Government Chemist

Review 2019

Department for Business, Energy & Industrial Strategy

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Contents

The Government Chemist and his team endeavour to make optimal use of expertise in a timely manner, and to extend that knowledge to stakeholders to strengthen the UK position in providing food and feed that consumers can trust.

Foreword from the Government Chemist

Perhaps it is the speed and intensity with which the year has passed that I find it hard to believe that I am already sitting down to write my introduction for this Government Chemist Review. Having now entered the transition period post-EU Exit, we await the full effects of the outcome of negotiations with our European neighbours and other international trade agreements, and of the review of the UK official control laboratory system.

Whatever the reasons, this year has seen a sustained demand in disputed measurements and associated casework interpretation. It is testimony to the scientists within the team that they have continued to meet the high standard expected and appreciated by our stakeholders.

Beyond the Government Chemist statutory function, this year has centred on the future direction and interactions necessary for this changing world. I have welcomed working closely with our sponsoring government department, our Programme Expert Group, Devolved Administrations, NGOs and industry to produce the Government Chemist Strategy[1] and the 2020-2023 Programme.

This has highlighted the benefits achievable through greater engagement, a process started from the first day of my role. Our Stakeholder Consultation Process brought together a broad cross-section of interested parties to identify the factors most likely to impact the Government Chemist Programme over the years ahead. These ideas framed the capability-building projects subsequently approved for delivery as part of the 2020-2023 Programme.

I was keen therefore that the new strategy should be bold and forward looking.

To protect consumers in tomorrow's world, the Government Chemist will need to help secure national and international compliance by influencing sound policy and regulatory decision-making. Whilst this necessitates continued definitive dispute resolution and support to the implementation of the UK enforcement system, it also requires expanded provision of advice to wider stakeholder communities and the greater influencing of measurement standards development and implementation.

The need to support government and other relevant groups across the UK requires continued world class measurement science and engagement to better realise the benefits from an innovative and growing UK agri-food sector. This requires more formal impact assessment of Governmnet Chemist interventions and provides opportunity for growing the Food Authenticity Network[2] internationally.

Maximising use of the UK measurement infrastructure to address future challenges means ensuring the Government Chemist maintains its position as an impartial and connected operator by future-proofing capabilities and synergies with stakeholders. Investing in the continued development of our core skill base will expand our expert technical provision and enable the more effective transfer of knowledge gained from referee analysis and wider work of the Government Chemist Programme to maximise impact.

Core to my heart is growing collaborative national skills initiatives, thereby extending the value of the Government Chemist function beyond first-line stakeholders to wider sector benefit. Practical support for practising UK Public Analysts and growth of the successful "Joint Knowledge Transfer Framework for Food Standards and Food Safety" will help realise enhanced skills across the sector and minimise the probabilities for disputes.

This ambition presents a challenge to all our current thinking and behaviours, but I am confident that the foundations laid out in terms of the visible outcomes highlighted in this year's Annual Review show us the path to future success is possible.

Dr Julian Braybrook
BSc, PhD, Hon DPhil, FRSC
Government Chemist

[1] https://www.gov.uk/government/publications/government-chemist-strategy-2020-2023
[2] http://www.foodauthenticity.global/

Note from the Chair of the Government Chemist Programme Expert Group

I have been the Chair of the Government Chemist Programme Expert Group for 10 years and I am glad to report that it remains an interesting, stimulating and rewarding role.

During 2019, the Government Chemist has seen a continued demand for the Referee Analyst role. Disputed cases have covered a range of topics and have been of increased complexity – including the completion of three Geneticaly Modified Organisms and three allergen cases. While the reason for the demand for cases of increased complexity is not well known at this point, I can say that it was met with the usual exacting standard of analytical rigour and considered expertise.

The Government Chemist has also been busy fulfilling its advisory role, whether in response to requests for advice, providing opinion in official consultations or participating in a wide range of working groups and committees. During 2019 we entered a new phase in the process of exiting the EU, and with the current pressure on reducing human impact on the environment we can envisage a further increase in the demand for dispute resolution and timely advice and opinion related to regulatory topics.

Following the findings of the Food Standard Agency (FSA) review of the UK's official food and feed laboratories, the Government Chemist team is well placed to contribute constructively to the next phase in the process, both supporting the development of reference methodology for food and feed testing and through the dissemination of knowledge developed through the discharge of the referee and advisory functions.

The publication of a yearly review requires a backwards look at the recent past. However, as you will read in Section 4, a key activity for the Government Chemist is preparing for the future. It was with this intention that a successful Stakeholder Workshop was organised in May at the Royal Society of Chemistry in central London. The event produced a plethora of issues and challenges that were whittled down and then prioritised into key topics to be considered in the next and future programmes. It was a packed and intensive event but also found to be extremely useful by all attendees.

The Government Chemist and his team endeavour to make optimal use of expertise in a timely manner, and to extend that knowledge to stakeholders to strengthen the UK position in providing food and feed that consumers can trust.

This review, once again, offers a glimpse of the excellent scientific work undertaken to address measurement challenges and to support enforcement of UK food law. I hope you enjoy reading it.

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

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What we do

The Government Chemist role was originally 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, fulfilling statutory and advisory functions, which are funded by the Department for Business, Energy and Industrial Strategy (BEIS).

The Government Chemist uses up-to-date and authoritative measurement procedures coupled with experienced interpretative skills to act as a fair and independent arbiter to resolve disputes. In doing so we protect consumers, provide a route of technical appeal for businesses and contribute to regulatory enforcement in sectors where chemical and bio-measurements are important.

Our statutory function

The Government Chemist's statutory function comprises science-based duties prescribed in several acts of Parliament. These duties (see Box 1 on page 7) cover public protection, safety and health, value for money, and consumer choice. Our most important responsibility is to act as a "referee analyst" resolving disputes between regulators and businesses, supported by our own independent measurements, interpretations and expert opinions. Thus, we reduce the burden on public finances as successful resolution often avoids recourse to legal processes. 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 designation of our home laboratory, LGC, as the UK National Measurement Laboratory (NML) and Designated Institute for chemical and bio-measurement.[3]

► **Section 3 looks at the year's completed referee cases.**

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 for policy, standards and regulations. We mainly deliver this function by responding to government calls for advice or published consultations, where there is a significant or important analytical science content. Consultation responses are published on the Government Chemist website; 2019 consultation reponses have been listed on page 22.

► **See Section 4 for more about the wider advisory function.**

Our capability building

Referee analysis is often most challenging 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. The Government Chemist Programme carries out capability-building projects to be prepared for demand for referee analysis in these areas.

► **Section 5 provides an overview of our current capability-building activities.**

Our governance

The Government Chemist Programme is funded by BEIS. Within that department, responsibility for the Government Chemist lies with the International Research and Innovation Directorate.

BEIS has put into place arrangements to ensure that the Government Chemist Programme is delivered competently, and that scientific standards, impartiality, transparency and integrity are maintained. The Government Chemist Programme Expert Group (GCPEG) provides independent scrutiny, overseeing the delivery, planning and quality of the programme and offering advice to BEIS regarding future priorities and strategic direction of the programme.

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

[3] www.lgcgroup.com/uk-national-measurement-laboratory/

GCPEG membership for 2019

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 also a visiting Professor at the University of Reading.

Robbie Beattie

Robbie is the Public Analyst, Agricultural Analyst and Food Examiner to nine local authorities in Scotland. As a senior manager with The City of Edinburgh Council he manages a portfolio of income generating assets.

Thomas Bell

Tom is the Head of Testing Strategy at the Office for Product Safety and Standards, BEIS having previously been the Science Team Manager at the consumer association, Which?.

Simon Branch

Simon is Director of Research, Development and Scientific Affairs at Herbalife and has sat on a number of committees including the RSC Science and Technology Board.

Andrew Damant

Andrew is an official UK delegate on numerous international committees and an advisor to various UK committees. Andrew retired from the FSA, where he led Surveillance, Methods and Laboratory Policy Team, in 2018.

Lucy Foster

Lucy is the Programme Manager for food chain research at the Department for Environment, Food and Rural Affairs (Defra) having previously worked at the FSA.

David Franklin

David leads the Strategic Projects Team in the FSA, which delivers cross-cutting science based projects, including policy for Official Control Laboratories and Sampling Strategy.

Stephen Garrett

Steve is the Food Authenticity Team Leader at Campden BRI, having previously worked at the Institute of Food Research.

Jonathan Griffin

Jonathan is a Public Analyst and Technical Manager for Kent Scientific Services and former President of the Association of Public Analysts.

Kasia Kazimierczak

Kasia leads a multidisciplinary team covering marine science and shellfish hygiene, authenticity, allergens, foodborne viruses and surveillance at Food Standards Scotland (FSS).

Chelvi Leonard

Chelvi is Policy Lead for Accreditation at the Office for Product Safety and Standards, BEIS. Chelvi was the UK representative at CEN and Codex meetings in the standardisation of analytical methods for food.

Brenda McRory

Brenda is a Technical Lead Officer at Suffolk Coastal Port Health Authority, based at the port of Felixstowe. Brenda currently leads on imports of fishery products, and is also involved with the import of foodstuffs of non-animal origin.

Andrew Millman

Andrew is the nominated representative for the British Retail Consortium. Andrew chairs the Authenticity and Chemical Contaminants and Emerging Risks Work Groups and is currently employed by Asda Stores Ltd, working within the Compliance team.

Helen Munday

Helen is the Chief Scientific Officer of the Food and Drink Federation (FDF). She has held this role since 2016 having previously worked for the trade association as Director of Food Safety and Science.

Declan Naughton

Declan is currently Professor of Biomolecular Sciences at Kingston University London with research interests spanning food safety, nutrition, natural products, performance enhancing drugs, inflammation, drug discovery and endocrinology. He is also Interim Associate Dean for Research for the Faculty of Science, Engineering and Computing at the university.

David Pickering

David is the Trading Standards Manager for the Buckinghamshire and Surrey Trading Standards Service. He has been the Chartered Trading Standards Institute Lead Officer for food for many years and represents the profession on numerous groups including the national Food Standards Focus group.

Sophie Rollinson

Sophie is the food science lead in Defra's Food and Farming Directorate and manages the Department's Food Authenticity Research Programme.

Roger Wood OBE

Roger is an experienced food analysis specialist, formerly a senior scientist in FSA. Roger has represented the UK at numerous EU methods of analysis and sampling working groups in the food and feed sectors over many years and has been Chair of a number of international food analysis working groups.

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
- The Food Safety (Sampling and Qualifications) (Wales) Regulations 2013
- Rheoliadau Diogelwch Bwyd (Samplu a Chymwysterau) (Cymru) 2013
- 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

Our 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,[4] have overall responsibility for case supervision. They prepare and sign Government Chemist certificates of analysis;
- Only the Government Chemist or Deputy, once satisfied that the case has been properly completed, may countersign certificates of analysis.

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

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Figure 1 Government Chemist Team and contact points

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Keynote Article: Recycled materials for food packaging

In our invited keynote article, Tony Lord, Principal Chemist at Smithers, gives his views on current issues in food contact materials.

Tony's work is concerned with non-routine chemical analysis and technical and legislative consultancy. He advises on EU and Food and Drug Administration (FDA) legislation relevant to food packaging.

Food comes into contact with many materials and articles, collectively called food contact materials (FCMs), before being eaten. Examples of FCMs include containers for transporting food, food processing machinery, packaging, kitchenware and tableware. These materials should be sufficiently inert so they don't adversely affect consumer health nor influence the quality of the food. To ensure the safety of FCMs, and to facilitate the free movement of goods, a series of legal requirements and controls are in place in the EU.[5]

Regulation (EC) No 1935/2004[6] of the European Parliament and of the Council of 27 October 2004 provides the framework law on materials and articles intended to come into contact with food, implemented in England by the Materials and Articles in Contact with Food (England) Regulations 2012 with equivalents in Scotland, Wales and Northern Ireland.

The national regulations implement the requirements of EU Directives (which are not directly applicable) relating to ceramic articles (84/500/EC) and regenerated cellulose film (2007/42/EC). They additionally maintain the controls on vinyl chloride polymer/co-polymer in Directive 78/142/EEC that are not covered under the Food Contact Plastics Regulation.[7]

The following regulations contain more detailed provisions related to FCMs:

- Regulation 2023/2006 on Good Manufacturing Practice
- Regulation 450/2009 on Active and Intelligent Materials and Articles
- Regulation 10/2011 (The "Food Contact Plastics" Regulation)
- Regulation 1895/2005 on the use of certain epoxy derivatives
- Regulation (EC) No 282/2008 on recycled plastic materials and articles intended to come into contact with foods.

The most recent amended versions of the above measures are available on EUR-Lex, the open access repository of European law.

Action is required to protect the environment and recycling should be encouraged, but there are issues that arise from the use of recycled materials. Let us consider two material types.

[5] European Commission, Food Contact Materials, https://ec.europa.eu/food/sites/food/files/safety/docs/cs_fcm_legis_pm-guidance_brochure_engl.pdf

[6] https://eur-lex.europa.eu/eli/reg/2004/1935/oj

[7] See the FSA website for general comments and links to national legislation across the UK: https://www.food.gov.uk/business-guidance/food-contact-materials

Plastics

Recycled food packaging must comply with Regulation (EC) No 1935 and recycled food contact plastics must comply with Regulation (EC) No 282/2008. This requires recycling processes having a valid application for a European Food Safety Authority (EFSA) opinion. There is currently no EFSA opinion covering microwave or oven use for recycled polymers. Co-extruding recycled polymer with virgin polymer results in migration of contaminants into the virgin layer so that these are not exempt from the requirements of Regulation (EC) No 282/2008.

Typical post-consumer waste chemicals observed in polyethylene terephthalate (PET) are 2-aminobenzamide and its acetaldehyde adducts, and benzene. Measured migration values are likely to exceed levels compliant with Regulation (EC) No 1935/2004 for oven applications or fatty foods.

Polyolefins (polymers produced from a simple olefin and used for packaging such as shink-wrap plastic, for example) are also being recycled; these absorb contaminants which can then migrate into fatty foods at a level approaching 100 %. Typical post-consumer waste related contaminants are fragrance compounds arising from detergents, soaps and shampoo bottles. There are no favourable EFSA opinions covering polyolefins.

It is common to find that some or all of the above considerations are not adequately addressed. Consumption of ready meals and packaged foods could approach 100 % of the daily diet and if migration of contaminants is high, it could present a possible chronic exposure to a wide range of Non Intentionally Added Substances (NIAS).

Testing of NIAS can be complicated by the generation of analytical artefacts resulting in errors in reporting. Examples are ethyl esters (transesterification with ethanol food simulant) and aromatic isocyanates and corresponding amines (thermal degradation of polyurethane adhesive in the inlet of gas chromatography instruments). There are numerous guidance protocols for assessments of NIAS but no standardised analytical methods across Europe.

Paper

Recycled paper may contain mineral oil (petroleum aromatic mixtures) which is of toxicological concern. Quantification methods exist but all are subject to large analytical error due to:

i) A broad chromatographic peak (difficult to integrate from the baseline background)
ii) Variable composition relative to calibration standards
iii) Interferences from waxes and alkanes.

Migration experiments are sometimes conducted, however there is no migration limit supported by toxicological studies or sufficient certainty in analytical results. A better approach is to demonstrate an intervening functional barrier using a mixture of individual mineral oil surrogate compounds (the broad mineral oil peak gives insufficient sensitivity and the possibility of incorrectly concluding an adequate barrier performance). This approach allows the breakthrough time to be calculated from interpolation of the graph obtained from an accelerated kinetic migration test.

Coated paper is increasingly substituted for plastics. There is no harmonised EU legislation for coatings. Substances such as perfluroalkyl compounds (PFOA and PFOS) and bisphenol A are sometimes used and are of concern.

Ultimately, food contact materials manufactured from recycled materials may be used safely but action is desirable on the following:

i) Progression by the European Commission to the next legislative phase by adopting a register of authorised recycling processes for plastics.
ii) Targeted training for local authorities on technical issues around recycling aimed at improving the quality of input waste to recycling processes. Recycling advice to households is often wrong or confusing.
iii) Adoption by the EU of specific regulatory measures for paper and coatings.
iv) Clear guidance on mandatory supply chain documentation with consideration given to a compliance surveillance system involving periodic auditing of supply chain documentation.
v) Adherence to EU standards by the UK after the EU-exit transition phase.

3

Dispute resolution

The Government Chemist underpins industry and public confidence in the food and feed official control system by guaranteeing independent impartial technical appeal to the highest standards. We maintain the credibility of this referee role by stringent governance, painstaking analytical rigour and well informed interpretation of the resulting data.

Analytical results must be interpreted in an increasingly global supply chain and often in increasingly complex scientific, legal and policy contexts. Our default analytical strategy practically amounts to a stand-alone method validation, and provides the necessary high level of analytical confidence. Significant analytical steps are witnessed by a second scientist and data transcriptions verified. The entire dataset is independently evaluated by statisticians for bias and outlying results and to yield a case specific measurement uncertainty if required. A certificate is drafted and reviewed by a qualified person and finally the case file is brought to the Government Chemist for peer review. If all steps are satisfactory the Government Chemist will allow the findings to be released.

The analysis of retained portions of samples referred to the Government Chemist (referee analysis) is more complex and resource intensive than the work of an official control or trade laboratory. This is necessary because:

- our results and opinion must be definitive and bear detailed scrutiny, sometimes at national and international level,
- referrals may be on matters close to a legislative limit hence analytical confidence in our data must be of the highest standard, and
- the problems we seek to resolve may occur where the science, the law or both are uncertain or controversial.

Overview of referee cases in 2019

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.

The statutory conditions for referral usually begin with the contemplation or commencement of legal proceedings where the prosecution intends to offer analytical evidence. During 2019, 14 cases were referred (Table 1), an increase from the seven cases in 2018.

Some of the disputes referred to us in 2019 were familiar to us – mycotoxin contaminants, pesticides residues, food contact materials and choking hazards. However, there was an increase in the number of alleged food allergen and genetically modified organisms referrals, and one case on alleged added sugars in honey led us to consider emerging research findings on stable carbon isotope ratio mass spectrometry.

Table 1 Overview of referee cases in 2019

Inland Authority	Port Health Authority	Central Competent Authority	Dispute	Other*
4 (29 %)	10 (71 %)	0	11 (79 %)	3 (21 %)

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

Allergens

Food allergy is a serious and growing problem, with an estimated two million affected people in the UK. In the absence of an accepted cure, affected individuals must avoid allergenic foods and may need to carry rescue medication throughout their life.

Labelling law imposes the obligation to disclose and emphasise the intentional presence of any of 14 priority food allergen groups (listed in Box 2). However, allergen avoidance can be difficult to achieve in practice, due to unintentional mislabelling or contamination of food with allergens. This results in a significant number of food recalls, but more importantly can cause adverse reactions, that in rare instances may progress to anaphylaxis and death.

Analysis of food and food ingredients for allergens is vital to protect the supply chain, support businesses and secure safe food for people with allergies. Allergen analysis can be challenging[8,9] although until relatively recently allergen referee cases (other than for sulphites) were rare. However, in 2019 we received three disputes about the results of allergen analysis. Two cases hinged on whether or not milk was present in the food and one involved crustacean proteins.

In the first case, an importer was on the verge of prosecution due to undeclared presence of milk in a batch of sweets. The importer, however, claimed that they had tested another bag from the same batch of sweets and no milk was found. The Government Chemist deployed three independent analytical approaches, (i) conventional Enzyme Linked Immunosorbent Assay (ELISA) in relative calibration mode, (ii) ELISA in standard additions mode and (iii) liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS).

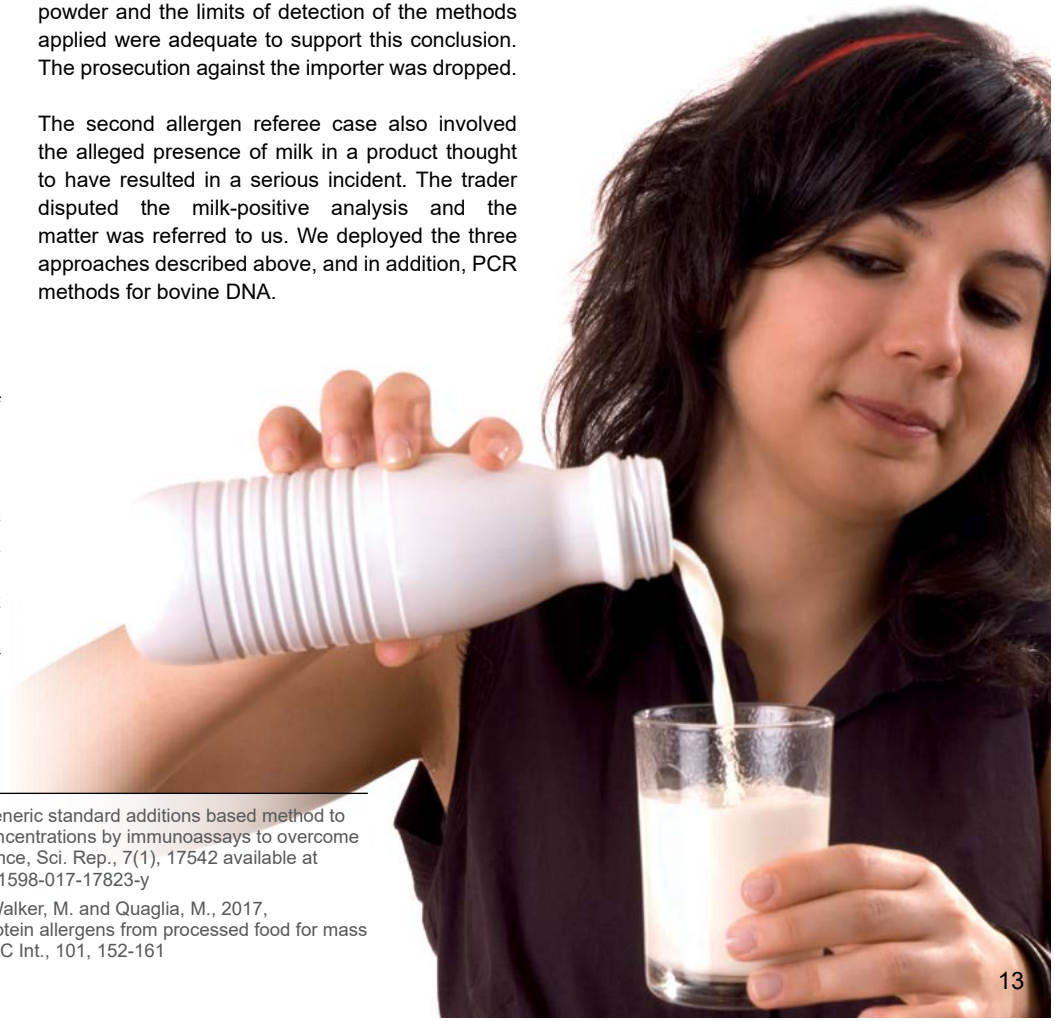
ELISA detects allergen proteins using antibodies raised against the food of interest. Conventionally, ELISA is applied using commercial kits that include calibration solutions. Scientists from the NML recently extended this application by using a novel standard additions approach. Whilst well known in chemical analysis,[10] standard additions had never previously been applied to immunological analysis. The inclusion of a series of standard additions, followed by log transformation and mathematical modelling of the resulting analytical signals, enables an estimation of the endogenous protein concentration.[11] Allergen proteins are too large to be analysed by conventional LC-MS/MS but when digested with an enzyme the resulting peptides can be detected and possibly quantified.

For the referee analysis of the sweets, three separate commercial ELISA platforms for the analysis of the main milk protein, casein, were applied to multiple replicate aliquots of multiple suspensions of individual sweets from the sample.

The best performing ELISA platform in terms of recovery of casein was also applied using the standard additions method. We also used an LC-MS/MS selective reaction monitoring method developed for the detection of peptides specific for the six most abundant proteins in cow's milk (αS1 casein, αS2 casein, β casein, k casein, β-lactogobulin and α-lactalbumin).[12] Reagent blanks, appropriate calibration solutions and sample aliquots spiked with skimmed milk powder were also included in the analytical run. Valid LC-MS/MS detection was assured by appropriate chromatographic retention times and the presence of peaks in at least two mass-to-mass transitions of peptide fragments. Additional certainty of detection was ensured by comparing the intensity ratios of the two most intense transitions for each sample with those observed for the spiked aliquots.

None of the sample analyses returned positive signals for milk protein and it was concluded that in the referee sample received, there was no evidence of casein or cow's milk proteins with a limit of detection between 0.1 and 0.2 mg kg^{-1} expressed as casein. The recovery of added skimmed milk powder and the limits of detection of the methods applied were adequate to support this conclusion. The prosecution against the importer was dropped.

The second allergen referee case also involved the alleged presence of milk in a product thought to have resulted in a serious incident. The trader disputed the milk-positive analysis and the matter was referred to us. We deployed the three approaches described above, and in addition, PCR methods for bovine DNA.



[8] Walker, M.J., Burns, D.T., Elliott, C.T., Gowland, M.H. and Mills, E.C., 2016, Is food allergen analysis flawed? Health and supply chain risks and a proposed framework to address urgent analytical needs, Analyst, 141(1), 24-35

[9] Walker, M.J., 2019, Food allergens: An update on analytical methods. In: Melton, L., Shahidi, F., Varelis, P. (Eds.), Encyclopedia of Food Chemistry, vol. 1, pp. 622–639, Elsevier

[10] Burns, D.T. and Walker, M.J., 2019, Origins of the method of standard additions and of the use of an internal standard in quantitative instrumental chemical analyses, Anal. Bioanal. Chem., 411(13), 2749-2753

[11] Pang S. and Cowen S., 2017, A generic standard additions based method to determine endogenous analyte concentrations by immunoassays to overcome complex biological matrix interference, Sci. Rep., 7(1), 17542 available at https://www.nature.com/articles/s41598-017-17823-y

[12] Based on: Groves, K., Cryar, A., Walker, M. and Quaglia, M., 2017, Assessment of recovery of milk protein allergens from processed food for mass spectrometry quantification, J AOAC Int., 101, 152-161

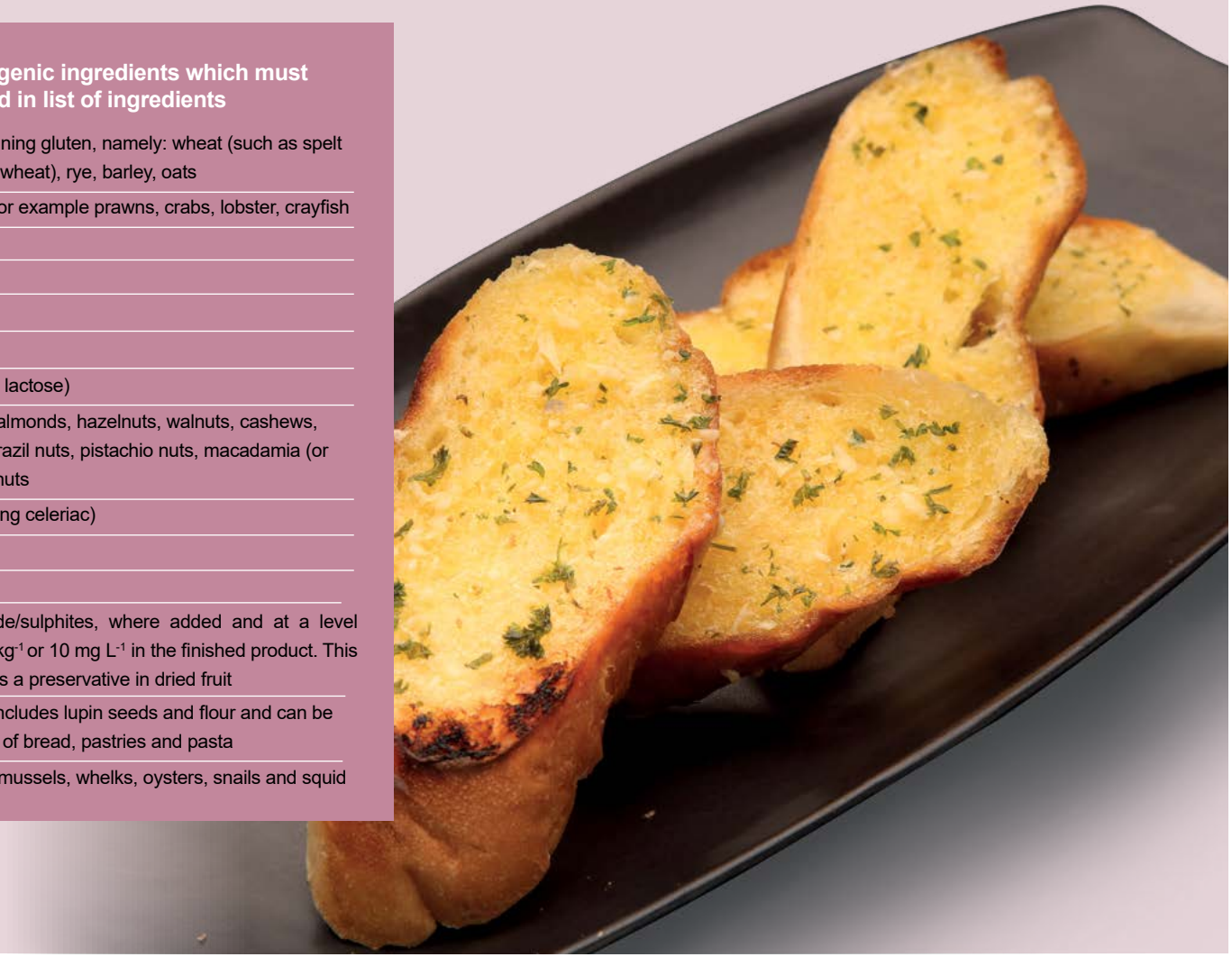
The ELISA applied by the Public Analyst was AOAC (Association of Official Analytical Chemists) approved and ISO/IEC 17025 accredited. Our initial results using this ELISA platform appeared to confirm the Public Analyst findings. Yet, as the Government Chemist investigation proceeded, it became apparent that the sample matrix was problematic for that ELISA platform, probably due to an elevated baseline caused by a matrix/antibody combination. The application of a second ELISA platform combined with the standard additions approach did not find milk proteins in the sample. The LC-MS/MS method detected two of the six major milk proteins but at very low concentrations. We reported that on a weight of evidence basis the sample may contain milk protein, although if it did, the concentrations involved were very low.

The third allergen referee case of 2019 was a sample of garlic bread in which the Public Analyst had, by a PCR technique, detected crustacean DNA. From this finding it was inferred that crustacean protein was probably present, but not declared in the product labelling. This was a risk for people with allergies to crustaceans such as shrimp, prawns, crabs, lobsters or crayfish. We deployed two independent analytical approaches, ELISA and PCR amplification of DNA by separate teams of scientists. A portion of the sample was analysed with a commercial ELISA platform for the analysis of crustacean tropomyosin. Multiple replicate aliquots of the sample were extracted and analysed alongside reagent blanks, appropriate calibration solutions and a sample aliquot spiked with a prawn extract (1 mg kg^{-1} prawn). A portion of the sample was dried and milled for the PCR analysis. DNA was extracted using published protocols, quantified and each extract assayed in triplicate by the application of one commercial real-time PCR platform. Known standard controls of specific copy number of target DNA were processed to carry out a standard curve alongside extraction negative controls, a positive control, raw prawn positive control, prawn run off positive control and no-template control blanks.

All positive controls yielded strong positive responses in both approaches. However the sample did not exhibit detectable amounts of tropomyosin, the major allergen protein in crustaceans, or crustacean DNA. Our findings were reported, resulting in the abandonment of any contemplated proceedings against the trader.

Box 2 Allergenic ingredients which must be indicated in list of ingredients

- Cereals containing gluten, namely: wheat (such as spelt and khorasan wheat), rye, barley, oats
- Crustaceans for example prawns, crabs, lobster, crayfish
- Eggs
- Fish
- Peanuts
- Soybeans
- Milk (including lactose)
- Nuts; namely almonds, hazelnuts, walnuts, cashews, pecan nuts, Brazil nuts, pistachio nuts, macadamia (or Queensland) nuts
- Celery (including celeriac)
- Mustard
- Sesame
- Sulphur dioxide/sulphites, where added and at a level above 10 mg kg^{-1} or 10 mg L^{-1} in the finished product. This can be used as a preservative in dried fruit
- Lupin, which includes lupin seeds and flour and can be found in types of bread, pastries and pasta
- Molluscs like, mussels, whelks, oysters, snails and squid



Choking hazards – Jelly confectionery

There have been instances worldwide of children and elderly people choking to death on certain soft slippery dome shaped jellies. These items, known as jelly mini-cups, are designed to be placed in the mouth in one bite. Food additive law[13] bans the use of a range of gel-forming additives 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. Our paper of 2012[14] remains the only published advice on how to test a product against the definition. We held a seminar to explain to enforcement officers and importers the reasons behind the law on jelly mini-cups and demonstrate how we test them. See Section 5 for more information.

After this successful seminar, we have received only one further case in which we upheld the Public Analyst's findings that the products conformed to the definition and the consignment was prohibited from entering the UK.

Food contact materials

Food contact materials (FCMs) include containers, packaging, cutlery, dishes and kitchenware that come into contact with food or water. To protect consumers, FCMs must not transfer unwanted chemicals into food in unacceptable quantities nor change the taste and smell of the food or drink. Safety limits are set in law on the transfer (migration) of specific compounds and there are also generic limits for non-specific, or overall, migration.

A referee analysis was requested on a sample of nylon ladles from which it was alleged excess primary aromatic amines (PAAs) could migrate. PAAs include compounds associated with a risk of bladder cancer in humans.[15] Only the migration of aniline and 4,4-methylenedianiline (4,4-MDA) were in dispute. We received six nylon ladles, of which five were tested by exposing them to food simulant over two hours at 100 ºC. The food simulant was then analysed by LC-MS/MS which demonstrated that for four of the ladles, migration of PAAs was below the regulatory detection limit. However one ladle exhibited migration of PAA, expressed as the sum of aniline and 4,4-MDA, in excess of the prescribed limit. This finding showed that the ladles were not made to good manufacturing practice and on both these grounds the consignment from which they originated was refused entry to the UK.

The Keynote article in Section 2 provides further context to the complexity of issues related to food contact materials and their possible impact on human health.

[13] Regulation (EC) No 1333/2008 of the European Parliament and of the Council of 16 December 2008 on food additives implemented in England by the Food Additives, Flavourings, Enzymes and Extraction Solvents (England) Regulations 2013 with devolved equivalents

[14] Walker, M. J., Colwell, P., Craston, D., Axford, I. P., and Crane, J., 2012, Analytical strategy for the evaluation of a specific food choking risk, a case study on jelly mini-cups, Food Anal. Methods, 5, 54-61

[15] Shahrestani, M., Tehrani, M.S., Shoeibi, S., Aberoomand Azar, P. and Waqif Husain, S., 2018, Comparison between different extraction methods for determination of primary aromatic amines in food simulant, J Anal. Methods Chem, 2018:1651629. doi: 10.1155/2018/1651629

GMOs

EU law[16] 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.

We dealt with three referee cases involving rice products from China during 2019 (see Table 2 for a summary of the cases). There are no genetically modified rice products authorised in the European Union.[17] From 2006 onwards some rice products originating in, or consigned from China, were found to be contaminated with the genetically modified rice Bt 63. 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 genes encoding for the *Bacillus thuringiensis endotoxin* CrylAb/Ac, genetically engineered as an insect resistance trait sequence.

The analytical approach we used for the quantification of GMOs is described in detail in the Government Chemist Review 2017 (p18).

The Government Chemist benefits from the synergy between our molecular biology team, and the UK National Reference Laboratory for GMOs, both hosted by LGC. 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.

Table 2 GMO cases in 2019

Case	1720-25	1720-27	1720-29	
Sample (if > 1)			A	B
Food type	Rice noodles	Rice balls	Rice noodles	
Disputed GM element	Cry1Ab/Ac	Cry1Ab/Ac	P35S, TNOS and Cry1Ab/Ac	
Government Chemist findings	Cry1Ab/Ac positive	Cry1Ab/Ac positive	P35S negative, TNOS negative Cry1Ab/Ac negative	P35S negative, TNOS negative Cry1Ab/Ac negative
Outcome	Consignment refused entry to UK	Consignment refused entry to UK	Consignment allowed entry to UK	

[16] 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/eli/reg/2003/1829/2008-04-10

[17] Commission Decision 2011/884/EU Recital 8

Mānuka honey – added sugar?

An unusual case was referred to us in 2019 on Mānuka honey, due to opposing laboratories obtaining virtually the same analytical data but differing in their interpretation of it. Two consignments labelled as monofloral Mānuka honey from New Zealand were detained at a UK port because official analysis appeared to show the presence of added sugars not normally found in honey.

The importer sought to cite a natural phenomenon of Mānuka honey as a reason for anomalies in the added sugar calculation. The Government Chemist was asked to give a reasoned opinion on the matter. Both the Public Analyst and the importer forwarded a large volume of high quality data derived from several advanced techniques. By generally accepted scientific standards some of these data showed the apparent presence in the samples of added sugars from sources not permitted to contribute to the carbohydrates of genuine honey.

The Public Analyst relied upon multiple analytical approaches, including two different isotope ratio mass spectrometry (IRMS) techniques that mutually corroborated the findings. Another approach, liquid chromatography-high resolution mass spectrometry (LC-HRMS) was reported to have detected addition of sugar syrup to the honey.

The importer also presented a large volume of high quality data derived from IRMS but lacked the advanced nature of other techniques applied by the Public Analyst, namely quantitative and non-targeted profiling by NMR, the separation capabilities of LC-IRMS and the resolving power of high resolution mass spectrometry.

However the importer drew attention to scientific studies in the peer reviewed literature that show a significant number of, on the face of it genuine, New Zealand Mānuka honeys that show isotope ratio patterns that might account for apparent added sugars of the kind found by the Public Analyst.

Essentially these studies posit a wholly innocent change in the extracted protein carbon isotope ratios that generates apparent added sugars in certain calculations. The phenomenon that could generate apparent added sugars is associated with the natural formation in Mānuka honey of a compound, methylglyoxal (MGO) from naturally occurring dihydroxyacetone (DHA), both characteristic of but not unique to Mānuka honey. Over the course of time and particularly with heat, DHA naturally produces MGO in Mānuka honey. Side reactions of DHA and MGO with protein components of the honey may provide mechanisms to explain the extracted protein carbon isotope differences that lead to calculated apparent C4 sugars. The samples in question contained elevated levels of both DHA and MGO, agreed by both parties. Moreover, other undisputed data such as the leptosperin content and absence of abnormal NMR profiles supported the genuineness of the queried samples. The Public Analyst is commended for presenting some of these data, confirming data supplied by the importer.

While there remained unexplained abnormalities in the carbon isotope data, we concluded that the totality of the evidence was insufficient to regard the queried samples as adulterated by way of added C4 sugars. The consignments were admitted into UK trade. We should note that we continue to look into this matter and nothing stated above should necessarily be taken to apply to any samples or imports other than the specific queried samples referred to the Government Chemist.

Mycotoxins

Mycotoxins are naturally occurring secondary metabolites produced by moulds. Given their toxicity, and the propensity for some forms to cause cancer, stringent controls are in place to reduce human exposure. 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 2019 three cases were referred to us on aflatoxins, two in consignments of peanuts and one in a consignment of chilli powder. For both peanut consignments we upheld the Public Analyst's findings and the cargoes were not permitted to enter the UK. For the chilli powder consignment we confirmed the importer's findings that the sample was well within the limits for aflatoxin B1 and total aflatoxins for chilli.

The Government Chemist Review 2017 (p12) provides details of the analytical method used for mycotoxin analysis.

Pesticides

All foodstuffs intended for human or animal consumption must conform to maximum residue levels (MRLs) for pesticides in order to protect animal and human health. MRLs are in general recommended by EFSA based on a risk assessment and adopted in law. Where a MRL has not been specifically set, a "default" MRL of 0.01 mg kg^{-1} is applied and products must not be placed on the market as food or feed if they contain a pesticide residue exceeding the prescribed MRL or default level.

Two pesticides cases arose in 2019. The first was on a consignment of fresh chillies. Both the Public Analyst and the importer's laboratories checked over 350 pesticides and generally agreed most were not detected and several were present below their MRLS. But they differed on chlorothalonil, a broad spectrum fungicide with a MRL of 0.01 mg kg^{-1} at the time of the case,[18] the former finding it present above the MRL but the latter finding it not detected.

We investigated this case using two methods: gas chromatography coupled with tandem mass spectrometry (GC-MS/MS) and liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS). GC-MS/MS was shown to be more selective and sensitive for chlorothalonil. Accordingly, three replicates of the laboratory sample were extracted on each of three days, cleaned up and analysed with the inclusion of a closely related isotopically labelled compound, dimethyl d6 tetrachloroterephthalate, as an internal standard added after the clean-up stage. Seven mass spectrometric transitions between precursor and product ions were available for chlorothalonil and three for the internal standard. The analytical batch dataset was examined according to prescribed criteria for identification including retention time window, signal to noise ratio and transition ratio tolerances. The mean concentration found was close to the MRL and the lower bound of the 95 % confidence interval truncated at zero. Hence it was not possible to confirm that the concentration of the residue exceeded the MRL beyond reasonable doubt. The consignment was therefore permitted to circulate in the UK.

The second pesticides case hung on the use of the word "organic". A consignment of "organic" sunflower kernels was tested at import resulting in a dispute between the Public Analyst and the importer's laboratory on whether or not the pesticides lenacil and trifluralin were present. Our GC-MS/MS findings were that lenacil was not present above its limit of detection but trifluralin was present at a mean concentration of 7.7 mg kg^{-1}. This concentration is below the MRL for trifluralin in sunflower seeds. However, regulations governing organic production and labelling of organic products prohibit the use of trifluralin, hence the description "organic" was not permitted to be applied.

[18] The EU approval of chlorothalonil expired on 31 October 2019 and an application to renew was rejected owing to safety concerns raised by the European Food Safety Authority (EFSA), see Commission Implementing Regulation (EU) 2019/677 of 29 April 2019 and EFSA, Auteri et al., 2018, Conclusion on the peer review of the peer review of the pesticide risk assessment of the active substance chlorothalonil, EFSA Journal 2018;16, 5126, https://efsa.onlinelibrary.wiley.com/doi/full/10.2903/j.efsa.2018.5126

Conclusions

Interesting and varied referee casework has again characterised the year under review, perhaps most significantly representing a distinct shift in the usual patterns of dispute samples seen over previous years. Of particular note in 2019 were recurring requests for advice on and referred samples of rice from China for GMO testing. As a result of this, we maintain the foremost and uniquely positioned laboratory situated within the UK with experience in the official methods for screening for GMO markers in rice.

Furthermore, against the run of usual outcomes which have tended to confirm the findings of Public Analysts, we overturned three cases in which food allergens had been alleged to have been found. This re-emphasises the known difficulties in food allergen analysis and we remain closely involved in several strands of work seeking to address this.

It demonstrates that we again have discharged the Government Chemist's duties to the highest possible standards through the use of the most sophisticated equipment where required, suitably high analytical replication rates, contextual and forensic awareness, and statistical assessment of our datasets. Of necessity, these measures require considerably more time and resource than routine testing.

We have continued to disseminate our learning from referee work via speaking engagements, our biennial conference, our website, and publications. Publication is a key measure of transparency in the discharge of the Government Chemist's responsibilities and it is a pleasure to acknowledge co-authors within LGC and externally. In particular Professor Duncan Thorburn Burns of the Institute of Global Food Security, Queen's University Belfast, continues to give generously of his time and experience in helping us publish the outcomes of our work in the scientific literature. We are grateful to Norman Michie MChemA, editor of the open access Journal of the Association of Public Analysts (JAPA Online[19]) where much of our output appears. This niche journal functions entirely on voluntary effort and has high salience among Public Analysts, hence an ideal vehicle to disseminate our research and referee findings.

[19] http://www.apajournal.org.uk./index.html

4

The advisory function

The Government Chemist provides specific advice related to measurement topics on a broad range of policy and regulatory developments to local, central and devolved administration governments, the European Union and the wider community of stakeholders. Scientific and measurement-based support is also provided to those industries where chemical and bio-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.

Enquiries from stakeholders

Many stakeholders regularly turn to the Government Chemist for advice on a wide range of topics. Often the enquiries are related to measurement techniques and result interpretation. Sometimes our expert opinion is sought on topical issues such as plastic particles in bottled water, allergen labelling or food contaminants. We answered over 71 requests for advice during 2019.

Figure 2 shows the origin of the source of the enquiries. Figure 3 shows the breadth of enquiries across many topics – allergens, authenticity and measurement issues being amongst the most common, the "other" category including enquiries on trace elements, sampling and sample preparation. In each case, we gave carefully considered advice, supplying a copy of peer reviewed research findings on the question, where applicable, or referring the enquirer to another source of information.

The enquirers are invariably grateful for our time and advice.

Source of enquiries

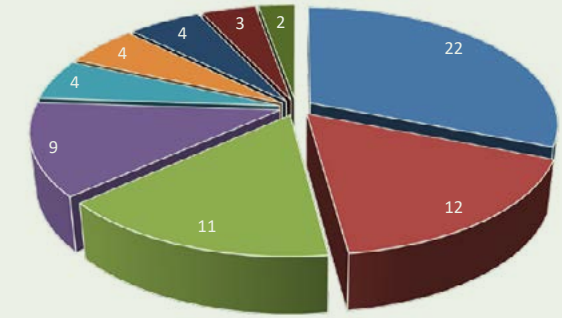


Figure 2 Distribution of enquiries by source

Enquiry topics

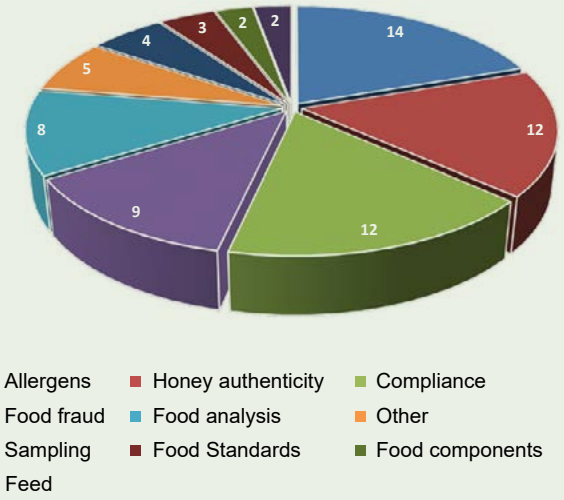


Figure 3 Distribution of enquiries by topic

Expert opinion to stakeholders

Government Chemist staff provide their expert input into a number of Committees (see Box 3) to influence the development of new legislation, standards and policy and ensure that they are based on sound measurement science and are fit-for-purpose.

Response to consultations

Consultations are carried out by the government (including the 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 policy, guidance or legislation, prior to enactment. They are considered by legislators to be an important part of the development process for new legislation.

The Government Chemist has continued to provide input to these official consultations, being well-placed through the additional expertise within the NML and wider LGC organisation across a range of analytical science, to respond authoritatively and independently where the consultations have chemical or bioanalytical measurement implications.

The full list of the consultations responded to by us during the year is shown in Box 4, a selection of responses being available at www.gov.uk/governmentchemist.

Box 3 List of committees to which the Government Chemist contributes

- Authenticity Methods Working Group (AMWG)
- Authenticity Steering Group (ASG)
- British Standards Institution Committee AW/275 – Food analysis – Horizontal methods
- British Standards Institution Committee AW/10 – Animal feeding stuffs
- European Committee for Standardisation (CEN) Food Authenticity Technical Committee
- CEN workshop 86 – Authenticity in the feed and food chain
- Codex Committee on Methods of Sampling and Analysis (CCMAS)
- European Network of GMO Laboratories (ENGL): Steering Committee & Plenary meetings
- Food Law Group
- Food Standards and Labelling Focus Group
- FSA(England)/DH Committee on Toxicity, COT – Food Contact Materials Joint Expert Committee
- Institute of Food Science and Technology (IFST) Science Committee
- Hazardous Substances Advisory Committee (HSAC)
- MChemA Exams Board Meeting
- University of Manchester Allergy Network (MFAN)
- Nanomaterials Environment and Health Industry Group (NEHIG)
- Nanomaterials Environment and Health Government Group (NEHGG)
- Royal Society of Chemistry (RSC) Analytical Methods Committee (AMC) Food and Feed Authenticity Expert Working Group
- Royal Society of Chemistry (RSC) Food Group
- Standing Committee of Analysts (SCA) board
- UK Chemicals Stakeholder Forum (UKCSF)

Box 4 Consultations to which the Government Chemist has responded

- Revised allergen labelling for foods pre-packed for direct sale (PPDS), lanched after the inquest into the death of Natasha Ednan-Laperouse.
- BS EN ISO 21572 Foodstuffs -- Molecular biomarker analysis -- Protein-based methods.
- ISO/TC34 N2078 ISO NWIP 24583 on “Quantitative nuclear magnetic resonance spectroscopy -- Purity determination of organic compounds used for foods and food products -- General requirements” via the BSI standards portal.
- National Food Crime Unit (NFCU): "Food Crime Strategic Assessment (FCSA) Information Requirement 2019".
- FSA consultation on the implementation of the Official Controls Regulation (EU) 2017/625 (OCR), where FSA were seeking stakeholder views in relation to:
  - The proposed implementation of legislation in England to provide for the execution of powers and enforcement of the Official Controls Regulation (EU) 2017/625 (OCR) in relation to the FSA areas of responsibility for food and feed law and animal health and welfare.
  - Their assessment of the impacts associated with the implementation of the legislation in England in relation to FSA areas of responsibility only.
- Defra consultation on the development of a National Food Strategy.
- BS EN ISO 7541 Spices and condiments – Spectrophotometric determination of the extractable colour in paprika.
- BS ISO 939 Spices and condiments – Determination of moisture content.

Horizon scanning

The Government Chemist team carried out a Stakeholder Workshop in May 2019 at the Royal Society of Chemistry, London to identify and prioritise the drivers which should shape the direction of the Government Chemist Programme 2020-2023.

Forty-three stakeholders from across the food and feed sector, including representatives from manufacturers, distributors, retailers, importers, regulators, legal and government, established the key drivers influencing the food and feed sector to which the proposed Government Chemist Programme 2020-2023 should respond.

The workshop comprised two components – an initial brainstorming followed by a prioritisation stage. In excess of 250 ideas were gathered in the initial brainstorming phase. From this wealth of information, participants identified the following as priority areas of concern:

- the impact of alternative packaging materials,
- the application of point-of-use analysis technologies,
- persistent issues with allergen testing,
- increase in food fraud in a global market, and
- lack of trust in emerging technologies as well as the databases they use.

The Government Chemist Programme 2020-2023 was finalised based on these findings and with the input of the Programme Expert Group during a Decision Conference in November 2019.

5

Impact of our work

The impact of the work of the Government Chemist Programme is broad and the effects can be observed in a number of ways.

Horizon scanning activities identify the areas where referee cases are more likely to arise, or where new 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 our research projects to support the areas identified.

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

The breadth of knowledge generated through the Government Chemist's advisory function – and disseminated through to government, European Commission and wider stakeholder communities – provides a secure scientific basis for more efficient and cost-effective regulations.

This is achieved by translating current capabilities into timely support and advice, by generating chemical and bio-measurement solutions for its own referee case use and for adoption by stakeholders, and by predicting future regulatory issues.

Building new capabilities

Developing and maintaining world-class molecular biology approaches for food analysis

The Government Chemist team is well versed in the use of molecular biology techniques for the successful resolution of referee cases. Molecular biology techniques, specifically quantitative real-time PCR, was a key technique used for the determination of *P. mahaleb* in cumin[20] in a previous referee case and the technique has been used extensively in meat speciation investigations. Modern molecular biology approaches (inclusive of real-time PCR, digital PCR and DNA sequencing) play an important role in successful food testing to help consumers make informed choices on the food they eat. The continued development and maintenance of the Government Chemist DNA testing capabilities is a key activity to ensure effective resolution of future cases and to underpin timely advice to industry, regulators and food testing laboratories.

During the 2017-2020 programme the molecular biology team has undertaken comprehensive work to maintain and further develop its leading position in the analysis of Genetically Modified Organisms (GMO). Malcolm Burns has been an active member of four EC European Network of GMO Laboratories (ENGL) Working Groups, providing guidance in the area of digital PCR analysis, DNA sequencing, DNA extraction and method performance requirements. The aim of this latter Working Group is to provide acceptance criteria for digital PCR methods, methods for the detection of products arising from new mutagenesis techniques (synthetic biology) and methods of analysis for GM animals, in the framework of GMO official controls.

Synergistic activities to help complement the National Reference Laboratory for GMOs in food and feed, a position also held at LGC, has seen the Government Chemist team be involved in nine GMO proficiency test rounds throughout the 2017-2020 Government Chemist Programme. One of the latest rounds, led by the EC, involved an international comparative test of a completely blind sample for the detection and quantification of authorised and unauthorised GMOs. The Government Chemist expertise on how to screen and quantify for any GM event in a blind sample, which could be considered representative of a real-life market sample, increased as a result. This may be of particular importance following EU exit, where access to databases and an effective screening approach are prerequisites for effective GM detection. Additionally, as a consequence of a related proficiency test round, LGC further extended its ISO/IEC 17025 flexible scope of accreditation for GMO analysis to include more modern real-time PCR instruments.

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

Work underpinning better DNA extraction approaches has also made a significant impact, as evidenced through workshops and working groups in which Government Chemist staff have been involved during the length of the current programme. Malcolm helped develop and chair a DNA extraction workshop with the EU Reference Laboratory (EURL) for GMOs in feed and food, held at the Joint Research Centre (JRC) in Ispra (Italy) in 2017. The workshop capitalised upon the shared knowledge and collective expertise and experiences of scientists from 19 different countries attending the workshop, and provided a forum for discussing and agreeing best measurement practice guidance for DNA extraction.

In recognition of his extensive expertise, Malcolm has been appointed as a specialist adviser to the FSA in relation to molecular biology analysis and food authenticity testing. He is also an author on the recent EC-JRC "Guidance Document on Measurement Uncertainty for GMO Testing Laboratories", which has been updated in line with current recommendations and use of new technologies, and is due for publication in 2020.

The Government Chemist function continues to make headway on evaluating and optimising digital PCR for food authenticity testing. An e-seminar, "An introduction to dPCR" was released in 2019[21] as a general introduction to the technique and the team collaborated on a published EC-JRC ENGL report[22] which addresses experimental considerations of dPCR for laboratories and makes recommendations for the transfer of existing real-time PCR methods into a dPCR format.

The combined expertise of the Government Chemist and NML scientists was crystallised in the publication of a Royal Society of Chemistry (RSC) book, "DNA Techniques to Verify Food Authenticity: Applications in Food Fraud".[23] This book is the most comprehensive and timely collection of material from those working at the forefront of DNA techniques applied to food authenticity, with additional contributions from other international experts in the field.

[21] http://www.foodauthenticity.uk/training

[22] Pecoraro S., Berben G., Burns M., Corbisier P., De Giacomo M., De Loose M., Dagand E., Dobnik D., Eriksson R., Holst-Jensen A., Kagkli D. M., Kreysa J., Lievens A., Mäde D., Mazzara M., Paternò A., Peterseil V., Savini C., Sovová T., Sowa S., Spilsberg B. Overview and recommendations for the application of digital PCR, 2019, EUR 29673 EN, Publications Office of the European Union, Luxembourg, ISBN 978-92-76-00180-5, doi:10.2760/192883, JRC 115736.

[23] Burns M., Foster L., Walker M. DNA techniques to verify food authenticity: Applications in food fraud, 2019, RSC publishing,

Mass spectrometry approaches for allergenic protein testing

Food allergies are estimated to affect 5 % of adults and up to 8 % of children. Whilst identifying allergens on food labelling is a regulatory requirement in many countries, cross contamination of food with allergens is also a serious problem. The analytical issues associated with food testing (lack of reference methodologies and suitable reference materials) represent obstacles for accurate control of food contamination and appropriate enforcement of legislation. This is resulting in increases in the number of incidents, referee cases and potential product recalls.

During the 2017-2020 programme, NML scientists have been working on a multi-stranded project to further develop existing capabilities in allergen detection using protein analysis by mass spectrometry.

The organic mass spectrometry team have been developing and validating targeted approaches to detect low amounts of allergens in referee case samples. A targeted method to monitor more than 30 peptides from milk allergen proteins, including β lactoglobulin, α-, β-S1 and S1 and k- caseins, was developed with adapted extraction protocols to detect the presence of milk in two referee samples (sweets and popcorn). The detection limit for these adapted protocols were shown to be below mg kg^{-1} as milk.

The team also evaluated the potential of untargeted high accuracy mass spectrometry methods combined with label-free approaches for quantification of allergen proteins in food matrices. These methods offer the advantage of also detecting “unexpected” allergen proteins, providing additional confidence in the identification of detected proteins.

Label-free quantification methods are widely applied in the proteomic area, but there is limited knowledge about their utility for food allergens and food authenticity measurements. The limitations for these methods include the accuracy of untargeted proteomic experiments being reliant on the use of a correct protein database, and the requirements for well-defined workflows to obtain quantitative information.

The aim of the project under the Government Chemist Programme was to evaluate the linearity, accuracy and detection limits of an untargeted method placing particular attention on the workflow used for data analysis and the development of an appropriate database based on experimental information.

A label-free mass spectrometry method was therefore developed, initially using milk and egg powder, and a database was created for the identification and subsequent quantification of milk and egg allergen proteins. The linearity of the method was assessed through spiking experiments and the method was further customised for quantification of food allergens in more complex matrices such as an LGC multi-allergen reference material (RM) at concentrations of 10 mg kg^{-1} and 250 mg kg^{-1} as protein, the latter using materials prepared upstream of the finished RM.

Proteins for all allergens were detected at both 10 and 250 mg kg^{-1} as protein, but the detection limits and accuracy of the method were poor when compared with targeted approaches. While this is not surprising, particularly for allergens present at very low levels in complex matrices, this approach provides complementary information to the targeted methods and within its limitation has proved to be useful to help solve referee cases.

Throughout the work programme, efforts to harmonise methods for the measurement of allergens have been made at the national level, by contribution to the current development of a best practice guide for analysis of samples for allergens in collaboration with the University of Manchester Food Allergy Network (MFAN), and at the international level through active participation in the NIST Food Safety Workshop in October 2019.

The work programme has also produced a significant number of publications related to allergen detection, labelling and management, already listed in previous annual reviews (2017 and 2018).

Sharing and transferring knowledge

The Government Chemist supports innovation and policy making by sharing knowledge gained through our work, particularly in referee analysis, with the analytical and regulatory communities to improve knowledge and skills.

Government Chemist conference

The Government Chemist conference is a biennial event. The last conference took place in 2018 and was reported in the Government Chemist Review of that year. At the time of writing, plans are underway to organise the next Government Chemist conference around the topic of "Safe food for tomorrow's world". The conference will offer perspectives from industry, regulators and academics on the future challenges in the manufacturing, packaging, retailing and testing of food that is safe and consumers can trust.

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 2019, 43 articles including news and reports were published on the Government Chemist webpages. The most frequently accessed documents are the quarterly updates on food and feed legislation, the Government Chemist review and articles about training events.

Training

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

Analysis & Examination of Food – APA Educational Trust annual course, University of Reading, April 2019

On behalf of APA Educational Trust the Government Chemist organises an annual fully residential week-long postgraduate course on chemical and microbiological official control science, the work of the Public Analyst. The course, over a two-year cycle, offers a distinctive learning experience, validated by active practitioners in the APA Training Committee. It offers a vibrant mix of lectures, laboratory practical sessions and interactive exercises, delivered by a wide range of experts.

The topics cover food safety (chemical and microbiological), authenticity, analysis and the law of food, water, feeding-stuffs and fertilisers. The course also provides an opportunity for participants to network with peer group and leading experts, senior academic researchers and policy officials.

The 2019 course ran in April. As a new feature this year, a day was set aside for a series of one hour lectures, designed to attract local authority sampling and enforcement officers. Three Trading Standards Officers and two Environmental Health Officers registered for this. The residential course was attended by 11 delegates, from Public Analyst laboratories in England and Scotland, and from research and academic organisations.

The delegates provided very positive feedback:

"Good introductions to the subject and will put me in a good position to practice and expand my knowledge at work"

"Extremely interesting. Gained a new skill [microscopy] and am able to talk a lot more confidently about some of the techniques used by Public Analysts"

"Perfect insight to industry research"

Workshop on assessment of jelly mini-cups

The Government Chemist organised a workshop (funded by the Joint Knowledge Transfer Framework for Food Standards and Food Safety) covering advice and guidance to interested groups on the assessment and evaluation of jelly mini-cups with the aim of disseminating expertise gathered through our involvement in assisting authorities and businesses to interpret the relevant legislation since 2004.

The workshop – which was oversubscribed – was attended by 22 delegates representing enforcement offices (Trading Standard Officers and Environmental Health Officers), Public Analysts and traders. The workshop included talks by Selvarani Elahi, Michael Walker and Ian Axford (expert in Consumer Safety Regulation) and practical sessions in the laboratory laboratory led by Kirstin Gray and Michael Walker. There was a good deal of exchange of information and opinion and the feedback was very positive.

The event helped clarify the thinking about emerging "borderline compliant" products appearing on the market and enhanced significantly the consistency with which this choking hazard will be regulated in the future.

Food allergy – Human, analytical and regulatory implications

The Government Chemist collaborated with the Institute for Global Food Security (IGFS) in Queen's University Belfast, to run a one day conference, "Food allergy – Human, analytical and regulatory implications".

With a special focus on helping SMEs understand more about food allergy, the conference in Belfast brought together well-known experts in the fields of food allergens, including specialists from the food industry, (both large and small companies, and caterers), researchers, enforcement officers and regulators.

The themes of the conference were risk analysis and risk management of food allergy focusing on the analysis of food allergens, their management in the supply chain and the regulation and enforcement of food law on allergens. Over 100 enforcement officers, MSc students and food industry personnel attended.

Michael Walker spoke on options for analysis of food allergens, how these can be improved, and touched on reporting and interpretational issues in his talk, "Orthogonal Analysis".

Seminar – Honey authenticity: determination of exogenous sugars by nuclear magnetic resonance

The Government Chemist, Defra, FSA and FSS held a UK seminar on honey authenticity in November attended by 57 people representing stakeholder organisations.

The aim of the seminar was to bring together representatives from, mainly UK, stakeholders involved in honey production and analysis to discuss this topic and ideally come to an agreed position. It was anticipated that the output of this seminar would help inform future UK government policy on the use of NMR for honey authenticity.
The seminar consisted of a series of presentations from invited experts that set the scene for the workshop part of the day, which involved participants discussing the suitability of NMR for enforcement purposes and to identify gaps and priorities to assessing the use of NMR for the appraisal of honey authenticity. There was consensus among the workshop groups that currently NMR-based methods are not yet suitable for the determination of exogenous sugars in honey for enforcement purposes.

In order to address this, the participants made a number of suggestions centred on the creation of a forum for continuing dialogue between all parties, provision of training, education and guidance on the production and analysis of honey, and standardisation of the application and interpretation of NMR approaches for the determination of exogenous sugars in honey.

The UK honey seminar provided a valuable forum for stakeholders to come together to discuss the use of NMR for the determination of exogenous sugars in honey and has produced some constructive ideas on how some of the current issues faced in the UK could be addressed.

Publications

Publishing peer reviewed papers, case studies and articles is an important aspect of the work of the Government Chemist. Publications enhance the impact of the programme and enable greater transparency into its activities. A selection of papers published in 2019 include:

Burns M., Walker M., Foster L., DNA techniques to verify food authenticity: Applications in food fraud, 2019, RSC, ISBN 1788011783, https://doi.org/10.1039/9781788016025. This book, commissioned by the Royal Society of Chemistry post the horsemeat incident, provides a fundamental understanding of modern DNA techniques as applied to food authenticity and adulteration testing. The book includes chapters from Malcolm Burns, Michael Walker and Selvarani Elahi, from the Government Chemist team, as well as from Timothy Wilkes, Victoria Moore, Gavin Nixon and Stephen Ellison from the NML at LGC. The editorial team collaborated with national and international experts in commercial and public laboratories, academia and regulators, providing international perspectives on quantitative analysis, the role of metrology and the need for harmonisation and standardisation.

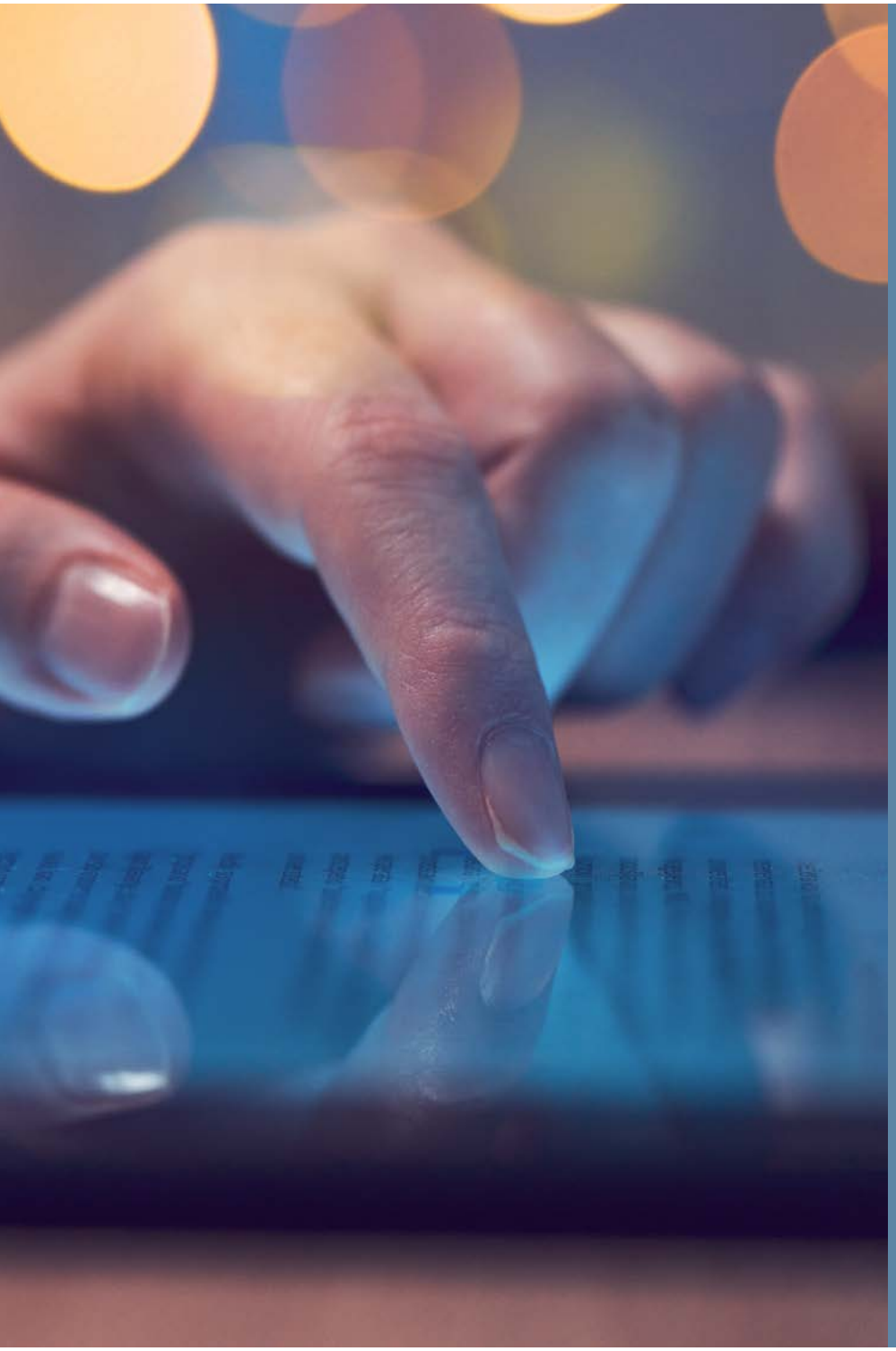
Pecoraro S., Berben G., Burns M., Corbisier P., De Giacomo M., De Loose M., Dagand E., Dobnik D., Eriksson R., Holst-Jensen A., Kagkli D. M., Kreysa J., Lievens A., Mäde D., Mazzara M., Paternò A., Peterseil V., Savini C., Sovová T., Sowa S., Spilsberg B. Overview and recommendations for the application of digital PCR, 2019, EUR 29673 EN, Publications Office of the European Union, Luxembourg, , ISBN 978-92-76- 00180-5, doi:10.2760/192883, JRC 115736

Dunn Galvin A., Roberts G., Schnadt S., Astley S., Austin M., Blom W.M., Baumert J., Chan C.H., Crevel R.W.R., Grimshaw K.E.C., Kruizinga A.G., Regent L., Taylor S., Walker M., Mills E.N.C., Evidence-based approaches to the application of precautionary allergen labelling: Report from two iFAAM workshops, 2019, Clin. Exp. Allergy, 49(9):1191-1200. doi: 10.1111/cea.13464

Case studies

Burns, D.T. and Walker, M.J., Critical review of analytical and bioanalytical verification of the authenticity of coffee, 2019, J AOAC Int., https://academic.oup.com/jaoac/article-abstract/103/2/283/5718357?redirectedFrom=fulltext

Burns, D.T. & Walker, M.J., Origins of the method of standard additions and of the use of an internal standard in quantitative instrumental chemical analyses, 2019, Anal. Bioanal. Chem., https://doi.org/10.1007/s00216-019-01754-w



Engagement with stakeholders

In addition to the regular participation in advisory committees described in the Advisory function section, the Government Chemist is invited to contribute to events organised by stakeholder organisations. Some of the highlights of the year are outlined below.

In February 2019 "Food Unwrapped" a Channel 4 series, featured Michael Walker in a programme on meat species in take-away meals. Michael presented DNA PCR results obtained by Lancashire Scientific Services and discussed their significance with one of the show's main presenters Matt Tebutt.

Michael Walker represented the Government Chemist at conference held to celebrate the 20th anniversary of the establishment of the Food Safety Authority of Ireland, FSAI. FSAI predated FSA by a year and was the first of the new "farm to fork" agencies set up across Europe in the aftermath of BSE. Michael gave a well-received talk on Government Chemist referee processes and casework illustrating "why laboratories get it wrong" and benefitted from networking with colleagues in the Irish, European and UK regulatory and agrifood science sectors.

Julian Braybrook and Michael Walker travelled to Northern Ireland to meet Professor Ian Young, Professor of Medicine, Queen's University Belfast and Chief Scientific Advisor to the Department of Health (NI). Whilst there, they also met Maria Jennings, Director for Regulatory Compliance, People and Northern Ireland and Michael Jackson, leading on the FSA Regulating our Future programme.

Julian Braybrook and Paula Domann visited the Leader of the Welsh Assembly and Chief Science Advisor to raise awareness of the independent statutory and advisory roles of the Government Chemist and to start an on-going dialogue regarding support for common future priorities.

Julian Braybrook presented case studies from the UK Referee Analysis and Advisory functions and Gill Holcombe and Michael Walker exhibited a poster on allergen reference materials (RM), at the NIST Food Safety Workshop held in Gaithersburg, Maryland (USA) in October.

Julian Braybrook and Selvarani Elahi met with the CEO of FSS, Geoff Ogle, in Edinburgh. Julian and Selvarani described the work of the Government Chemist and current collaborative efforts with FSS. Geoff gave an overview of FSS priorities and future joint initiatives were discussed.

Julian Braybrook and Selvarani Elahi met with the new Head of the (NFCU), Darren Davies. The role of the Government Chemist, the NML and the Food Authenticity Network (FAN) were discussed. It was agreed that it would be mutually beneficial for the NFCU and the FAN to work together more closely.

Areas of collaboration with stakeholders

It is clear that collaboration with other organisations with common or complementary interests not only helps us discharge our roles efficiently, but also contributes to a more rapid development and implementation of methods and standards. During 2019 we engaged with a wide range of stakeholders on a variety of topics.

Food allergens

The Government Chemist has a long standing interest in food allergen analysis. Our interest focuses on the three areas of:

1. Metrologically traceable food allergen analysis (the science of ensuring allergen measurements agree from one laboratory to another across the globe).

2. Resolution of analytical disputes about allergen measurement (referee cases).

3. Food allergen management – to better understand industry best practice in order to interpret allergen measurement results in the right context and offer advice to businesses, regulators, enforcement authorities and consumers on request.

Our collaboration with a range of experts on food allergy described in our 2018 report continued. In 2019 we focused on improving food allergen analysis.

We contributed to an iFAAM[24] peer reviewed publication on precautionary allergen labelling.[25]

We hosted and helped to organise a meeting of the University of Manchester Food Allergy Network (MFAN) devoted to best practice in allergen analysis.

Michael Walker and Malvinder Singh represented the UK at a European Network of Food Allergen Detection Laboratories meeting at the European Commission's Joint Research Centre in Belgium. This Network aims to establish best practice in the measurement and reporting of results from food allergen testing in Official Control Laboratories across Europe.

Michael Walker was one of a panel of speakers at a meeting of the Food Standards Agency's Northern Ireland Food Advisory Committee (NIFAC) meeting. Chaired by the NI FSA Board member this was an opportunity for NIFAC to input in advance of decisions on enhanced labelling following the death of Natasha Ednan-Laperouse, who had a fatal allergic reaction to undeclared sesame in a take-away baguette.

Michael Walker also contributed to an Imperial College Medical School Food Allergy MSc course with a lecture entitled "Public health issues and food allergy" to an audience of around 30, mainly paediatricians and GPs.

Gill Holcombe and Michael Walker took part in the MoniQA Task Force on Food Allergen Reference Materials, delivering lectures on improved allergen analysis through the use of appropriate reference materials at the 3rd MoniQA International Symposium, Food Fraud Prevention and Effective Food Allergen Management in Rockville, USA.

Michael Walker played a key role in the organisation of the well-attended conference on food allergy in Queen's University Belfast (QUB) reported in the Training section of this review.

[24] iFAAM – Integrated approaches to food allergen and allergy management, http://research.bmh.manchester.ac.uk/iFAAM/

[25] Dunn Galvin A., Roberts G., Schnadt S., Astley S., Austin M., Blom W.M., Baumert J., Chan C.H., Crevel R.W.R., Grimshaw K.E.C., Kruizinga A.G., Regent L., Taylor S., Walker M., Mills E.N.C., 2019, Evidence-based approaches to the application of precautionary allergen labelling: Report from two iFAAM workshops, Clin. Exp. Allergy, 49(9):1191-1200. doi: 10.1111/cea.13464

International Atomic Energy Agency (IAEA) and Queen's University Belfast (QUB)

The Government Chemist Programme includes a project on "Rapid and Point-of-Test Devices for Food Testing", in which the Scio™ (pocket NIR spectrometer) has been evaluated. This platform has also been used as part of the on-going international trial on the gross adulteration of oregano involving 30 laboratories worldwide. The organisers (QUB and IAEA) have reported initial findings that show the instrument was able to correctly identify five out of the six oregano samples circulated. Further analysis and finalisation of the reported results is underway.

The Government Chemist team also collaborated with both of these organisations to extend the reach of the Food Authenticity Network internationally to try to help build capability and capacity in other countries; Selvarani Elahi gave a presentation on the network at a Ghana Science Conference and workshop on food authenticity at the invitiation of Professor Elliott of QUB and at a meeting of the IAEA Asia-Pacific Technical Regional Cooperation Project in Vietnam.

Scotch Whisky Research Institute (SWRI)

The Government Chemist collaborated with the Scotch Whisky Research Institute (SWRI) to understand if ambient ionisation mass spectrometry could be used as an aid for flavour profiling. This work largely focussed on using the Atmospheric Solids Analysis Probe (ASAP-QDa) instrument to detect a series of volatile flavour compounds identified by SWRI as important for whisky profiling. Initial work indicated that most of these compounds could be detected using the instrument, however, when they were prepared at concentrations typically found in whisky distillates, the compounds could not be detected with high confidence. Our overall conclusion was therefore that ASAP-QDa is not a useful tool for this application.

In addition to the work on flavour profiling, a software called LiveID (provided by Waters) was assessed as a tool for analysing ASAP-QDa data for whisky authenticity. LiveID was used to analyse data generated using the whisky brand sample sets provided by SWRI. Although this was a small study, initial results were encouraging with good predictive accuracy achieved for these samples. LiveID provides an easy to use tool for quickly analysing ambient MS data and it should have many applications in the food area.

Food Authenticity Network

A public-private partnership funding model was developed and has been in operation since January 2019 to support the sustainable growth of the Food Authenticity Network; at the end of 2019, the Network was supported by funding from three organisations with discussions in progress with many more.

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