



Association of Public Analysts

Study Guide for the Mastership in Chemical Analysis (2nd Edition)

December 2001

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This guide has been prepared by the Training Committee of the Association of Public Analysts. The Training Committee membership at the time of preparation and publication of this booklet was as follows:

M V Clare
D G Forbes (Editor)
P Hancock
C M Hunt (Secretary)
C McDonald
C R Stevens
K J Swan
B Taylor (Chairman)
G S Taylor

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Registered Address:

Burlington House
Piccadilly
London
W1V 0BN

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Correspondence relating to this training guide should be sent to the committee secretary at the address given below.

C M Hunt
West Yorkshire Analytical Services
P O Box 11
Nepshaw Lane South
Morley
Leeds
LS27 0UQ

Tel 0113 3837555
Fax 0113 3837551
E-mail chunt@wyjs.org.uk

Studying for the Mastership in Chemical Analysis Examination

CONTENTS

Section	Page No.
Foreword	3
Introduction	4
The Association of Public Analysts	6
Part A Theory of General Analytical Chemistry	
1 Analysis	7
1.1 Basic Methodology	7
1.2 Calibration and Standardisation	7
1.3 Critical Examination of Source of Error and Interference in Analytical Chemistry	7
1.4 Cryoscopy	7
1.5 DNA Amplification Techniques	8
1.6 Electrochemistry	8
1.7 Enzyme and Immunological Techniques	8
1.8 Gravimetry	9
1.9 Mass Spectrometry	9
1.10 Methods of Measurement including the Determination of Mass	9
1.11 Polarimetry	9
1.12 Radiochemistry	9
1.13 Refractometry	9
1.14 Separation Techniques	9
1.15 Spectrometric Techniques	10
1.16 Titrimetry	10
1.17 Bibliography	10
Part B Theory of Applications of Analytical Chemistry	
1 Food	11
1.1 Analysis with analytical quality assurance (including statistics and measurement reliability)	11
1.2 Composition and Chemistry	12
The Main Classes of Food	12
Additives and Contaminants	16
1.3 Microbiology and microbiological examination	17
1.4 Human Nutrition	19
1.5 Food Storage and Spoilage	20
Processing, Manufacture, Distribution	20
1.6 Safety Aspects of Food	22
1.7 Bibliography	23

Contents

2	Agriculture	26
2.1	Fertilisers	26
2.2	Plant Nutrition	26
2.3	Feeding Stuffs	27
2.4	Animal Nutrition	27
2.5	Bibliography	28
3	Water for Human Consumption	29
3.1	General Introduction	29
3.2	Water Treatment and Distribution	30
3.3	Water Analysis	30
3.4	Water Microbiology	31
3.5	Bibliography	32
4	Pesticide Residues	33
5	Statistics and Quality Assurance	35
5.1	Statistics	35
5.2	Quality Assurance	35
5.3	Bibliography	36
6	Policy and Law	37
6.1	The Law relating to Food, Agriculture and Water for Human Consumption	37
6.2	Policy Relating to Scientific Enforcement and Practice	39
6.3	Bibliography	39

Part C Practical

1	Scope of Study	40
1.1	Practical Analytical Experience	40
1.2	Food	41
1.3	Agriculture	41
1.4	Water for Human Consumption	41
1.5	Microscopy	42
2	Preparing Reports for Legal Purposes	43
2.1	Introduction	43
2.2	The Certificate of a Public Analyst or Food Examiner	43
2.3	Agriculture Act Certificates	43
2.4	Water Reports	44
2.5	Criminal Justice Act 1967	44
2.6	Bibliography	44

FOREWORD

I have great pleasure in commending the second edition of this study guide to the profession of Public Analysts and Scientific Advisers. In particular I trust that candidates for the Mastership in Chemical Analysis (MChemA) examination, their counsellors and others involved with training scientists will find it to be a helpful manual.

The MChemA is the post-graduate qualification prescribed by the Food Safety (Sampling and Qualifications) Regulations 1990 for a person to be appointed as a Public Analyst in the UK. Responsibility for the examination and the award of a Mastership remain with the Royal Society of Chemistry, from whom copies of current regulations and syllabus for the examination may be obtained.

The first edition of this guide was published in 1991. This second edition has been prepared following the revision by the Royal Society of Chemistry of the Regulations and Syllabus for the MChemA in 2000.

This study guide is one of a series of booklets produced by the Association's Training Committee for those preparing for the MChemA examination. Other booklets in the series are 'Legislation', 'Microscopy', 'Candidate's Record of Professional Training and Experience', 'Certificate Writing' and 'Food Complaints'. These publications complement the study guide but are published separately.

The Training Committee would welcome corrections to the text if necessary and constructive comment on ways of improving future editions of this study guide. The Committee acknowledge the considerable editorial input given by Mr Derek Forbes and accept responsibility for any occasions where this advice has not been followed. Correspondence should be sent to the Secretary at the address given at the foot of the acknowledgements page.

Brian Taylor
Training Committee Chairman
Association of Public Analysts

INTRODUCTION

The Public Analyst

There is no organisation which actually bears the name “the Public Analyst Service”. Public Analysts are individuals who are appointed by Food Authorities to carry out responsibilities entrusted to them by the Food Safety Act 1990. They are qualified to do so by virtue of having passed an examination in the chemistry, microbiology and microscopy of food, water and agricultural fertilisers and feeding stuffs originated in 1898 by the then Institute of Chemistry, and accepted by the Local Government Board as the required qualification for Public Analysts. This examination eventually became, first the Branch E of the Fellowship Examination, and then the Mastership of Chemical Analysis of the Royal Society of Chemistry. As the latter, it is incorporated in the Food Safety (Sampling and Qualifications) Regulations 1990 as the qualification certifying the competence of Public Analysts.

Although the examination originated with the analysis of food, drugs and water, in practice Public Analysts undertake work in many other fields and are named in some other legislation as the appropriate persons to carry out analytical work. These areas include consumer protection, pollution and drink-driving offences. Many Public Analysts are also appointed as Agricultural Analysts. However not all Public Analysts will be involved in these fields since there is no statutory requirement for samples relating to consumer protection or pollution to be taken routinely and analysed, and therefore not all local authorities will submit such samples to their respective Public Analyst.

The characteristic which is unique to the Public Analyst profession is the involvement in the analysis of materials linked to an interpretation of the law relating to those materials. This is exemplified in the Public Analyst’s Certificate, the format of which is now defined in the Food Safety (Sampling and Qualifications) Regulations 1990. The certificate is a legal document which declares the result of an analysis of a food and which, unless challenged, will be accepted as evidence in a court of law. This is a right which Public Analysts have won over many years and which should be the aim of all candidates to respect and retain. The importance of the certificate has always been recognised by the examiners and by the members of the profession. Two former Presidents of the Association of Public Analysts have thought it sufficiently important to write papers on the subject in the Association’s journal and candidates should read and understand these papers early on in their preparation. They will learn that brevity is a desirable quality in the preparation of certificates, for the more that is said the greater the chance that counsel will find some irrelevant point on which to criticise the certificate and challenge its soundness.

A clear understanding of the laws relating to the sample being analysed is necessary if the course of the analysis is to be properly planned. At the outset the candidate will be unable to carry out this planning unaided, but should be prepared to assume responsibility as experience is gained. From the start it is essential to learn to observe all the facts concerning the sample and its labelling and description and to make clear records of relevant details.

The Fundamentals

As an educational background, the new entrant requires the following:

- a) a degree leading to graduate membership of the Royal Society of Chemistry or successful completion of the GRSC examination. It is probable that analytical chemistry theory will not have been taught at first degree level to the standard required in modern laboratories. For this reason an examination in the theory of analytical chemistry is now included in the MChemA syllabus, exemption from which can be gained by possession of an approved qualification containing appropriate analytical chemistry, such as an MSc degree. For those without such qualification, Part A of the examination is the first objective they must aim for. For those candidates who do not have access to a course designed specifically for Part A of the examination, a course leading to the Certificate of Applied Chemistry (CAC) will provide a framework of study but must be supplemented with additional reading. For those not able to attend a CAC course, the texts in the series Analytical Chemistry by Open Learning provide an excellent framework of study but must be supplemented by the extra reading. Past examination papers provide a means of assessing the importance of various parts of the syllabus and of the candidate's progress.
- b) in addition, some knowledge of physics, microbiology, biochemistry, molecular biology, botany, mycology, entomology, physiology, food technology and statistics is important. At least one of these subjects may have been studied to 'A' level or higher but it is very unlikely that more than two would have been. Some knowledge of all will eventually be very useful and it may be better if formal instruction at a basic level can be obtained early on. Together with experience gained in the laboratory, a candidate should be able to acquire the necessary expertise.

The Applications of Analytical Chemistry

For Part B, the Association of Public Analysts is taking a leading role in the formal training of Public Analysts through regular courses and through the publication of this study guide which aims to assist candidates to carry out their training in their own laboratories. Over the years with advances in analytical chemistry and their applications through instrumentation, the examination has been brought more in line with practice in the laboratories. Training in these new techniques, where necessary, has been organised by the Association through work-shops. A more general training is carried out in courses arranged by the Association in a two year cycle, held once per year and covering the main aspects of the MChemA syllabus. The study guide aims to fit into this pattern by providing guidance for the continuing training programme, with personal guidance from the counsellors.

The study guide includes bibliographies compiled from literature recommendations which Public Analysts have found to be useful in their work, and supplied by RSC for Part A.

It was decided that web-sites should not be included in the bibliographies. Although they clearly can be a very useful information source it would be difficult to produce a list which would remain current. As an alternative, relevant web-sites with information for candidates will be identified and listed in a section of the Association's web-site (<http://www.the-apa.co.uk>).

THE ASSOCIATION OF PUBLIC ANALYSTS

In the mid-nineteenth century Arthur Hill Hassall, a physician and microscopist, brought to the notice of a wide audience the state of foodstuffs on general retail sale in London. He found among other things that spices were bulked with flour, sawdust and bone ash, milk was watered and toxic pigments of lead, arsenic and mercury were being used to colour confectionery, tea, pickles, bottled fruits and many other foods.

Hassall's work led to the formation in 1857 of a Parliamentary Committee to investigate food adulteration and the Committee's report resulted in 1860 in the first ever Food Act - "An Act for Preventing the Adulteration of Articles of Food or Drink".

Local Authorities were responsible for the administration of this Act (and a succeeding one in 1872 which also included drugs) and the authorities were empowered to appoint Public Analysts to examine the purity of articles of food and drink. However, it was not until the Food and Drugs Act of 1875 that the authorities were *compelled* to appoint Public Analysts.

This 1875 Act made it an offence to sell any food which was not of the nature, substance or quality demanded and this provision, which is the cornerstone of food enforcement law, has been reproduced in every Act up to and including the Food Safety Act 1990.

In order to promote their work, to exchange methods of analysis and agree consistency in enforcement and food standards, Public Analysts formed the Society of Public Analysts in 1874 and one year later they published the Society's Journal known as *The Analyst* of which the aim stated on the frontispiece was "A monthly journal devoted to the advancement of analytical chemistry". Interestingly, because of the Society's precarious financial state, *The Analyst* was run as a commercial concern by two of the members and only taken back into Society ownership in 1904.

The pioneering work of the Society and of many of its individual analytical chemists led to the virtual elimination of the grosser forms of food adulteration by the turn of the 20th century.

Many of the early Public Analysts had originally had skills in other branches of analysis, in medicine or engineering, and the Society decided to institute an examination to be a sign of minimum competence to practice as a Public Analyst. This examination was established in 1898 under the auspices of the Royal Institute of Chemistry.

The Society grew in membership as the Society of Public Analysts and other Analytical Chemists (SPOAC) until it consisted predominantly of non-Public Analysts and *The Analyst* was publishing very little of direct relevance to the food law enforcement scientist.

In order to regain control of their own destiny, Public Analysts broke away from the SPOAC in 1953 and formed The Association of Public Analysts. The remaining analytical chemists formed the Society for Analytical Chemistry which later merged into the Royal Society of Chemistry.

One of the aims of the Association of Public Analysts is to promote the interests of Public Analysts and to maintain the status of the profession. This is carried out by representation of Public Analysts or the Association of Public Analysts on:

Local Bodies - such as regional groups with Trading Standards and Environmental Health Officers

National Bodies - such as British Standards Institution (BSI), Local Authorities Co-ordinating Body on Trading Standards (LACOTS), Local Government Association (LGA) and the Food Standards Agency (FSA)

International Bodies - such as the European Standards body (CEN), Food Law Enforcement Practitioners (FLEP) and European Food Law Association (EFLA)

Part A Theory of General Analytical Chemistry

1 Analysis

For the candidate who needs to take the Part A examination, reference to the Regulations and Syllabus 2000 published by the Royal Society of Chemistry is essential. It is not possible in this guide to give more than an indication of the range of knowledge required of each section of the syllabus.

The object of Part A is to build up a solid foundation of the science on which the applications are based so that the candidate appreciates the reason why certain procedures are insisted on and that short cuts are not taken. Ideally this should be completed before work is started in the laboratory but candidates can aid themselves and other laboratory staff by developing an inquiring mind to find the reasons behind the procedures followed. Above all, it is important to cultivate the art of observation and clear recording of facts as they are encountered. It is necessary for new entrants to the profession to learn to observe and record facts about samples, from submission details, sample labels, and condition of the sample when received, to facts about the analysis itself and the result.

1.1 Basic Methodology

Basic principles underlying analytical chemistry are stated in the following laws or equations:

Law of Mass Action
Le Chatelier Principle
Beer - Lambert Law
Nernst Equation

Study of the texts given in the bibliography at the end of this Part will help the candidate to master the principles.

1.2 Calibration and Standardisation

Absolute methods
Comparative methods
Traceability to national standards
Calibration of volumetric glassware
Checking of balance accuracy
Wavelength and absorption scale checks on spectrophotometers
Temperature measurements

1.3 Critical Examination of Source of Error and Interference in Analytical Chemistry

Accuracy, precision and error
Classification of errors
Methods of expressing precision of a set of results
Confidence limits
Rules for significant figures
Errors due to interference and means of avoidance
Uncertainty of measurement

1.4 Cryoscopy

1.5 DNA Amplification Techniques

Isolation of DNA from cellular components
Factors influencing degradation
Purification
Removal of RNA

Quantification of DNA
Spectrophotometric
Chemical
Enzymic

Detection of specific DNA sequences
Complementary DNA Probes
Southern blotting (DNA fragments)
Northern blotting (mRNA fragments)

DNA sequencing

Polymerase Chain Reaction (PCR)
Selection and use of primers
Competitive or MIMIC PCR
Nested PCR
Real time PCR
Random amplification of polymorphic DNA (RAPD)
Restriction enzymes

1.6 Electrochemistry

Basic principles
Galvanic cells
Electrolysis
Conductance methods
Potentiometry
Voltammetry
Coulometry
Ion selective electrodes

1.7 Enzyme and Immunological Techniques

Enzyme methods
Methods to assess activity of enzymes, e.g. test for previously frozen poultry/meat, diastase activity in honey, phosphatase
Enzymes as an analytical tool, e.g. assays of sugars, alcohol, ascorbic acid and other organic acids

Immunological techniques
Enzyme linked immunosorbent assays
Radio-immunoassay (RIA)
Immunoaffinity chromatography
Serotyping of micro-organisms
Identification of cryptosporidia and other pathogens
Immunohistochemical staining

1.8 Gravimetry

Solubility and solubility product
Formation and properties of precipitates
Co-precipitation phenomena
Water in solids

1.9 Mass Spectrometry

Mass Spectrometry
Formation of the mass spectra
Types of mass analysers
Structural elucidation of simple spectra

HPLC/MS
HPLC/MS interfaces
Methods of ionisation

GC/MS
GC/MS interfaces
Methods of ionisation

Other hyphenated techniques

1.10 Methods of Measurement including the Determination of Mass

Mass and weight
Equal arm and substitution balances
Errors in weighing

1.11 Polarimetry

Specific rotation
Mutarotation
Double polarisation methods

1.12 Radiochemistry

Properties of ionising radiations
Application of radiotracers to analysis

1.13 Refractometry

1.14 Separation Techniques

Distillation
Solvent extraction, partition coefficients
Chromatography, paper, thin-layer, gas, high performance liquid, ion-exchange
Electrophoresis, including capillary
Gel permeation chromatography
Solid phase microextraction

1.15 Spectrometric Techniques

Origins of vibration-rotation and other spectra
Beer-Lambert law, derivation and applications
Infra red, including FTIR
Ultra-violet/visible spectroscopy, including diode array detection
Fluorescence techniques including XRF
Flame emission
Atomic emission
Atomic absorption
Inductively coupled plasmas
ICP/MS
NMR

1.16 Titrimetry

Acidimetry and alkalimetry
Precipitation processes
Oxidation/reduction reactions
Non-aqueous titrations
Chelate titrations
Compleximetry

1.17 Bibliography

The following book list is recommended by the Royal Society of Chemistry:

Skoog D A, West D M, Holler F J, *Analytical Chemistry: an Introduction*, 6th Edition, Saunders College Publishing, (Harcourt Brace), 1994, ISBN 003097285X

Skoog D A, Holler F J, Neiman T A, *Principles of Instrumental Analysis*, 5th Edition, Saunders College Publishing (Harcourt Brace), 1997, ISBN 0030020786

Robards, Haddard, Jackson, *Principles and Practice of Modern Chromatographic Methods*, Academic Press, 1994

Lajunen L H J, *Spectrochemical Analysis by Atomic Absorption and Emission*, Royal Society of Chemistry, 1992, ISBN 0851868738

Miller J C and Miller J N, *Statistics for Analytical Chemistry*, 3rd Edition, Ellis Horwood, 1997, ISBN 0130309907

Fifield F W and Kealey D, *Principles and Practice of Analytical Chemistry*, 3rd Edition, Blackwell Science, 1990, ISBN 0632053844

In addition some techniques and methods not covered by this list should be included in the candidate's reading:

DNA techniques
Enzyme linked immunoassay (ELISA) techniques
Current affairs in analytical chemistry
Current developments in data quality

Part B Theory of Applications of Analytical Chemistry

1 Food

1.1 Analysis with analytical quality assurance (including statistics and measurement reliability)

1.1.1 Sampling, sample preparation and storage of samples

1.1.2 Choice of method of analysis
Degree of accuracy required
Equipment available
Preparation of the sample solution
Presence of interfering materials
Methods of confirmation of result

1.1.3 Analysis, calculation and checking of results

1.2 Composition and Chemistry

- 1.2.1 The major chemical components of food: carbohydrates, fats, nucleic acids and proteins; their structure, properties, reactivity and occurrence in food
- 1.2.2 The minor naturally occurring chemical components: minerals, vitamins, dietary fibre, aromatic substances, natural colours, toxic substances and anti-metabolites
- 1.2.3 Food additives; the nature, properties and function of the various classes of food additives
- 1.2.4 Contaminants of common occurrence which may present a serious hazard in food; substances used in agriculture which may find their way into food
- 1.2.5 The properties of water and of aqueous solutions; water activity; reactions between water and food components
- 1.2.6 The major chemical reactions involving food components induced by enzymes or by processes used in the manufacture of food
- 1.2.7 The chemistry of spoilage; influence of air, light and temperature

The Main Classes of Food

- 1.2.8 Meat and meat products
 - Methods of determination of added water
 - Processes which affect water content
 - Meat content; the Stubbs and More method and variations
 - Identification of species
 - Detection of non-meat nitrogenous matter
 - Preservatives in meat
 - Meat spoilage
 - Identification of offals in admixture with muscle
 - Mechanically recovered meat
 - Quality evaluation
 - Connective tissue assessment
 - Recognition of frozen and thawed poultry
 - Water content of poultry
 - Meat and yeast extract
 - Hydrolysed protein
 - Formed and reformed meat products
- 1.2.9 Fish and fish products (including crustacea)
 - Methods of determination of added water
 - Methods of determination of ice-glaze
 - Identification of species
 - Fish content
 - Spoilage of fish and its detection
 - Physical form of cores (coated products)

1.2.10 Milk and dairy products

Liquid milk, composition and analysis, determination of added water, heat treatment
Contamination and taints in milk, both chemical and bacteriological
Condensed milk, analysis, categories of condensed and dried milk, filled milk
Cream
Cheese
Cheese analogues
Vegetarian cheese
Yogurt
Whey proteins and other whey-based products
Casein
Butter
Butter milk
Butter oil
Ice cream
Milk based drinks
Rennet and chymosin (“vegetarian rennet”)

1.2.11 Sugar and preserves

Classical methods of analysis - copper reduction, polarimetry, refractometry
Separation of sugars by chromatography
Enzymic methods
Identification of brown sugars
Analysis of honey, detection of adulteration
Jams and preserves
Sugar confectionery

1.2.12 Chocolate and chocolate products

Beans, Nibs, etc
White chocolate
Plain (dark) chocolate
Milk chocolate
Liqueur chocolates
Chocolate bloom
Cocoa and fat reduced cocoa
Cocoa and chocolate drinks
Drinking chocolate
Chocolate (flavoured) drinks
Triglyceride analysis
Cocoa butter equivalents (substitutes)

1.2.13 Cereals and flour

- Composition, grades of flour
- Improvers and bleaching agents
- Mineral additions
- Self-raising flour
- Dietary fibre
- Bread and pasta
- Wheatgerm
- Vitamins
- Canned milk puddings
- Biscuits
- Flour confectionery
- Other grain products from barley, oats etc
- Durum wheat
- Gluten
- Folic acid fortification

1.2.14 Oils and fats

- Classical methods - iodine value, saponification value, acid value, peroxide value, density, refractive index, thiocyanogen value, hydroxyl value, acetyl value
- Detection and determination of saturated, mono-, and poly-unsaturated fats
- Detection of cis-cis, cis-trans isomers in oils
- Hydrogenated vegetable oils
- Cholesterol and other sterols
- Solvent contamination and other processing contamination
- Shelf life, methods of determination
- Rancidity, acid value, peroxide value, Kreis test
- Antioxidants
- Erucic acid
- Fatty acid profiles, identification
- Lard
- Spreadable fats
- Olive oil (EC methods of analysis)
- Modified fats e.g. olestra, plant stanol esters

1.2.15 Fruits and vegetables and their products

- Identification of species
- Estimation of fruit content of fruit products (determination of parameters and proportions of mixed fruit)
- Soft drinks, fruit juices and nectars
- Tomato products, mould counts

1.2.16 Fermentation products

- Wine - alcohol content, acidity, contaminants, preservatives, characterisation
- Herb/Fruit/Vegetable wines
- Mead
- Fortified wines
- Natural deposits
- Beer and lager - alcohol, original gravity, flavour components, characterisation
- Spirits - alcohol, brand identity, higher alcohols
- Cider and perry
- Vinegar and non-brewed condiment and substitutes

1.2.17 Spices, herbs and condiments

Chemical characteristics - essential oils, ash, silica, water/alcohol soluble residue
Adulteration and contaminants
Curry powder
Mixtures and stuffings
Salt
 Iodised salt
 Sea salt
 Salt substitutes
 Low sodium salt

1.2.18 Beverages

Tea
 Adulterants
 Simulated teas
 Herb teas (Rooibosch, Maté, Camomile and other herb teas)
Coffee
 Coffee products and mixtures
 Adulterants
 Substitution (dandelion and chicory)
 Instant coffee (including extraction rates)
 Quality of coffee bean e.g. robusta, arabica

1.2.19 Functional foods and Nutraceuticals

Lycopene
Fish oils and omega-3 fatty acids
Phyto-oestrogens
Inulin
Probiotics e.g. bifidus
Prebiotics e.g. fructo-oligosaccharides
Vitamin and/or mineral supplements
Chitosan and other “wonder” slimming products
Carotinoids

1.2.20 Foods containing GMO's

Detection
Quantification
Threshold values
Licensing and authorisations
Effects of processing
Ethical issues
Substantial equivalence

1.2.21 Other miscellaneous foods

- Soups
- Table jellies
- Starch products
- Baking powder
- Baby foods
- Egg and egg products, salad cream, mayonnaise
- Nuts
- Pickles and sauces
- Snack foods
- Sandwiches

Additives and Contaminants

1.2.22 Assay of additives for purity

1.2.23 Detection and determination of the following additives in foods:

- Antioxidants
- Bulking aids
- Colourings
- Emulsifiers and stabilisers
- Flavourings, natural, nature identical, artificial
- Freezants
- Mineral oils
- Miscellaneous additives
- Modified starch
- Preservatives
- Processing aids
- Solvents
- Sweeteners

1.2.24 Detection and determination of contaminants and residues

- Extractives from materials in contact with food
- Fluorides
- Mycotoxins
- Odours and taints
- Pesticide residues (see 4)
- Trace poisonous metals
- Veterinary residues
- Radioactive nuclides

1.3 Microbiology and microbiological examination

1.3.1 Basic microbiology

Microbial morphology

Bacteria, yeasts, fungi, viruses, protozoa

Factors affecting growth

Nutrients

Temperature

Relative humidity

Presence and concentration of environmental gases

Pathogenicity

Infective dosage

Exotoxins, endotoxins

1.3.2 Characteristics of food that affect microbiological growth

pH value

Moisture content and water activity

Oxidation/reduction potential

Nutrient content of food

Anti-microbial constituents

Biological structure

1.3.3 Determination of numbers of micro-organisms in foods

Classical methods

Direct microscopic counts of viable and non-viable cells

Standard plate counts (pour plates v spread plates)

Most probable numbers (MPN)

Dye reduction methods

Enrichment methods

Advanced methods

Fluorescent antibody techniques

Gram negative endotoxins

Electrical impedance

Serotyping

Other methods

1.3.4 Factors affecting microbiological condition of food

Processing treatment and initial bacteriological load

Storage temperature and atmosphere

Hygiene conditions under which the food is processed

Adequacy of packaging, handling and storage

Effects of freezing

1.3.5 Indicators of food quality

Coliform bacteria

Use as indicator of standard of quality of water

Use in dairy and other food products

Total aerobic counts as indicators of hygiene quality

Significance of spoilage organisms such as Pseudomonas species and Lactic Acid bacteria

Yeast and mould count

1.3.6 Microbiological standards criteria

Advisory Committee on Microbiological Safety of Food
Statement of micro-organisms of concern
Class Plan Sampling
 Number and size of samples to be examined
 Appropriate limits for the food in question
 Proportion of samples to conform to the limit

1.3.7 Bacterial food poisoning

Gram positive cocci
 Staphylococci
 Streptococci
Gram positive spore-bearing bacteria
 Cl. perfringens (welchii)
 Cl. botulinum
 B. cereus
Gram positive non-sporing bacteria
 Listeria monocytogenes
Gram negative bacteria
 Salmonella
 Escherichia coli
 Vibrio parahaemolyticus
 Campylobacter jejuni
 Yersinia enterocolitica

1.3.8 Microbiological assays

General principles
Antibiotic assays
Vitamin assays

1.3.9 Microbiological significance of specific food processes

Preservation
 Pasteurisation, sterilisation, smoking, etc.
Temperature control systems
Cook/chill, Sous-vide
Animal husbandry residues
Irradiation
Modified atmosphere packaging
Fermentation

1.4 Human Nutrition

1.4.1 General

Food and its passage through the gastro-intestinal tract, digestion, absorption of nutrients and excretion of residues

The water balance; food as a source of energy and essential nutrients; nutrient contents of raw foods and their natural variation

1.4.2 Classes of substances of dietary value; their nutritive and energy contribution to food

Proteins, peptides and amino acids

Carbohydrates

Lipids

Dietary fibre

Polyols

Ethanol

Organic acids

Polydextrose

Vitamins

Minerals and trace elements

1.4.3 Processing and nutrient content

The effect of processing, distribution and storage on the nutrient content of food

1.4.4 Nutrition and health

Diet related diseases

Food intolerance

Food allergies

1.4.5 Nutritional labelling

Official factors for conversion of protein, fat and carbohydrate to energy values

Recommended Daily Allowances

Health and medicinal claims

1.5 Food Storage and Spoilage

- 1.5.1 The requirements of the food package and of materials used in its production in relation to the food being packaged
- 1.5.2 Identification and properties of packaging materials
- 1.5.3 Methods used for the assessment of specified characteristics
Barrier properties, coating weights, grease resistance, colour, finish, flaws, visual defects, dimensions, integrity of seams and seals
- 1.5.4 Problems of migration and taint
- 1.5.5 Analysis of headspace gases taken from closed containers
Methods for measuring vacua in vacuum packed products
Pressure in cans
- 1.5.6 Minimum durability
Approaches to product shelf life evaluation
Factors affecting shelf life
Sensory testing

Processing, Manufacture, Distribution

- 1.5.7 Food raw materials are subjected to various processes during manufacture in order to present them to the market in a sound, attractive and safe condition. During manufacture of compound foods and meals, other processes can be used so that the finished product may present a different character from that expected from a consideration of the original constituents
- 1.5.8 The standard for comparison

The Public Analyst is required to certify that a sample is of the nature, substance or quality demanded by the purchaser though with the advent of many regulations this is often reduced to ensuring that the sample complies with any relevant statutory requirements. Processing will produce changes in the characteristics of the food concerned and the analyst must know something of the processes involved
- 1.5.9 The principal processing procedures used in this respect are as follows:

Preparation of raw materials

Preservation by a variety of methods including:

Heating (pasteurising, sterilising)

Cooling (chilling, freezing)

Drying

Osmotic preservation (use of sugar or salt)

Acidifying

Chemical preservation

Controlled atmospheres

Irradiation

Combinations of these processes

Other physical processes designed to reduce in size, separate mechanically, form, mix, emulsify, extract, membrane concentrate, distil, bake, roast, fry, coat, fill

Chemical, bio-chemical (including genetic manipulation) and enzyme techniques used to effect conversions and produce new products

1.5.10 Chemical reactions during preparation and processing

During the processing operations changes in the nature of the food may occur; these may include the following:

- Auto-oxidation of fats
- Protein denaturation and hydrolysis
- Non-enzymic reactions causing colour or flavour changes
- Lipolysis
- Surface absorption
- Maillard reaction

1.5.11 Quality assurance and control

Principles of, and approaches to, QA and QC in the food industry
Hazard Analysis and Critical Control Points (HACCP)
Good Manufacturing Practice
ISO 9000 series

1.6 Safety Aspects of Food

1.6.1 Food Hygiene

HACCP

Personal hygiene

Premises hygiene

Premises registration

Toxicological Aspects

1.6.2 Toxins naturally present in food

1.6.3 Food contaminated by contact with the environment

Pollution by industrial discharge

By application of agricultural sprays

Chemicals transferred from wrappings/containers

Chemicals introduced during processing or from additives/cooking vessels

1.6.4 Development of toxins by microbiological growth

Bacteria - see section 1.3.7

Fungi e.g. aflatoxins

Algae

1.7 Bibliography

- Kirk R S and Sawyer R, *Pearson's Composition and Analysis of Foods*, 9th Edn. Longman Scientific and Technical, 1991, ISBN 0582409101
- Ranken M D and Kill R C, Editors, *Food Industries Manual*, Blackie Academic and Professional, 1997
- Sutherland J P, Varram A H and Evans M G, *A Colour Book of Food Quality Control*, Wolfe Publishing Ltd, London, 1986
- Paul A A and Southgate D A T, *McCance and Widdowson's Composition of Foods* (and Supplements), HMSO, London, 1991
- Official Methods of Analysis of the A O A C*, 16th Edn., Association of Official Analytical Chemists, Washington, DC., 1999 (5th revision)
- Martin P G, *Manuals of Food Quality Control*, WHO,
a) 14/1 Food Control Laboratory (FAO Food and Nutrition paper), Rome, 1997
b) 14/2 Additives, Contaminants, Techniques, Rome, 1980
c) 14/7 Food Analysis: General Techniques, Additives, Contaminants and Composition, Rome, 1986
d) 14/8 Food Analysis: Quality, Adulteration and Tests of Identity, Rome, 1986
- Parry J W, *Spices: Their Morphology, Histology & Chemistry*, Food Trade Press Ltd, Westerham, Kent, 1962; Vol. 2, Food Trade Press, London, 1969
- Christie W W, *Lipid Analysis*, Pergamon Press, Oxford, 1982
- Herschdoerfer S M, Editor, *Quality Control in the Food Industry*, Vols 1 - 4, Academic Press, London, 1986
- Patterson, Editor, *Biochemical Identification of Meat Species*, Elsevier, London, 1985
- I.C.U.M.S.A, Schneider F, Editor, *Methods of Sugar Analysis*, British Sugar Corporation, Ltd, Peterborough, 1994
- Bailey A J, Editor, *Recent Advances in the Chemistry of Meat*, Royal Society of Chemistry, Special Publication, No. 74, London, 1984
- Ministry of Agriculture, Fisheries and Food, *Manual of Nutrition*, HMSO, London, 1995
- Department of Health and Social Security, *Diet and Cardiovascular Disease*, Report on Health and Social Subjects No. 28, HMSO, London, 1980
- Bender A E, *Dictionary of Nutrition and Food Technology*, 5th Edn, Butterworth Scientific, London, 1990
- Fellows P J, *Food Processing Technology - Principles and Practice*, Ellis Horwood, 1990, ISBN 0-13-596354-0
- U S Food and Drugs Administration, *Bacteriological Analytical Manual*, Association of Official Analytical Chemists, Arlington, VA, 1995
- Buchanan R E and Gibbons H E, *Bergey's Manual of Determinative Bacteriology*, 9th Edn, Williams & Wilkins Co, Baltimore, 1993
- Smith G, *Introduction to Industrial Microbiology*, Edward Arnold, London, 1986

Part B: Theory of Applications of Analytical Chemistry: Food

Onions A H S, Allsopp D, and Eggins H O W, *Smith's Introduction to Industrial Mycology*, 7th Ed, Edward Arnold, 1981, ISBN 0713128119

Harrigan M E and McCance M E, *Laboratory Methods in Food and Dairy Microbiology*, Revised, Academic Press Inc, London, 1982

Dart, R K, *Microbiology for the Analytical Chemist*, Royal Society of Chemistry, 1996, ISBN 085045244

Vanderzant and Splittstolssen, *A Compendium of Methods for Microbiological Examination of Foods*, 3rd Edn, ISBN 0-87553-173-3

Mound L, Editor, *Common Insect Pests of Stored Food Products*, British Museum (Natural History), London, 1989

Collins Guide to Wildlife in House and Home, ISBN 000219726X

Kurtz O'D L and Harris K L, *Micro-Analytical Entomology for Food Sanitation Control*, Association of Official Analytical Chemists, Washington D C

Busvine J R, *Insects and Hygiene*, 3rd Edition, Chapman and Hall, London, 1980

Crosby N T, *Food Packaging Materials: Aspects of Analysis and Migration of Contaminants* Applied Science Publishers Ltd, London, 1981

Plastics for the Food Industry, British Plastics Federation and British Industries Biological Research Association, London, 1986

Paine F A and Paine H, *A Handbook of Food Packaging*, 2nd Edn, Blackie Academic, 1992, ISBN 0-216-93210-6

Wallis T E, *Analytical Microscopy*, 3rd Edition, J and A Churchill, London, 1965

Flint, O, *Food Microscopy*, Savile Bradbury, ISBN 187274804X

Royal Microscopic Society, *Microscopy Handbooks 01, An Introduction to the Optical Microscope*, Revised Edition 1989, BIOS Scientific Publishers Ltd, Oxford, ISBN 0198564198

Jepson M, *Biological Drawings with notes*, Part I, 28th Impression, John Murray, London, 1982, ISBN 071950726X

Jackson B P and Snowdon D W, *Powdered Vegetable Drugs*, J & A Churchill Ltd, London, 1968

Samson R A, Hoekstra E, Frisvad J C and Filtenborg O, Editors, *Introduction to Food-Borne Fungi*, 4th Ed, Centraalbureau Voor Schimmelcultures, Baarn, Netherlands, ISBN 9070351277

Sawyer R, *Pollen Identification for Beekeepers*, University College Cardiff Press, 1981, ISBN 0906449294

Sawyer R, *Honey Identification*, Cardiff Academic Press, 1988, ISBN 1871254000

Code of Practice on the Declaration of Fish Content in Fish Products, UK Association of Frozen Food Producers, British Frozen Food Federation, British Retail Consortium, British Hospitality Association, Sea Fish Industry Authority, LACOTS, APA

Warner and Eskin, Editors, *Methods to Assess Quality and Stability of Oils and Fat-containing Foods*, AOCS Press, 1995, ISBN 0-935315-58-6

British Meat Manufacturers' Association Advice Notes P0006, P0007, P0008, P0009, P0010, P0014, 11/12 Buckingham Gate, London

Coulter T E, *Food: the Chemistry of its Components*, 2nd Edition, Royal Society of Chemistry, 1989

Dietary Reference Values for Food, Energy and Nutrients in the U K, Report on Health and Social Subjects No 41, Department of Health, HMSO, 1991

Mortimore S and Wallace C, *HACCP - A Practical Approach*, 2nd Edition, Aston Publishers, 1998

Southgate, D A T, *Dietary Fibre Analysis*, Royal Society of Chemistry, 1995, ISBN 085045562

Davidson, A, *The Oxford Companion to Food*, Oxford University Press, 1999, ISBN 0192115790

2 Agriculture

2.1 Fertilisers

- 2.1.1 An Agricultural Analyst must be proficient in the application of statutory analytical methods to the analysis of fertilisers and be able to report on results of analysis in the manner required by the Regulations
- 2.1.2 The methods of analysis of fertilisers are based on general, classical and instrumental techniques. Methods of analysis and the permitted limits of variation between the results obtained and the declared values, are set out in Statutory Instruments which implement European Directives on fertilisers. The legislation also specifies compositional and labelling requirements
- 2.1.3 Analysis of fertiliser samples can include:
Major nutrients
 Nitrogen: total, nitric, ammoniacal, urea
 Phosphorus pentoxide: total, soluble in various media
 Potassium oxide: total and water soluble
Trace elements: boron, cobalt, copper, magnesium
Pesticides and herbicides
Particle size
Neutralising value

2.2 Plant Nutrition

- 2.2.1 General biology
Photosynthesis
Autotropism
Biochemistry of plants
- 2.2.2 Factors affecting growth
Nutrient requirements
pH values
Molecular bonding effects and aspects of soil chemistry related to plant nutrition
Recommended levels of nutrients and fertilisers
Water and nutrient availability
- 2.2.3 Trace elements
Control of concentration is essential
Essential minimum levels
Phytotoxicity threshold

2.3 Feeding Stuffs

2.3.1 The analysis of feeding stuffs involves the use of statutory methods where these are available. In the absence of an appropriate statutory method, other suitably validated methods may be used. Invariably, feeding stuffs need some assessment of their major nutrient composition

2.3.2 Analysis of feeding stuffs can include:

Major components: oil, protein, fibre, starch, sugar, ash, moisture

Minor nutrients: calcium, magnesium, manganese, iron, copper, selenium, iodine

Vitamins and pro-vitamins: vitamins A, D

Additives: anti-oxidants, colourants, preservatives, emulsifiers, stabilisers and, in pet food, acidity regulators

Heavy/dangerous metals: arsenic, cadmium, lead, mercury, zinc,

Anions: fluoride, nitrate

Undesirable substances: aflatoxins, castor plant, ergot, gossypol, hydrocyanic acid, pesticide residues, foreign seeds

2.4 Animal Nutrition

2.4.1 Animal dietetics

Dependence on food in organic form (ultimately from plants)

Traditional feeding, herbivorous and/or omnivorous

Effects of conversion to omnivorous feeding

2.4.2 Essential factors of animal diets

Adequacy of diet; balance of major and minor nutrients

Relative advantages of diet of meat or vegetable produce

Energy lost from diet; efficiency of conversion of diet to useful human food

2.4.3 Additives to animal feeds

Purpose of addition

Legislative implications

2.4.4 Toxicity of feeding stuffs

Presence of plant material toxic to animals

Potential harm from other additions to the diet

2.4.5 Organic production methods

2.5 Bibliography

- MAFF *Fertiliser Recommendations*, 6th Edition, Stationery Office Books Ltd, 1994
- King, J O L, *Veterinary Dietetics*, Bailliere, Tindall and Cox, London, 1961
- Evans, R E, *Rations for Livestock*, MAFF Bulletin No. 48, HMSO, London, 1961
- ADAS, *The Analysis of Agricultural Materials*, MAFF Reference Book No.427, HMSO, London, 1985
- Trace Element Deficiencies in Field Crops*, MAFF Booklet No. 2197, HMSO, London
- Price, C H, *Molecular Approaches to Plant Physiology*
- Millar, C E, *Soil Fertility*
- Frean, W, Editor, *Principles of Food and Agriculture*, Blackwell Scientific Publishers, London, 1992
- WHO, *Handbook of Human Nutritional Requirements*, HMSO, London, 1974
- Bender, A E, *Nutrition and Dietetic Foods*, Leonard Hill, Aylesbury, 1973
- Greer, R, and Woodward, R, *The Good Nutrients Guide*, Robert's Publications, London, 1985
- Tyler, C, *Animal Nutrition*, Chapman and Hall, London, 1964
- Black, N M, *Minerals and Vitamins for Cattle and Sheep - A Plain Man's Guide*, Providence Press, 1990, ISBN 0903803259
- Chandler, E A , *Feline Medicine and Therapeutics*, Blackwell, 1994, ISBN 0632033614
- Gorman, N T, *Canine Medicine and Therapeutics*, Blackwell, 1998, ISBN 0632040459
- Hamence, J H, *Deleterious Ingredients*, J Assoc Publ Analysts 1977, 15, 107
- McDonald, Edwards and Greenhalgh, *Animal Nutrition*, Longman, 1978, ISBN 0582441579
- Nelson R H, *An Introduction to Feeding Farm Livestock*, Pergamon, 1979, ISBN 0080237576
- Ansfield, M, *Immunoassay for Detection of Ruminant Proteins*, Food and Agricultural Immunology, 1994, 419 - 433
- Swan H and Lewis D, *Feed Energy Sources for Livestock*, Butterworths, ISBN 0408707135
- Cooper M R and Johnson A W, *Poisonous Plants in Britain and their Effects on Animals and Man*, Reference Book 161, HMSO, ISBN 0112425291

3 Water for Human Consumption

3.1 General Introduction

3.1.1 Water cycle

Inter-relationships of all water; evapotranspiration, soil moisture deficit, water table, discharge and abstractions, aquifer recharge and compensation flows

3.1.2 Groundwater

Basic geology, sandstone and limestone aquifers; boreholes, wells and springs; general composition,

Possible advantages such as reliable quality, low temperature and protection from surface pollution

Possible disadvantages such as limited yield, fissuring and ingress of chemical or microbial pollution

Spa and mineral waters: origin and characterisation including unusual constituents

Influence of industry, farming and other activities on quality; nitrates, pesticide residues, solvents and other trace organics

Salinity and hardness

Uses of groundwater; domestic, commercial food premises

Legislation and quality

Natural mineral waters/spring waters

3.1.3 Surface waters

Basic composition and variability; vulnerability to quality changes due to weather, agriculture, industry and other activities

Agriculture threats; run off, nutrient enrichment, slurry disposal, pesticide use, special wastes e.g. from fish farms

Industrial and other threats; run-off from urban or industrial premises, trade effluents, sewage effluents, storm overflows, tip leachates

Classification of surface waters

Uses of surface water; water supplies, irrigation, cooling, dilution, navigation and recreation/amenity; influence on quality for human consumption

Legislation and quality

3.1.4 Bottled Water

Legislation and quality

3.1.5 Special issues

Basic virology, Giardia and Cryptosporidia protozoan parasites and water supplies

Legionella

Amoeba and water supplies; blue green algae and toxins in raw water reservoirs

Radioactivity and water

3.2 Water Treatment and Distribution

3.2.1 Simple treatment

Disinfection, types of disinfectant such as chlorine, chloramine, bromine, ozone, ultraviolet, advantages and disadvantages, principles of use and action, values, modes of application and control

3.2.2 Conventional surface water treatments

Pre-treatment, flocculation and sedimentation, rapid filtration, slow sand filtration, flotation, principles, advantages and disadvantages, role of chemical additions, pH control

3.2.3 Advanced treatments

Activated carbon and ozone, ion-exchange, reverse osmosis, desalination, principles, advantages and disadvantages

3.2.4 Fluoridation

Principles, methods and controls

3.2.5 Process and pipe materials including domestic plumbing

Influence on water quality, advantages and disadvantages, cement mortar relining, epoxy lining, other organic lining

3.2.6 Mixing of treated waters

Advantages and disadvantages
Corrosion, precipitation, taste and odour etc

3.3 Water Analysis

3.3.1 General

Sampling techniques including importance of container and sample pre-treatments Factors in selecting analytical method: accuracy, bias, interference etc
Quality control and statistical analysis of results

3.3.2 Chemical analysis for gross constituents

Hardness, minerals, nitrates, etc

3.3.3 Chemical analysis for trace constituents:

metals, pesticides, polyaromatic hydrocarbons, trihalomethanes, solvents etc

3.3.4 Special analysis

Taste and odour
Toxicity testing
Role of gas chromatography and mass spectrometry

3.4 Water Microbiology

- 3.4.1 Organisms indicative of faecal pollution
 - Enumeration and interpretation of results, most probable number (MPN), statistical approach
 - Other micro-organisms
 - Colony counts and their significance
 - Pseudomonas group
 - Nuisance organisms
 - Pathogenic organisms

- 3.4.2 Standards of bacteriological quality
 - Water entering the distribution system
 - Surface water, clarified and disinfected
 - Ground water, disinfected
 - Samples from the distribution system
 - Private supplies
 - Supplies for particular locations or uses
 - Emergency procedures
 - Special samples and consumer complaints
 - European Community: requirements of the Water Directive
 - Comparison of the Community approach with traditional UK approach - see relevant legislation

- 3.4.3 Sampling
 - Frequency of sampling for bacteriological examination
 - Collection, storage and transport of samples
 - Sampling techniques

- 3.4.4 Methods of examination
 - Laboratory hygiene and safety
 - Preparation of samples
 - Determination of indicator organisms
 - Multiple tube method
 - Membrane filtration
 - Rapid methods

3.5 Bibliography

Twort, Hoather and Law, *Water Supply*, Hodder and Stoughton, London, 2000

Holden, W S, Editor, *Thresh, Beale and Suckling: The Examination of Waters and Water Supplies (Revised: E. W. Taylor; 1958)* J and A Churchill, London, 1970

Water Treatment Technology, DoE, HMSO, London, 1979

Hunt D T E and Wilson A L, *The Chemical Analysis of Waters, (General Principles and Techniques)*, 2nd Edition, Royal Society of Chemistry, London, 1993, ISBN 0851867979

Greenberg, A E and Eaton A D, *Standard Methods for the Examination of Water and Waste Water*, American Public Health Association, Washington, D C, 2000

WHO, *Guidelines for Drinking Water Quality*, Volumes 1-3, HMSO, London, 1984 -1986

DoE Standing Committee of Analysts, *Methods of Examination of Water and Associated Materials*, HMSO, London, 1976 onwards

The Bacteriological Examination of Drinking Water Supplies 1982, HMSO, London, 1982

DoE *Guidance on Safeguarding the Quality of Public Water Supplies*, HMSO, London, 1989

Cheeseman, R V and Wilson, A L, *A Manual on Analytical Quality Control for the Water Industry*, NS30, Water Research Centre, 1989

The Microbiology of Water 1994 Part 1, Drinking Water Report on Public Health and Medical Subjects No 71, HMSO

4 Pesticide Residues

- 4.1 Definitions of pesticides;
implications of differences between definitions in the Food and Environment Protection Act 1985 and the Pesticides (Maximum Residues in Crops, Food and Feeding Stuffs) (England and Wales) Regulations 1999 (there are equivalent regulations for Scotland in 2000)
- 4.2 Definitions of Acceptable Daily Intake and Maximum Residue Limit
Implications of the differences between the two
- 4.3 Sampling procedures
Recommended method of sampling for the determination of pesticide residues (CAC/PR5-1984)
Implications of differences between Food Safety Act and Codex sampling provisions
- 4.4 Portion of produce to be examined
Portion of commodities to which Codex maximum residue limits apply and which is analysed (CAC/PR6-1984)
- 4.5 Major classes of pesticides
Organochlorine
Organophosphorus
Thiocarbamates
Chlorophenoxy acids
Triazines
Miscellaneous pesticides
- 4.6 Pesticide/commodity combinations
Choice of range of pesticides to determine
Detection limits/reporting limits
- 4.7 Analytical procedures
Extraction of pesticides from specimen
Choice of solvent systems
Continuous hot extraction
Continuous cold extraction
Manual extraction
Co-distillation
Supercritical fluid extraction
Safety precautions in handling solvents
Clean-up procedures
Need for clean-up
Solvent partitioning
Column chromatography; silica, alumina, carbon, fluorosil, use of silver nitrate
Large columns, micro columns, solid phase extraction techniques
Gel permeation chromatography
Chemical techniques
Co-sweep distillation techniques
Final determination
Choice of procedure to use
Gas liquid chromatography; choice of columns, choice of detectors, detector types, derivatisation techniques
HPLC; choice of columns, choice of detectors, optimisation of detectors, choice of solvent systems
Colorimetric techniques
Mass spectrometry and “combined” mass spectrometry

4.8 Confirmation

Need for confirmation, false positive results

Use of alternative gas liquid chromatography columns

Use of alternative gas liquid chromatography detector systems

Chemical techniques

Diode array detector techniques

Mass spectrometry by full scan and selective ion (theoretical understanding only)

5 Statistics and Quality Assurance

5.1 Statistics

- Types of error
- Mean, Standard Deviations and Distribution
- Confidence limits
- Propagation
- Significance tests
- Quality control and sampling
- Errors associated with instrumentation
- Non-parametric errors
- Robust methods
- Experimental design, optimisation and pattern recognition
- Proficiency testing

5.2 Quality Assurance

- The development of a quality assurance system
- Accreditation of laboratories for various functions
- Standardisation of methods
- ISO 9000 Series
- BS EN ISO/IEC 17025:2000 - standard for Official Food Control Laboratories
- The APA protocol on quality assurance
- External quality assurance schemes e.g. FAPAS, FEPAS, Aquacheck
- APA approved methods of analysis

5.3 Bibliography

Miller J C and Miller J N, 3rd Edition, *Statistics for Analytical Chemistry*, Ellis Horwood, 1997, ISBN 0130309907

Moroney M J, *Facts from Figures*, 2nd Edition, Harmondsworth Penguin, 1953 (1978 reprint)

Wood R, Nilsson A and Wallin H, *Quality in the Food Analysis Laboratory*, Royal Society of Chemistry, 1998, ISBN 085404566X

Caulcutt R and Boddy R, *Statistics for Analytical Chemists*, Chapman and Hall, 1983, ISBN 041223730X

Crosby N T and Prichard F E, *Quality in the Analytical Chemistry Laboratory*, (Analytical Chemistry by Open Learning), John Wiley and Sons, 1995

Lawn R E, Thompson M and Walker R, *Proficiency Testing in Analytical Chemistry*, Royal Society of Chemistry, 1997, ISBN 0854044329

6 Policy and Law

6.1 The Law relating to Food, Agriculture and Water for Human Consumption

6.1.1 It is of importance for the Public Analyst to have a thorough working knowledge of the law as applied to the various fields of work. Such knowledge will have primary influence upon the selection of work to be carried out on samples submitted for analysis and examination, the planning of scientific investigations and surveys to be undertaken and the interpretation of scientific results with a view to report and possible legislative action

6.1.2 Candidates must appreciate that legislation is in a state of continual evolution and it is therefore important to keep abreast of the potential, and actual, changes in the law by regular attention to case law reports, the enactment of relevant new legislation, EC Regulations and Directives, recommendations of advisory bodies such as the Food Advisory Committee together with a knowledge of proposals for regulations as issued from time to time by the various government ministries and departments

6.1.3 Principle legislation involved: The Food Safety Act, 1990

Candidates should have a good overall understanding of the entire Act but the following sections are of particular importance:

Section 1:	Meaning of food and other basic expressions
Section 2:	Meaning of sale etc
Section 5:	Food authorities and authorised officers
Section 7:	Rendering food injurious to health
Section 8:	Selling food not complying with food safety requirements
Section 9:	Inspection and seizure of suspected food
Section 14:	Selling food not of the nature or substance or quality demanded
Section 15:	Falsely describing or presenting food
Section 21:	Defence of due diligence
Section 27:	Appointment of Public Analysts
Section 28:	Provision of facilities for examinations
Section 29:	Procurement of samples
Section 30:	Analysis etc of samples (including issue of certificates of analysis)
Section 31:	Regulation of sampling and analysis etc
Section 34:	Time limit for prosecutions
Section 40:	Power to issue Codes of Practice
Section 41:	Power to require returns
Section 42:	Default powers
Section 53:	General interpretation

The act is much shorter than the predecessor Food Act 1984 but candidates should be aware of the powers conferred on Ministers to make Regulations and to issue Codes of Practice. A good working knowledge of, and understanding of the principles of, the various Regulations made under the Act will be required

6.1.4 Subordinate Regulations of the Food Safety Act

Regulations will include those relating to food composition, food additives, contaminants, labelling, food treatments, hygiene and miscellaneous matters such as materials and articles in contact with food and Regulations covering the appointment and functions of Public Analysts

6.1.5 The European Communities Act, 1972

Certain subordinate regulations made under this Act have a direct bearing on the work of the Public Analyst, particularly in the fields of food and water, e.g. milk and milk products, water content of frozen poultrymeat.

In addition to EC Regulations, students should also be familiar with the various EC Directives which generally become implemented by enactment through national regulations

6.1.6 The Food and Environment Protection Act, 1985

In particular its importance in relation to pesticide usage and pesticide residues

6.1.7 The Agriculture Act, 1970

Part IV of the Act governs the sale, composition and labelling of fertilisers and feeding stuffs and provides for the appointment of Agricultural Analysts. Subordinate Regulations made under the Act deal separately with fertilisers and animal feeding stuffs, and lay down prescribed methods of analysis and reporting requirements, by way of prescribed certificates, for such articles

6.1.8 The Trade Descriptions Acts, 1968 and 1972

Provision of analytical and advisory services are frequently required in support of proceedings taken by Local Authorities under the Acts

6.1.9 The Water Act, 1989

An understanding of the Act, its associated EC Directive and the Water Supply (Water Quality) Regulations, is necessary

6.1.10 Further Study

It is strongly recommended that the prime source of study should be the original documents, i.e. Acts, Regulations, Committee Reports, etc themselves. Additionally this should be supported by the study of appropriate law reports as appearing in various publications. Of value in this respect are the monthly Bulletins of the APA, which also provide information in respect of new legislation

Note: A separate guide listing relevant primary and secondary legislation has been published by the Training Committee of the Association of Public Analysts. It is entitled "Legislation" and is upgraded on a regular basis. The latest edition (4th) was published in 1999.

6.2 Policy Relating to Scientific Enforcement and Practice

6.2.1 Current situation

Food

Regulations under the Food Safety Act 1990: few prescribed methods of analysis, value of compositional standards, views for and against.
Enforcement through food authorities: in England and Wales these are the London boroughs, district councils, metropolitan authorities and unitary authorities; in Scotland the authorities are the islands or district councils.

Agricultural materials

Enforcement of Agriculture Act 1970 by similar authorities as above; analysis by prescribed methods

Water for human consumption

Appointment of Public Analysts and Agricultural Analysts by relevant authorities

6.2.2 Future development

Awareness of continuing and new developments

EC legislation in form of regulations and directives; directives incorporated into statutory instruments

Changes in emphasis under EC legislation from compositional standards to informative labelling

Developments in food analysis

Change in emphasis from retail to factory sampling; desirability of retention of some retail sampling; application of scientific expertise to factory inspection, problems arising from unequal distribution of factories to be inspected between food authorities

Developments in food law enforcement

The framework agreement on local authority food law enforcement

6.3 Bibliography

Atwood B, *Butterworths Food Law*, 2nd Edition, Butterworths, Reed Elsevier (UK), 2000, ISBN 0406895481

Rowell R, General Editor, *Butterworths Law of Food and Drugs*, Butterworths, London, 1999, ISBN 0406996474

Part C Practical

The Part C examination consists of:

the submission of a **portfolio of evidence** which demonstrates

- (i) practical analytical experience
- (ii) proficiency in preparation of certificates and reports
- (iii) knowledge of product labelling

and an **examination** involving the candidate in

- (iv) microscopic identification of unknown materials
- (v) interactive exercises
- (vi) certificate writing

1 Scope of Study

1.1 Practical Analytical Experience

Evidence of practical experience across a range of analytical techniques. It is not necessary to have competence in all the techniques listed below, but candidates should be able to demonstrate involvement in a cross-section during the course of their career:

- Classical wet chemistry
- Mass Spectrometry
- Atomic Absorption/Emission Spectroscopy
- Inductively Coupled Plasma Spectrometry
- High Performance Liquid Chromatography
- Gas Chromatography
- Non-instrumental Chromatography
- ELISA
- Microscopy
- Infra Red Spectrometry
- Materials and Articles in Contact with Food
- Electrophoresis
- DNA measurement techniques

The depth of involvement in each technique should be declared, e.g. authorised analyst, method validation, research, etc. The examiners will usually verify the contents of this part of the portfolio through a visit to the candidate's laboratory. For the interactive exercises candidates will be expected to have knowledge of the following topics and to be able to apply that knowledge and their experience in completing the various exercises and questions.

1.2 Food

Analysis of and interpretation of analytical data for the main categories of foods for general chemical composition, additives and contaminants:

- Meat and meat products
- Fish and fish products
- Milk and dairy products
- Sugars and preserves
- Cereals and flour
- Oils and fats
- Fruits and vegetables and products
- Fermentation products
- Spices, herbs and condiments
- Beverages
- Other miscellaneous foods

Additionally candidates will be expected to have the ability to identify chemically or microscopically the various foods and food ingredients and appropriate additives and contaminants.

1.3 Agriculture

Candidates will be expected to be familiar with methods found in relevant regulations made under the Agriculture Act, be able to interpret results from these methods and use them in preparation of appropriate reports and certificates.

1.4 Water for Human Consumption

Candidates will be expected to be familiar with methods used for analysis of water for human consumption such as those published by the Standing Committee of Analysts (Blue Book methods). They will also be expected to be able to demonstrate knowledge of the microbiological examination of water and to be able to interpret the results of both chemical analysis and microbiological examination.

1.5 Microscopy

1.5.1 The application of microscopy in the fields of botany, zoology, microbiology, histology, entomology, mycology. In particular, applications in these fields which are necessary for the investigation of a wide range of complaints relating to food and drinking water including biochemical examination of body fluids, urine, crystals and blood cells

1.5.2 Analytical microscopy procedures

Mounting slides; techniques and mountants

Chemical reactions and microphysical techniques

Staining techniques

Quantitative procedures

Use of polarised light, dispersion staining, reflected and transmitted light

1.5.3 Vegetable microscopy

Botanical structures of plants; roots and rhizomes, stems, woods and barks, leaves, flowers, seeds and fruits, fibres and hairs

Powdered vegetable material; pollen, starches, cereals, spices, ground food substances, paper and packing material

Moulds and fungi (edible and inedible)

1.5.4 Animal microscopy

Histology of animal tissues (edible and inedible)

Animal hairs, feathers

Basic entomological structures and distinguishing features, common pests

Microscopy of water, aquatic species, animal culae, fish scales, cryptosporidium

Parasites, internal and external, bacteria

Potential food and water contaminants

1.5.5 Mineral and some other organic aspects

Fibres; glass, mineral wool, asbestos, man-made fibres, plastics

Dusts

1.5.6 In addition, microscopical examination is required for the identification of components of feeds and for the recognition of vegetable contaminants. The analyst must also be able to detect any deterioration and infestation in a feeding stuff

Note: *Detailed lists of specimens for study are given in the separate guide 'Specimens for Microscopy' published by the Training Committee of the Association of Public Analysts*

2 Preparing Reports for Legal Purposes

2.1 Introduction

Competence in certificate writing will need to be demonstrated in both the portfolio of evidence and during the practical examination. For the portfolio of evidence between 40 and 60 examples of the candidate's best efforts should be submitted.

An appreciation is required of the role of the Certificate as a legal document which may, under prescribed circumstances, be offered in Court as evidence and be sufficient evidence of the facts contained therein. The Certificate must be drawn up in a manner such as to offer no chance for its validity to be questioned. The need must be recognised for its wording to be direct, factual and concise, ignoring all irrelevant matter, while providing sufficient information such that a court may be enabled to come to a judgement regarding the case before it. A contrast has to be seen between this and the more general style of report which serves largely as a communicative document which may be much more expansive and of a discursive nature as occasion demands.

Proficiency in the drafting of official certificates may best be achieved by regular practice involving simulated conditions, taking advice from counsellors and senior colleagues, together with the establishment of a close rapport with regard to certificates issued from the candidate's own laboratory.

2.2 The Certificate of a Public Analyst or Food Examiner

A knowledge and appreciation is required of:

The provisions of the Food Safety Act 1990 and Regulations made thereunder governing the various requirements and use of the Certificate of Public Analyst or Food Examiner

The prescribed form of Certificate as laid down in the Food Safety (Sampling and Qualifications) Regulations 1990 (and a comparison with the Public Analysts Regulations, 1957 SI No 273)

2.3 Agriculture Act Certificates

Prescribed forms of certificates of analysis for fertilisers and feeding stuffs are required by Section 77(4) of Part IV of the Agriculture Act 1970. Familiarity with the structure and application of such certificates is necessary, separate certificate forms being prescribed for fertilisers and for feeding stuffs.

For fertilisers, the prescribed form of certificate is laid down by Regulation 7 and Schedule 3 of the Fertilisers (Sampling and Analysis) Regulations 1978 (SI 1978 No 1108). A full understanding is also required of the guidance notes which accompany the prescribed form, such understanding being directly dependent upon awareness of the requirements of the Fertilisers Regulations 1990 (SI 1990 No 887), particularly with regard to the prescribed statement of certain compositional details (the statutory statement) and specified limits of variation.

For feeding stuffs, the prescribed form of certificate is laid down by Regulation 7 and Schedule 3 of the Feeding Stuff (Sampling and Analysis) Regulations 1999 (SI 1999 No 1663), appropriate guidance notes similarly being included. A proper understanding of these is required together with the related requirements of the Feeding Stuff Regulations 2000 (SI 2000 No 2481) with regard to statutory statements and limits of variation.

2.4 Water Reports

There is no prescribed format for the reporting of water results but the same principles apply:

- (i) What is the result?
- (ii) What is the standard?
- (iii) What is the deviation?

The information should be presented in a clear format easily followed by the client.

2.5 Criminal Justice Act 1967 - Proof by written statement

The general provisions of Section 9 of the Act for proof by written statement should be understood, together with an understanding of the conditions laid down for the admissibility of such a statement as evidence.

Recognition should be accorded to the value and application of such a statement in circumstances where no specific prescribed certificate is laid down including its use in relation to articles of foodstuffs (e.g. consumer complaints) not sampled in accordance with the Food Safety Act, articles analysed under The Trade Descriptions Act and articles analysed under Consumer Protection/Consumer Safety legislation.

Note: A separate guide entitled “Certificate Writing” has been published by the Training Committee of the Association of Public Analysts.

2.6 Bibliography

Moir D D, *The Certificate of the Public Analyst*, J Assoc Publ Analysts, 1965, 3, 3

Hamence J H, *The History of the Public Analyst's Certificate and Present Day Requirements*, J Assoc Publ Analysts, 1973, 11, 59

Painter A A, Editor, *Butterworths Law of Food and Drugs*, (as amended), Butterworth, London, 1980

Certificate Writing - Training Guide, Association of Public Analysts, 1994