Introduction

This is the annual report of the Examiners for the Mastership in Chemical Analysis for the year ending 31 December 2018. 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

Two candidates sat this paper and answered the same five questions, 4 – 8.

Omitted Questions:

Questions 1 and 2 were in the general area of food microbiology. Question 1 aimed to test a knowledge of the broad application of PCR assay of food from sample preparation, equipment operation and experimental design through to data interpretation. Question 2 looked for a broad understanding and application of the standard DNA sequencing, Sanger, process to an indication of the impact of Next Generation Sequencing, NGS.

Question 3a focused on volatile organic compounds and extraction methods prior to gas chromatographic analysis. Part b looked for an understanding of the total and selected ion modes of operation in GC/LC-MS.

Attempted Questions:

Question 4 broadly assessed knowledge of the fundamental concepts of gas chromatography. Part a involved calculating concentrations based on method of internal standardisation, one candidate obtained full marks while the other only partial credit. Part b i and ii required determination of standard chromatographic parameters such as plate height...
– neither candidate attempted these sections. Answers to part b iii on varying parameters to optimise the chromatography were vague and contradictory.

**Question 5a** covered basics of atomic spectroscopy in terms of the atomisation process. Both candidates identified the individual steps in the process but were unable to highlight those steps in which problems could occur and how to overcome them. **Part b** involved interpretation and use of a standard addition calibration curve.

**Question 6** required an understanding of the instrument configuration in ICP-OES - marks and critical examination of 3 sets of ICP-OES analyses with respect to accuracy, precision and significance testing. A low score resulted from one candidate confusing atomic emission with atomic absorption and misinterpreting the questions relating to the data sets.

**Question 7** was divided into a number of sections each dealing with different aspects of gas chromatography ranging from types of columns, stationary phases, temperature programming to detectors and their characteristics. Both candidates did reasonably well on this question but disappointingly lacked a more in depth knowledge of such a core analytical technique.

**Question 8** was divided onto 3 sections: (a) on sample digestion techniques in particular microwave digestion; (b) use of control charts and (c) proficiency testing schemes. Both candidates achieved their highest scores. Both achieved full marks for part (b). The lower scoring candidate missed highlighting the more key points of the sample digestion techniques and PT schemes.

**Part B**

Three candidates sat the examination in October 2018, only one sitting paper B2 following success in paper B1 in October 2017. The second candidate was re sitting both papers after an unsuccessful resit of paper B2 in 2017 and the third was sitting both papers for the first time.

**Paper B1**

Of the eight questions set, only one (question 1) was not attempted by either of the candidates who sat the paper. This question requested the legislation relating to added water in meat to be detailed with the second part asking the candidates to explain how you would assess an official sample taken from a poultry processing plant producing whole frozen chickens for the presence of extraneous water. Knowledge of the legislation which controls added water in meat (Regulation (EU) No 1169/2011 and Regulation (EC) No 543/2008) was therefore necessary with the latter describing how to establish the presence of extraneous water in frozen poultry via both moisture and protein analysis.
In Question 2a, for 10 marks, the examiners asked for the main aspects of the Bread and Flour Regulations 1998 which relate to composition and labelling to be summarised. Regulations 4, 5 and 6 here cover the composition of flour, additional ingredients and restrictions on use of the term ‘wholemeal’ and ‘wheatgerm’, and a summary of these should have been included in the answer together with those of schedules 1 and 3 which cover essential ingredients and their required quantities. This is not one of the more common regulations applied in the day to day work of a Public Analyst but, nevertheless, it should be understood.

Question 2b asked the candidates to explain how you would prepare both a bread and a flour before analysing to ensure compliance with the regulations. This is a common challenge, and the key thing here was to explain how you would achieve a homogenous sample. Additives in flour can be notoriously unevenly distributed so an emphasis on mixing should have been covered in the answer. Preparation of bread to produce an even sample is very difficult without air drying first before preparation to obtain an even crumb, any results being back corrected for the moisture lost.

Question 2c asked for the analysis of propionic acid in flour to be detailed. This would be along the lines of a sample weight being mixed with a solution of phosphoric acid, diethyl ether and valeric acid whilst cooling in an ice bath. The supernatant is then injected on to GC for quantification.

Question 3 asked the candidates to detail the legislation relating to composition and labelling of food supplements in a question worth 20 marks. This is a complex area and hence a good understanding is required, which is covered in the Food Supplements (England) Regulations 2003 and Directive 2002/46/EC which it implements. Regulation 6 of the 2003 regulations specifically details the labelling requirements of food supplements and an explanation of this regulation should have been included in the answer. The Schedules and Annexes of the Regulation and Directive also covers the units vitamins and minerals should be declared in and the permitted forms of vitamins and minerals which can be included, both of which should have been mentioned in the answer. Food supplements are a complex area of food legislation, and a common labelling assessment request, therefore a detailed knowledge of this area of law is essential for any practicing Public Analyst.

Question 4 was a further 20 mark ‘essay style’ question asking the candidates to detail the requirements, in legislation and guidance, which control how nutrition information can be presented on a food label. This is a common non-compliant area on all food labels and Regulation (EU) 1169/2011 covers many of the aspects which should have been included in the answer to show an understanding of this area of law. When compulsory, article 30 describes what the mandatory information should and may contain and the form in which it should be given, including how it can be repeated and the criteria for the inclusion of vitamins and minerals. Article 31 includes adaptation of information after food is prepared and Article 32 covers how the information can be expressed, referencing the units in Annex XV and presentation in the form of a percentage of the reference intakes (Annex XIII), and article 33 explains how the information can be given per portion.

Articles 34 and 35 further involve presentation of nutrition information and the additional forms it can be given in (eg Traffic Light format).
In terms of guidance, the published document for the control of compliance with EU legislation on Regulation (EU) No 1169/2011, December 2012 covers how numerical information should be presented and additional guidance from the Department of Health explains the format of ‘front of pack’ information (Guide to creating a front of pack (FoP) nutrition label for pre-packed products sold through retail outlets).

Nutrition claims could also be included, where information not stipulated in Annex XV of 1169/2011 can be given in the same field of vision according to article 10 of Regulation (EC) No 1924/2006.

**Question 5a** asked the candidates to outline the legislation and guidance controlling the labelling and composition of honey. The current Honey Regulations, Part II, covers various honey product names and Part III their criteria for use referencing compositional requirements in schedule 1. Part 4 of the regulation covers additional labelling requirements and general labelling would be covered by Regulation (EU) No 1169/2011 on the provision of food information to consumers. Current guidance also covers voluntary labelling associated with the unsuitability for consumption by children under 12 months of age as recommended by the British Honey Importers and Packers Association.

**Question 5b** asked for a summary of how the authenticity of Manuka honey might be established which is quite topical now due to the high price the product demands thus the opportunity for substitution. In the first instance, the compositional requirements of honey, as stated in the Honey Regulations, could be discussed to ensure the product meets these and use of the reserved description is justified. But other key area to include would be the unique characteristics of manuka honey such as the pollen content (assessing either microscopically of by DNA), its antibacterial properties (which could be assessed via the compound methyl glyoxal) and unique natural compounds present which could be used as a ‘marker, to identify such as Leptosperin.

**Question 6** related to the assignment of a date of minimum durability to a food, with the candidates being asked to discuss the process in relation to a meat product. This was again an ‘essay style’ question worth 20 marks. Many aspects could be included and explained in this answer namely critical control points in the process which could affect durability and a reference to Regulation 2073/2005 which does not permit micro-organisms, their toxins and metabolites to be present in quantities which could be an unacceptable risk to human health. Distribution, storage temperature, packaging and use of the food and quality of the ingredients used could also be mentioned as well as the use of additives to preserve. This may help to set the scene before going onto discuss the processes on how to establish the date of minimum durability which could include estimation during product development (confidence in producing the food consistently from batch to batch), predictive microbiological testing, shelf life testing, challenge testing, organoleptic assessment, open life testing, redox potential, water activity, pH, salt in the water phase, scientific literature searching and historic data. Results should also be considered against safety margins and codes of practice could be referenced.
**Question 7** went slightly back to basics and asked the candidates to explain the function of four additives and how you would detect them. This was set to allow the examiners to see if the candidates had a grasp and an understanding of food additives and the routine techniques used to detect them.

The first was sorbic acid, a preservative and anti-mould compound commonly found in drinks, for which the method of quantification was requested. So an outline of an HPLC procedure, after dilution or direct injection, was expected including UV or diode array detection and measurement using external standards of known purity.

The second was potassium ferrocyanide which is typically used as an anticaking agent in salt. One such direct method of analysis is a colourimetric procedure where ferrous ferrocyanide is produced after complexation which can be measured by UV/visible spectrometry (in the visible spectrum at around 700 nm) and quantified against potassium ferrocyanide external standards of known purity and concentration. Alternatively, an HPLC method could be employed to determine the presence of ferrocyanide ions using a mobile phase comprising sodium perchlorate and sodium hydroxide (NaOH) and using a detection wavelength of 221 nm. Samples are typically just dissolved in NaOH and filtered before injection.

The third additive, which required an identification of its function and how to quantify, was sorbitol. This additive has a dual function in food and can act as both a sweetener and a humectant. Analysis to quantify would be via an HPLC technique after either dissolution and filtration, or further clean up using clearing agents. HPLC coupled with UV-VIS detector, an evaporative light scattering detector (ELSD) or a refractive index detector would allow a sensitive and quantitative assay of the polyol in various confectionery products using external standards of known purity.

Lastly, the function and quantification of nitrite in meat was questioned which is typically used to cure and preserve this food. One such method would be to initially extract the sample with water then warm to 60-70 °C on a water bath for 30 minutes under vigorous stirring. The sample is then cooled and cleared with Carrez reagents (for deproteinization) before analysis by either HPLC/UV, ion chromatography or a colourimetric procedure. In all cases, external standards of known purity should be used to establish a calibration graph for quantification and details of the instrument conditions should be provided.

**Question 8** delved into the area of general claims and looked to assess the candidate’s broader knowledge as the definitions or explanations lay in both guidance and legislation. Of the eight terms included in the question, six could be found the Food Standards Agency’s 2008 Guidance entitled “Criteria for the Use of the Terms Fresh, Pure, Natural etc. in Food Labelling”, a document often used when routinely assessing food labels. These were the terms ‘natural’, ‘pure’, ‘fresh’, ‘traditional’, ‘premium’ and ‘homemade’ and the detailed definitions should just have been given here. The definition and terms of reference for use of the claim ‘organic’ could be found in Council Regulation (EC) No 834/2007. Finally, there is no current definition in law or guidance relating to ‘local’ and therefore the examiners were looking to see if the candidates could use standard dictionary descriptions, or definitions from other reputable sources, to be able to assess the claim.
Paper B2

All three candidates sat this paper and six of the eight questions set, within the three sections of the paper, were attempted. Only questions four and seven did not attract an answer, the first of these being in section 2 (Agriculture). This question related preparation of a ‘salt lick’ and how to analyse for iron, zinc, potassium and iodine. This type of material is very difficult to prepare and may require samples to be removed by boring or breaking at evenly distributed points to obtain a homogeneous sample. Two of the methods of analysis requested were for iron and zinc which are statutory procedures detailed in Regulation (EC) No 152/2009 laying down the methods of sampling and analysis for the official control of feed. An ICP-MS method has recently been approved to insure levels of iodine remain below legislative limits and the level of potassium may be assessed by acid digestion or dry ashing followed by flame photometry or flame emission with a suitable modifier. The second question was in section 3 (Water) where the candidates were asked to give examples of three sources of taints in water, how you would confirm their presence and how you would eliminate the problem. Taints can result from metal contamination (iron, zinc) and these levels can be determined spectrophotometrically. Pipe replacement could resolve. Alternatively bacterial or treatment contamination (chlorine) could be the cause and microbiological or active chlorine testing would confirm with processes being adjusted to prevent in the future. Interestingly, two candidates chose to attempt both questions from section 1 (Policy) rather than an additional question from section 2.

Question 1 (Policy) asked the candidates to outline how the safety of additives is assessed and to give an opinion on their use including examples where they have been replaced or modified as a result of health concerns. All the candidates identified ‘Southampton Colours’ as an example of additives which have had their limits reduced in foods following published papers by Southampton University linking the colours (in conjunction with Benzoic Acid) with effects on attention and activity in children. Although not banned, the levels have been reduced to allow food businesses to seek alternative natural options which will give the food a similar appearance. Other examples could have included nitrites (which have been linked to cancers) or artificial sweeteners-v-natural. In terms of the assessment, the examiners expected the candidates to mention Regulation (EC) No 1333/2008 on food additives, EFSA and the re-evaluation of additives from 20/1/2009 (Regulation (EC) No 257/2010), technical specifications (Regulation (EC) No 231/2012) and separate assessments for children under 12 weeks of age. A discussion along the lines that information for assessment is submitted via governments, universities, research establishments, food companies and other interested parties was expected leading to acceptable daily intakes (ADI) and no observed adverse effect levels (NOAEL) and safety factors being evaluated through scientific assessment thus leading to a suitable assessment of the maximum permitted level set in law to be reviewed.

Question 2 (Policy) asked the candidates to give four examples of the so called ‘free from’ foods which are available to consumers who want to exclude certain ingredients from their diets. Compositional and labelling issues, which arise as a result of the omission or substitution, also had to be explained in the answer. Some good examples were given by the candidates although these mainly revolved around ‘allergen free’ claims but some nutrition components such as sugar and salt were also identified. Other example which could have
been included were alcohol, caffeine, additives and sources of animal (vegan or vegetarian) but the candidates failed to explain the compositional issues which could arise as a result of the ingredient absence. Some examples here could be appearance, flavour, texture, durability, inclusion of additives or the loss of nutrients which could result in deficiencies in the diet and which would have to be replaced. The labelling is covered in existing legislation such as Regulation (EC) No 1924/2006 (nutrition claims), Regulation (EU) No 828/2014 (Gluten) or 1169/2011 (general labelling and allergens). Allergen threshold in guidance could also be covered here.

**Question 3 (Agriculture)** was an ‘essay style’ question worth 20 marks which asked the candidates to detail the labelling requirements of an EC Fertiliser. So a good knowledge of Regulation (EC) No 2003/2003 was required, which is important once the candidates progress, and it was pleasing to see that all three attempted the answer. The labelling of EC fertilisers is covered in articles 6, 9, 10, 11, 17, 18, 19, 21, 23 and Annex I of the regulation and a summary of these areas should have been included in the answer. These cover compulsory statements and identifications, names of the fertiliser itself, the compulsory labelling and markings, languages to be used, the declaration of secondary and primary nutrients and the means of identification of nutrients.

**Question 5 (Agriculture)** was split into two parts and part a asked the candidates to detail the labelling requirements of a complete pet feed. In a similar approach to question 3, a good working knowledge of the legislation (Regulation (EC) No 767/2009) controlling this area of law essential for a future Agricultural Analyst and again it was pleasing to see that all three candidates attempted the question but with varying degrees of success. The examiners expected the candidates to summarise articles 15, 17 and 19 which cover the mandatory labelling information such as the name of the feed, business name and address, approval number (if materials of animal origin present, batch or lot reference number, net quantity, list of additives (if present), moisture content (if necessary), species of animal for which the feed is intended, minimum storage life, list of feed materials, QUID (if necessary), compulsory nutrition information and freephone number or other means of communication. The relevant information in Annexes I, II and VII should also have been included.

**Question 5b** asked what criteria needs to be met before health claims can be made on the same label. This aspect of feed labelling is covered in article 13 of Regulation (EC) 767/2009 namely that any claim is objective, verifiable by the competent authorities and understandable by the user of the feed. Also, the person responsible for the labelling should ensure that the claim is based on scientific substantiation, either by reference to publicly available scientific evidence or through documented company research. A feed shall also not claim that it will prevent, treat or cure a disease, with the exception of coccidiostats and histomonostats.

**Question 6 (Agriculture)** asked the candidates for the details of how you would analyse an official fertiliser sample for total sulphur trioxide, total calcium, water soluble potassium and total nitrogen by iron reduction. These methods are detailed in the Fertilisers (Sampling and Analysis) Regulations 1996, schedule 2, methods 3a (nitrogen), 15 and 20 (calcium), 11 (water soluble potassium) and 16 and 19 (sulphur). Details of each method was worth 5
marks and therefore reasonably concise summaries of each was required which should have included weights of sample, reagent names, temperatures, acid/base molarities, times, extraction procedures and endpoint determination.

Finally, **Question 8 (Water)** asked the candidates to explain the different types of treatment available, and their limitations, to ensure water complies with the current quality regulations. All candidates attempted the question and performed well overall. The answer should have included all the standard means of water purification including flocculation and coagulation, chlorination, sedimentation, other disinfecting techniques (ozonation, UV treatment, brominating), filtration/ultra-filtration, ion exchange and oxygenation. There are limitations to these techniques. Some examples that could have been included here were e.g. that flocculation and precipitation using aluminium based compounds must be controlled to avoid contamination and an effect on appearance of the water could also result from inappropriate use. This technique can also remove fluorides. Chlorination can produce harmful by-products such as trihalomethanes so care is needed (could use chloramine-but this is a corrosive chemical), some bacteria are chlorine resistant (cryptosporidium), ozone has a low half life and so residual may not kill off pathogens and additional disinfectants may need to be added (iron and manganese (nutrients) can also be lost), UV treatment can only destroy microorganisms but cannot remove other contaminants and is effected by water turbidity.

**Part C**

No candidates sat the examination in 2018. One candidate was eligible to attend the practical examination, following a successful portfolio assessment, but deferred for 12 months.

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