2017-2020 Government Chemist programme is addressing measurement issues impacting regulation and enforcement in the chemical and environmental sectors, the Chemical Industries Association[41] (CIA) was consulted to ascertain current areas of concern to their members. The CIA provided feedback on the following areas:

- Characterisation of nano boron nitride;
- Detecting low level chemical or protein impurities (at parts per billion concentrations) in products manufactured using biotechnology;
- Determining carbon particulates and nitrogen oxides (NOx) emissions from diesel fuel incorporating surfactant technology;
- Evaluation of the chemical profile in water, soil and air surrounding manufacturing industrial sites;
- Development of sensitive analytical methods to support the lifecycle assessment, impacts on the aquatic environment following the use of bisphenol A (BPA) and bisphenol A diglycidyl ether (BADGE) in epoxy resins and the subsequent disposal of final cured products (e.g. landfill).

These issues were expanded to give legislative context and presented to the GCPEG. The GCPEG suggested that the National Reference Laboratory for materials and articles in contact with food should be approach to see if methods developed for BPA and BADGE in relation to food can be applied in this context. They also made several other useful suggestions that Government Chemist staff will action.

Discussions will take place on the capability and capacity to deliver some of the projects listed above and any other projects that come to the attention of the Government Chemist. This information will be used to identify one to three mini-projects that can be started in 2018 and completed over the lifetime of the Government Chemist programme.

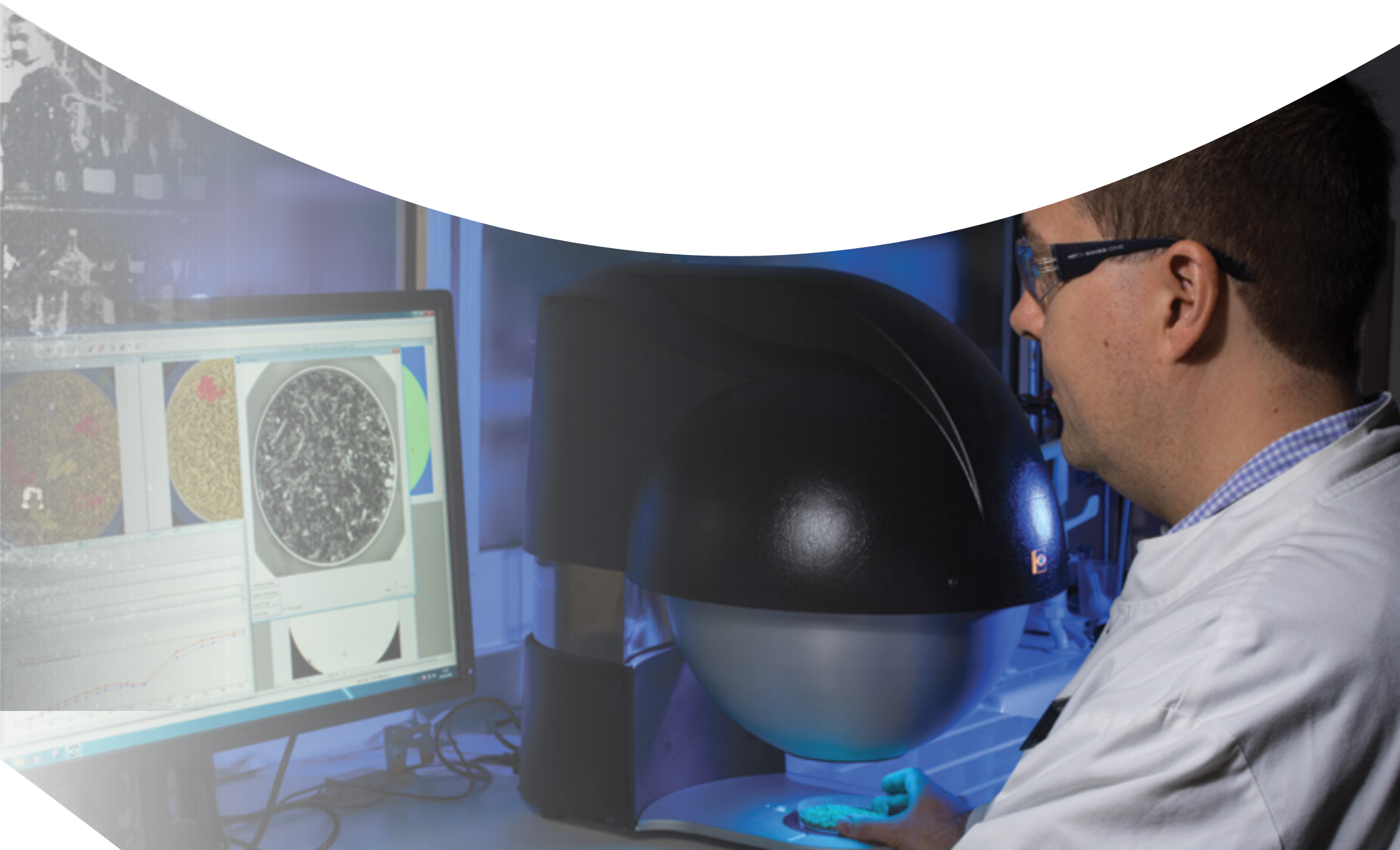
[41] The Chemical Industries Association is the leading organisation representing and advising chemical and pharmaceutical companies located across the UK. Its core membership is a diverse mix of chemical and pharmaceutical companies operating within the UK. CIA covers eight policy areas: climate change, energy, health and safety, employment, trade, environment, chemicals management and economic growth.

GLOSSARY

See the International Vocabulary of Metrology[42] for the current definitions of terms used in measurement science

APA	Association of Public Analysts
BEIS	Department for Business, Energy and Industrial Strategy
CEN	European Committee for Standardisation
CIA	Chemical Industries Association
CCFICS	Codex Committee on Food Import and Export Inspection and Certification Systems
Defra	Department for Environment, Food and Rural Affairs
DIT	Department for International Trade
DNA	Deoxyribonucleic acid
dPCR	Digital PCR
EC JRC	European Commission Joint Research Centre
ELISA	Enzyme-linked immunosorbent assay
ENGL	European Network of GMO Laboratories
EU-RL GMFF	EU Reference Laboratory for GM Food and Feed
FBO	Food or feed business operator
FSA	Food Standards Agency
FSS	Food Standards Scotland
GCPEG	Government Chemist Programme Expert Group
GMO	Genetically Modified Organism
HSAC	Hazardous Substances Advisory Committee. Expert committee providing advice to Government on hazardous substances, toxicology, risk assessments.
HSQC	Heteronuclear single quantum coherence – experiment used frequently in NMR spectroscopy of organic molecules
IAEA	International Atomic Energy Agency
IFST	Institute of Food Science and Technology
LC-MS/MS	Liquid chromatography-tandem mass spectrometry
LLM	Master of Laws
MBA	Master of Business Administration
MChemA	Mastership in Chemical Analysis – this RSC qualification is required for appointment as a Public Analyst or as an Official Food Analyst
MRL	Maximum residue limit
MS	Mass spectrometry
MSI	Multispectral imaging
NEHGG	Defra-led Nanomaterials Environmental and Health Government Group
NEHIG	Nanomaterials Environment and Health Industry Group
NGS	Next generation sequencing
NMR	Nuclear magnetic resonance
NPS	New psychoactive substance
Official Food Analyst	A person qualified under the Food Safety (Sampling and Qualifications) Regulations (1990 and/or 2013) (see also MChemA and Public Analyst)
PCR	Polymerase chain reaction
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 authority, responsible for carrying out checks on food and feed consignments
Public Analyst	Analytical scientist appointed under statute by UK local authorities to provide an official food or feed control function and scientific advice for the enforcement of many acts of Parliament
qPCR	Quantitative (real-time) PCR
RSC	Royal Society of Chemistry
SCA	The Environment Agency's Standing Committee of Analysts
SEO	Second expert opinion in the context of Article 35 of Regulation 2017/625 on official controls
UKCSF	United Kingdom Chemical Stakeholders Forum
WFD	European Union Water Framework Directive
WHO	World Health Organisation

[42] International Bureau of Weights and Measures, International vocabulary of metrology – basic and general concepts and associated terms (VIM), 3rd Edition, JCGM 200:2012, 20012, https://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf



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