Introduction
This is the annual report of the Examiners for the Mastership in Chemical Analysis for the year ending 31 December 2017. These general comments are intended for candidates and their counsellors, to help them understand the expectations of the examiners and to aid their preparations for the MChemA.

The MChemA Regulations, Syllabus and Guidance Notes can be found on the RSC website at http://rsc.li/mchema.

Part A

One candidate sat the Part A paper. The five questions selected of the eight questions were 1, 2, 6, 7, and 8.

1. The first two sections of the four-part question probed a candidate’s understanding of three common liquid chromatographic techniques with reference to properties of the mobile and stationary phase, and factors affecting elution order. For reverse phase HPLC the answer would be:
   Silica coated with thin film of liquid stationary phase [non polar] such as Octadecylsiloxane (ODS) [C18]. The corresponding mobile phase will be polar and is typically a mixture of water with methanol or acetonitrile. Interaction requires partitioning (between two liquid phases) and elution order based on polarity.

   Parts 3 and 4 focussed on detectors: their role and operating principles (of two common detectors). In general, detector responds to analytes as they elute from a chromatographic column, generating a chromatogram that provides retention times, heights and areas for each peak. The ideal detector: high sensitivity, high levels of reproducibility, good stability / reliability, ease of use, a linear response over a wide concentration range, rapid response. FID: Similar response to all organics (universal detector). Hydrogen flame burns organics producing charged species that can conduct charge through flame. Signal generated proportional to concentration.

2. This question focussed on recognition and understanding of elemental analysis and the spectroscopic techniques employed in typical analyses.

   From the schematic diagrams, candidates should recognise: ICPAES, FAAS, UVVis and FES.

   Explaining processes involved in making measurements:
   ICP:
   Solution nebulised into plasma.
   Heat of plasma desolvates; vaporises, atomises and excites elements (based on Boltzman distribution).
Excited state elements emit characteristic wavelengths as electron drops down to lower excited or ground state which identifies them – qualitative.
Intensity of emission is directly proportional to concentration – quantitative.

**FAAS:**
Solution nebulised into flame.
Heat of flame desolvates; vaporises, atomises elements.
Hollow-cathode lamp of element to be quantified is used – it emits characteristic wavelengths of that element only.
Choose most sensitive wavelength to monitor absorption by element in the flame – photon promotes ground state electron to an excited state reducing intensity of HCL beam.
Beers law quantifies amount: Absorption directly proportional to concentration.

**UV-vis:**
Solution placed in fixed pathlength cuvette.
Light source to cover UV visible range is deuterium lamp for UV and tungsten lamp for visible.
Analysis requires that there is a UV-visible spectrum produced by analyte species, the maximum of which is used as the wavelength for quantitation.
Beers law applies as analyte species absorbs incident radiation: Absorption directly proportional to concentration.

(b) involved selection of the most appropriate technique (not limited to those identified in previous section) for two analysis examples:
Four group I and II elements per sample, so ideal for emission technique rather than absorption technique.
Sample volume not an issue.
Limit of detection (LOD) not an issue as all techniques can easily deal with >1 ppm.
At that concentration level all techniques have excellent accuracy and precision.
Simple and inexpensive and easy to run (don’t need specialised technical staff, and it would be easy to train the clinical technician to use the instrument), and it can do the elements required – best choice is flame emission spectroscopy FES.
Single element which would immediately suggest absorption technique.
Sample volumes very sample and low LOD required, this can only mean GFAAS ICPAES has required LOD but can’t deal with such small volumes. Could dilute sample, but that would compromise LOD achievable

3. (not attempted)
This question looks at the design and function of pH electrodes and then a more specific ion selective electrodes, F⁻. Recognition of the Nernst equation:
\[ E = C - 59 \log[F^-] \] where slope is ~ 59 mV and its use in determining unknown concentrations by plotting \( E \) (mV) vs \( \log[F^-] \).

4. (not attempted)
Elemental analysis technique ICP-MS was the focus of this three-part question.
(a) Removal of neutrals and photons by taking advantage of their lack of charge using either a shadow stop or off-axis design. Neutrals/photons maintain straight path charged particles deflected to MS by electrostatic lenses.

(b) A – skimmer cone
B – Sampler cone
C – plasma
D – sample
E – To turbo pump

(c) Polyatomic species of same atomic mass as analytical ion of interest for example. Ar polyatomics are particularly notorious eg $^{40}$Ar$^{16}$O interferes with $^{56}$Fe. Collision cell mode uses He which reduces the kinetic energy of the polyatomics and thus preventing their exit from the collision cell into MS. Hydrogen most common (but will accept ammonia or methane). Can breakdown and/or neutralise polyatomics by:
- Chemical reaction: $\text{Ar}_2^+ + \text{H}_2 = \text{ArH} + \text{ArH}^+$
- Charge Transfer: $\text{Ar}_2^+ + \text{H}_2 = \text{ArAr} + \text{H}_2^+$

5. (not attempted)
This four-part question focussed on the theory and application of GC-MS.
(a) Maximising sensitivity:
- Optimum conditions for MS (maximum permissible voltage across the multiplier and full tuning of optics)
- “Clean Source” (sensitivity degenerates with time as the source plates become coated with sample debris)
- “Splitless” injection on GC
- Solvent extraction prior to analysis
- Single Ion Monitoring [SIM] - particularly important

(b) Use SIM – single ion monitoring.

(c) Calculation based on internal standardisation:
\[
\text{(mass HC / mass DD)} = \text{RRF (Area HC / Area DD)}
\]
From multi-standard: \((200 / 50) = \text{RRF (6358 / 1268)} \Rightarrow \text{RRF} = 0.798\)
From sample: \((? / 50) = \text{RRF (2496 / 1245)}\)
\[\Rightarrow ? = 50 \times 0.798 \times (2496 / 1245)\]
\[= 80 \text{ ng (from 200 cm}^3\)]
\[\Rightarrow 0.4 \text{ ng cm}^{-3} \text{ (ppb)}\]

(d) Chlorine (and bromine) have very distinctive isotope patterns in organic molecules and their presence completely dominates any minor effects due to $^{13}$C etc.
(i) $^{12}\text{C}_{10}^{1}\text{H}_{5}^{35}\text{Cl}_{7}$ comes at m/z = 370. Peaks due to presence of $^{37}\text{Cl}$ then occur at 2 amu intervals as follows (taking Cl ratio as 3:1):

(ii) $^{12}\text{C}_{10}^{1}\text{H}_{5}^{35}\text{Cl}_{2}^{79}\text{Br}_{5}$ comes at m/z = 590. Peaks due to presence of $^{37}\text{Cl}$ and $^{81}\text{Br}$ then occur at 2 amu intervals as follows (taking Cl ratio as 3:1 and Br ratio as 1:1)

6. This four-part question focussed on the technique of enzyme linked immunosorbent assay (ELISA):

Looking for the candidate to elaborate on the four topics of:

- Coating/capture: direct or indirect immobilization of antigens to the surface of polystyrene microplate wells.
- Plate blocking: addition of irrelevant protein or other molecule to cover all unsaturated surface-binding sites of the microplate wells.
- Probing/detection: incubation with antigen-specific antibodies that affinity-bind to the antigens.
- Signal measurement: detection of the signal generated via the direct or secondary tag on the specific antibody.

7. This three-part question focusses on organic MS and the different modes of ionisation, ion separation and finally interfacing with HPLC.

(a) (i) Electron Impact Ionisation (EI): The energy of the electrons is significantly greater than that of typical chemical bonds leading to bond cleavage and ion formation. There then follows a process of molecular ion formation, collision, fragmentation and rearrangement to produce a unique and characteristic set of ions for that substance. The same sample will always fragment in the same way, giving the same peaks in the MS.

(ii) Fast Atom Bombardment (FAB): bombarding species are usually rare gases (e.g. Ar, Xe). The gas is first ionised using a filament and then passed through an electric field where it is accelerated to increase KE. After acceleration, the energetic ions are focussed into a secondary collision chamber where they encounter a second supply of gas. Some of the resultant collisions of ions and atoms leads to an exchange of momentum.

(iii) Chemical Ionisation: Gaseous atoms/molecules of the sample are ionised by reaction with other ions produced independently by ionisation (modified EI) of a special chemical reagent gas, e.g. methane, butane, ammonia.

    • Main difference to EI: Reagent has much less energy than electron beam → much less fragmentation (simpler spectra) ie “softer ionisation”.

(b) Quadrupole MS

consists of 4 cylindrical steel rods, rigidly held in position, acts as a “mass filter” – selectively removing all other ions at a particular m/z.

Magnetic Sector:

The ions formed in the ion source (e.g. EI) are accelerated between two slits by a high voltage, V.

When the ions pass into a magnetic field, they are forced (by the field) to travel in circular paths.
Higher m/z resolution obtained with magnetic sector compared with QuadMS.

(c) Advantages of coupling an MS to a chromatographic system (ie MS requires pure single substance for analysis and spectral interpretation and the analysis of a mixture would result in a “mixed mass spectrum”. Chromatography allows the advanced separation of a mixture before MS analysis). Eluent is a liquid and transferring this into the vacuum system is a major problem. Need to preferentially remove the flow of mobile phase without loss of analyte sensitivity.

8. This four-part question looks at proficiency testing and the metrics associated with PT results.
What is PT? Regular distribution of test materials to participating labs for independent testing and results returned to organiser. Assists participants in detection of shortcomings in their test procedures and encourages remedial action.
Why is it necessary? To confirm that analytical labs are actually achieving accurate results for the sample type normally analysed – to monitor continued satisfactory performance.
Use of z score to confirm performance +2 - −2 is satisfactory (recognition that a value near '0' doesn't indicate better performance than say -1.5), −3 to +3 questionable and outside −3 and +3 unsatisfactory.

(b) Assess Aquacheck results – all fine except three:
Chloride at z = 2.5 questionable requires interrogation by lab manager.
>3 and < −3, required more in-depth look. One seemed to be a problem with decimal place and other with transposed numbers.
z-score calculation \( z = (x_p - x_a) / \text{standard deviation (SD)} \) \((x_p = \text{participant value}; x_a = \text{assigned value})\). Use equation to find standard deviation and then use this to determine new z score.

\[
SD = \frac{(97.67 - 89.25)}{0.55} = 15.31
\]

\[
Z \text{ for } 79.38 = \frac{(79.38 - 89.25)}{15.31} = -0.65
\]

(c) Robust statistics use median values not mean – less susceptible to extreme values

Robust mean is median value -

\[Z \text{ score} = \frac{(\text{participant value} - \text{median value})}{\text{MAD}_E}\]

(d) Apply one tailed t-test

Part B

Three candidates attempted paper 1 in October 2017, including one who was re-sitting this single paper. Two of the candidates answered the same questions (1, 3, 5, 7 and 8) and the other answered four of the same five replacing question 5 with question 2. Three candidates also attempted paper 2 in October 2017 and, between them, all eight questions set were attempted.
Question 1(a) asked the candidates to detail the main requirements of Regulation (EC) No 1924/2006 relating to Nutrition and Health claims in food. This is an important piece of legislation which, once qualified, the candidates will be expected to know very well and so the question was testing the breadth of knowledge and understanding in this area. With 14 marks being allocated, a detailed answer was required to cover aspects such as definitions, criteria for use of claims, comparative claims, authorised Health claims, nutrition profiling (showing an understanding that this requirement is still within the law), pictorial claims, brand names/trade marks, non specific claims, restrictions, types of health claim as well as the Annex of authorised nutrition claims. 1(b) looked in more detail at the Annex of the legislation and asked the candidates to explain the requirements of two nutrition claims, ‘source of protein’ and ‘contains vitamin A’. To make the claim relating to protein, the candidates had to know that protein has to contribute at least 12% of the energy of the food and vitamin A has to be present at a significant quantity which is 15% of the reference intake for solid food or 7.5% for drinks.

Question 2, which was only answered by one of the candidates, asked them to discuss the legal requirements for the labelling of fish. The allocation here was 10 marks but a concise summary of the main requirements of the legislation was expected to include products covered, definitions, labelling requirements (scientific name, method of catch, catch area, commercial designations, fishing gear), traceability etc. The second part of the question asked for detail on how the fish content of a coated fish fillet product could be determined. Here the candidates had to describe whether they would separate the fish and coating, and weigh each to begin with, or whether the whole sample should be prepared and tested. Separating the coating, however, could introduce a loss of proteinaceous material. Detailed analysis for the proximate testing (fat, protein, moisture and ash) should have been given to allow any carbohydrate to be identified and corrected for once the nitrogen factors were introduced. The calculation should have also identified that fat has to be taken into account, once the fish content is established, from the ratio of nitrogen to nitrogen factor.

Question 3(a) asked for the meaning of the terms ‘Meat’, Meat product’ and ‘meat preparation’ to be given. These terms are all defined in law and so should have been quoted and explained to obtain the 6 marks.

The second part of the question (14 marks) again tested the candidates knowledge of food analysis, as it asked how a mince sample would be analysed to ensure compliance with the Food Information Regulations. The candidates, therefore, should have been aware of the requirements in Regulation (EC) No 1169/2011 and the tests which should be employed (fat, nitrogen/protein and hydroxyproline). This would allow the limits for fat and the protein / collagen (hydroxyproline x 8) ratio to be assessed allowing categorisation of the mince to be established.

Question 5 involved a discussion on DNA analysis and its application and limitations in determining the composition and safety of foods. One candidate gave a very detailed answer which included its areas of use, a comprehensive overview of real time PCR, quantification,
differences between genomic and mitochondrial DNA, problems in quantifying meat using the technique, fish identification and cytochrome B, cost, problems with mixes, high processing/poor DNA, microbiological applications and GM. Other areas which could have been covered were gene databases, allergens, food provenance, animal breed and plant variety.

Question 7 tested the candidates understanding of the term 'reserved description' and then asked them to detail how they would analytically assess the composition of honey, marmalade, milk chocolate and instant coffee. So a good understanding of legislation including reserved descriptions was needed to answer this question. The Jam, Honey and Chocolate regulations include the criteria for use of the terms ‘marmalade’, ‘honey’ and ‘milk chocolate’ and these should have been included in the answer. The Coffee and Chicory Extract Regulation covers instant coffee.

Question 8 asked the candidates to outline how they would test for five analytes, from a list of eight, and to discuss the significance of the results. Important issues to take into account here were:

i). Ponceau 4R in takeaway meat/sauce meal. This is not permitted in sauces and requires the proportions of meat and sauce to be established before analysis as the sauce component will typically contain the colour and not the meat.

ii). Benzoic acid in an alcopop. The only source of the additive is the soft drink used so the result should be worked back to its proportion in the drink.

iii). Melamine in a milk product. The chemical is not permitted and so an assessment of the safety of the food would be required if it was found.

iv). Mercury in shellfish. Head and thorax meat is excluded from the MRL.

v). Cadmium in crab meat. Limits do not apply to brown meat.

vi). Monosodium glutamate in an instant noodle product. The level found needs to be related to the product as consumed.

vii). Sorbitol in reduced sugar confectionery. Used to sweeten but high levels have laxative effects. Depending on the detection technique, co-elution with other sugars can occur. It is also a multi functional additive (a humectant and a sweetener).

viii). Erucic acid in pickled vegetables in oil. This is a contaminant and the maximum permitted level relates to the oil constituent of a compound food and so results must be calculated in this way.

Questions not attempted by any of the candidates were 4 and 6 which related to materials and articles in contact with food and pesticides respectively. These are more specialist testing areas and therefore not every laboratory (and candidate) will have had the opportunity to gain experience in this area, which could be one reason why the questions were avoided.
Paper 2

Questions 1 and 2 were the policy questions, one relating to sugar and health and the other revolving around the control of non-animal origin food imports.

The ‘sugar’ topic asked the question of how food producers, legislators and manufacturers could assist in improving the health of the nation with regard to the sugar content of food and to give specific examples in the answer of how these changes would effect human health.

The examiners were expecting to see a comment on the link between sugar and obesity/high energy intake and the long term cost in health care. The discussion should have given examples of how producers, legislators and manufacturers could help to improve this giving examples which could include reformulating with zero calorie additives such as stevia (cost/flavour/additive argument), nano-technology (more sweetness using less), alternative sweetening (novel food ingredients and government support to pursue and assess), taxation on high fat/saturates/salt foods also, extending sugar taxation to other foods, tighter accuracy of nutrition information and bring into law, better control on front-of-pack information relating to accurate portion sizes, reducing the cost of smaller pack sizes encouraging larger pack buys which will be consumed and not discarded, forcing nutrient profiles, labelling similar to cigarettes to discourage over consumption of sugar etc.

In question 2, the candidates were expected to explain their understanding of the current system controlling the import of non-animal origin foods into the EU which should have included Regulation (EC) No 669/2009 and the fact that goods covered here can only be brought through approved designated ports of entry (DPE). The list is continually updated and amended with new products coming on, products being withdrawn and products having their frequency of check altered. As the pass rate of tests carried out either improve or reduce, then the frequency of check will be altered. Safety issues will also result in RASFF notifications which will also increase sampling rates (10 consecutive passes required through any DPE before testing is reduced). So this current EU wide system should have been outlined and discussed with a view to commenting on its effectiveness in ensuring food safety in the UK. No plan is fool proof and not all goods are stopped and checked and so part of the discussion could have revolved around this question “do we have the resource to spot problems with goods brought into the UK unchecked”? The question of BREXIT could also have entered the argument and how the system would work when EU legislation is not harmonised with that of the UK if you felt this could be the case.

Question 3 required another essay style approach when the candidates were asked to detail the regulations relating to the use and labelling of additives in animal feeds. This should have been straightforward, if the subject had been thoroughly researched, and the examiners expected the following to be included:

-Additive authorisation and Regulation (EC 429/2008)
-Register of feed additives (Annex I and II) and restrictions for use Annex II additives subject to article 10(2) of Regulation 1831/2003
-Transitional measures following re-registration requirements after Directive 70/524/EC
-Labelling of additives (Regulation (EC) No 767/2009 and tolerances Definition (Regulation EC No 1831/2003 article 5 and examples of additives.
-Safety of additives in excess (Regulation (EC) No 178/2002)
-Histomonostats and coccidiostats (and carryover) (Directive 2002/32/EC)
-The enforcing UK SI Regulations and Act.

Question 4 asked for the definitions of the terms primary, secondary and micro nutrient which should have been essentially a list of the nutrients in these categories with the inclusion that these are essential for plant growth with micro nutrients required in smaller concentrations than those of primary and secondary to benefit the plant. The second part asked how the following affected plant growth with the responses expected to include:

i). Nitrogen (major component of chlorophyll and essential for amino acid/protein and sugar production for growth)
ii). Potassium (regulatory role, stomata operation (CO2/O2/Water uptake and expel), gradient osmosis assistance in root for water uptake)
iii). Sulphur (sugar transport and protein/starch synthesis, chlorophyll production, disease resistance, seed formation, nitrogen fixing)
iv). Molybdenum (nitrogen fixing..nitrate-nitrite-ammonia-amino acids)
v). Magnesium (Photosynthesis-building block of chlorophyll, enzyme and protein synthesis)
vi). Iron (Chlorophyll and protein production, enzyme function, energy transfer and metabolism, nitrogen fixing)
vii). Zinc (growth hormone production, a constituent of enzymes, chlorophyll production).

Question 5 was the only question all three candidates answered which asked them firstly to outline the legislation which controls the contamination of animal feeds. The answer here should have included the principle points of Directive 2002/32/EC (and the enforcing UK SI Regulation), Regulation (EC) 178/2002 (on feed safety), Regulation (EC) No 183/2005 on feed hygiene and any current commission recommendation on chemical or microbiological components, not covered by 2002/32/EC, which may effect feed safety (article 15 of 178/2002).

The second part of the question, which was given equal marks, asked which mycotoxins would normally be associated with a compound feed containing maize, oats, soya and citrus pulp and detail how you would test for them.

Maize is normally associated with fumonisin toxins (B1 and B2) and probably aflatoxin B1 in addition. Oats tend to be more associated with T-2 and HT-2 toxins (and aflatoxin B1), Citrus pulp with citrinin and ochratoxin A and soya is more likely to contain zearalenone (ZON) and aflatoxin B1.

The methods for each are mainly extraction followed by Immunoaffinity extraction/clean-up and HPLC/Fluorescence but LCMSMS analysis after extraction and solid phase clean up can also be applied. Due to the marks allocated, a reasonable outline of the procedure was required e.g. 50g blended with 70% methanol,dilution/buffer, immunoaffinity extraction/concentration followed by HPLC/Fluorescence (FLD) and measurement with potency checked external standards.

Questions 6 and 7 were both only answered by one candidate. In question 6, details on how a formal agriculture sample should be analysed for total phosphorus (non EC Fertiliser), copper (compound feed), calcium (non EC Fertiliser) and sugar (compound
feed) were required. For 5 marks each (20 in total) a reasonably detailed summary of the official methods stated in The Fertilisers (Sampling and Analysis) Regulations 1996 and Commission Regulation (EC) No 152/2009 should have been given to include weights, reagents, acid/base strength, temperatures and times. One of the four procedures (Phosphorus) is gravimetric, two (Ca and sugar) are carried out by titrimetry and copper requires atomic absorption as the final detection and so additional information with regard to these techniques should also have been included.

In question 7, one of the two water questions, the candidates were asked to describe how a drinking water could be tested for trihalomethane’s, taste and odour, iron, cyanide and nitrite. For four marks each (20 marks in total), once again a reasonable outline of each of the procedures should have been given. Trihalomethanes are analysed by GCMS after either solid phase headspace micro extraction or solvent/liquid-liquid extraction (headspace after 150 degree C heating for 60min). External standards of known purity and C13/deuterated internal standards would also be used for quantification. Taste and odour is an interesting one (as these tests tend to be combined in reality) but the examiners were looking for the candidates to mention something about checking bacterial load before the tests were attempted. At least a three panel member assessment can then be carried out, after bottles are shaken, with sufficient headspace required for odour and blank waters to be included for comparison and the odour intensity to be established. For taste, decanting into clean receptacles is needed for a taste score to be given.

Iron is analysed using atomic absorption (or furnace) or ICP and measurement against certified external standards. It is important here to ensure that standards are matched to the samples. Iron response is suppressed with hydrochloride acid strengths below 1%.

Cyanide levels can be established by titration using silver nitrate and a silver sensitive indicator and details of the reagents would be required as well as the way of calculating the level. Alternatively, a distillation technique in alkali conditions can be applied followed by colourimetry using pyridine pyrazolone. Ion chromatography is also an option.

Nitrite is more straightforward and ion chromatography would be the technique of choice here as it is not usually present in high concentrations. Colourimetry using p-aminophenylmercurcaptoacetic acid in the presence of hydrochloride acid is another option, but again reasonable detail on both the techniques (calibration, mobile phase, wavelengths etc) was required.

The final question, question 8, asked the candidates to give the definitions of a bottled water, a spring water and a natural mineral water and, for fourteen marks, detail the legislative requirements relating to their composition and labelling.

A comprehensive knowledge of the Natural Mineral Water, Spring Water and Bottled Water Regulations 2007 was needed here as the definitions are included in this legislation as are all the compositional and labelling requirements for all three types of water.

**Portfolio**

This year, one candidate also successfully completed their portfolio of evidence after the examiners asked for some certificates and labels to be re-worked. A marking scheme was trialled and the candidate was awarded a percentage mark which covered the overall presentation, the candidate’s analytical experience range and
quality of this work as well as the certificates and labels put forward. This marking scheme has since been further adjusted and will be used for future portfolios submitted in 2018 onwards.

**Part C**

Three candidates sat the examination in September 2017. All were successful.

The questions set, as previous examinations, involved three reports, three samples for microscopic examination and two interactive exercises. To pass, a 50% mark overall had to be achieved, with the certificate section demanding at least a 50% mark individually, and both the microscopy and interactive sections had to attain at least 40%.

The certificate questions covered both food and feeding stuffs, the first relating to a traditional shortbread. Here the butyric acid level was low and the fat profile was subsequently effected. So questioning the name of the food, use of the term traditional and contravention under the Food Safety Act was expected. There were some labelling anomalies relating to the nutritional information presented, which would have scored extra marks. This particular question was answered well and the certificates presented were suitably constructed and factually correct.

The second certificate involved an animal feed which was described as a complementary feed. Because this was technically just straw, it was not a mixture of at least two feed materials and therefore could not be described as a complementary feed and was actually a feed material (forage). The clue was in the statutory statement information provided and, as a feed material, the maximum limits associated with the heavy metal levels reported were different. As a result, once corrected to 12% moisture, all the heavy metal levels were satisfactory. There were, however, labelling anomalies associated with the statutory statement information supplied which, as always, would have scored extra marks. If the classification of the feed was incorrect, and the sample reported as unsatisfactory as a result, then the candidates were marked considerably down.

The third certificate was a vodka. The alcohol level was low, and outside the tolerance for a drink with a declared alcohol content of 37.5%, and a trace level of propylene glycol was reported. The key thing here was to recognise that the alcohol was low, quoting the legislation controlling this (Regulation (EU) No 1169/2011), and that the food could not be described as ‘vodka’ due to the fact that the minimum alcohol content of 37.5% was not achieved. The low level of propylene glycol was as a result of the compound being used as a marker. If the units were misread, and the presence deemed to be contaminating the food, then again the question was significantly marked down as this would result in an incorrect interpretation and certificate in real life.

The three microscopy samples tried to reflect typical laboratory scenarios that may be presented. The first was a simple two component mixture of mince and rusk. Here the candidates were expected to identify the characteristic meat muscle fibres but also the presence and type starch in the sample. The second was a three component mixture of marjoram, sage and oregano which was presented in the form of a supplement capsule as each have on-hold health claims relating to them. Herb identification can usually be achieved by recognising the structures of the trichomes associated with each, if a component of the leaf, as well as looking for other characteristics. The
difficulty here was the similarity between marjoram and oregano. The third was a mixture of tobacco and tea, as counterfeit tobacco samples can contain extraneous material to dilute or even completely substitute. Green tea was used here to dilute.

It was pleasing to see the candidates perform well in this section of the paper. In all cases neat, well labelled diagrams were presented with the mounting solutions being recorded and magnifications being highlighted. If reference materials were used to assist identification these were also recorded, or if text books were the means of doing this similar references were included on the exam papers. As always, the examiners looked at the slides prepared to check whether multiple mounts were used to assist the candidates, chemical spot tests were carried out to assist identifications and whether the slides were prepared well and labelled.

The interactive exercises both involved foods, one being a chocolate product with an unusual taste and the other being walnuts (pickled) which had caused illness.

Taking the chocolate product question first, all the candidates successfully identified that the taint was due to rancidity. The important thing here was that, as the product was a snowball with a coconut topping, the chocolate had to be separated from the rest of the components of the compound food before any testing for rancidity could be carried out. All the candidates did this and also requested reference materials of chocolate to compare the rancidity test results against. Material from the same batch would have registered similar results but that from a different batch would not. Only one candidate requested chocolate from a different batch and also noted a colour difference but did not have time to request the rancidity tests on this material. The chocolate had a high peroxide value and free fatty acid. The reference chocolate from the same batch had a low peroxide value but only a slightly lower value than that of the complaint. This was because the chocolate was mainly chocolate flavoured palm oil where the oil had become rancid. Palm oil is naturally high in oleic acid and so, if this was used as an indicator of rancidity, an anomaly was produced. The chocolate therefore did not meet the reserved description for ‘milk chocolate’ and so, if the candidates had requested a suit of tests to check the validity of the description, this the problem would have been spotted.

The second interactive question were pickled walnuts and was set to test the candidates with regard to a potential contamination issues. The way forward in the first instance was to establish the symptoms, and the speed of onset of the illness alleged, which all the candidates did. Further questioning with regard to the number of individuals effected, other foods consumed, the source of the product, the labelling of the food and other circumstances at the time of consumption would have all helped to build a picture of the problem before any analysis was attempted. Consideration, as always, must be given to the potential of microbiological contamination in relation to storage of the sample while questioning, but all the candidates correctly ruled this possibility out. So the route then was to establish an alternative contamination as the onset of the symptoms was very quick (nausea/headache/increased heart rate within an hour).

All the candidates considered an appropriate selection of contaminant tests, including mycotoxins, but only one established the link between the symptoms and high pesticides. In this case it was chlorpyrifos, which is a broad-spectrum insecticide killing insects upon contact by affecting the normal function of the nervous system via inhibiting the breakdown of acetylcholine. The peeled walnuts had been exported from
China and were soaked in the pesticide solution to destroy infestation. The Navel Orangeworm (NOW) and moths (Plodia) are the most common pest for walnuts. These will most likely infest the kernels once the hull begins to split and expose the nut. A lot of credit was given to the candidates for identifying contaminants as the cause of the problem and justifying the tests selected (and providing detailed methodology) even if the ultimate cause was not established.