Undergraduate Teaching Syllabus in Analytical Chemistry

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Undergraduate Teaching Syllabus in Analytical Chemistry

Fourth Report by the Committee of the Education and Training Group of the Analytical Division of the Royal Society of Chemistry

The current members of the Committee are:

Dr. E. J. Greenhow (Chairman)  Dr. J. D. R. Thomas
Mr. A. F. Smith (Vice-Chairman)  Dr. L. A. Gifford
Dr. J. F. Tyson (Honorary Secretary)  Dr. C. Graham
Mrs. M. I. Arnold (Honorary Treasurer)  Dr. A. Townshend
Dr. E. K. Harrison  Dr. J. G. Pritchard
Dr. C. W. McLeod

Former members of the Committee under the Chairmanship of Dr. J. G. Pritchard were Dr. D. M. W. Anderson, Mr. H. A. Glastonbury, Mr. B. Mills, Dr. J. M. Skinner and Professor D. Thorburn Burns.

Introduction

This syllabus is put forward for the purpose of meeting deficiencies in the analytical chemistry content of many of the undergraduate chemistry courses in the United Kingdom.

In the past few years it has been argued that, in general, undergraduate training in analytical chemistry in the United Kingdom is inadequate to meet the current requirements of industry and that extensive in-service training and/or supplementary postgraduate work is necessary to remedy this situation. The first and second reports of the Committee have pointed out the generally low level of staffing in analytical chemistry as opposed to other areas of chemistry in most universities.\(^1\)\(^2\) The polytechnic sphere contains relatively more emphasis on analytical chemistry.\(^1\)\(^2\) The third report of the Committee has stressed the general opinion that exists in industry to the effect that new graduates who present themselves for careers primarily as analysts are, in many respects, insufficiently prepared for the job.\(^5\) In addition, individual opinions to this effect have been widely publicised.\(^4\) The Royal Society of Chemistry published, in July 1980, the report of a Working Party on "The Supply of and Demand for Analytical Chemists" and items from this report, including the results of a survey among employers of analytical chemists, have recently been highlighted.\(^5\)\(^6\)

One of the main conclusions from the survey was that employers of analytical chemists are not, on the whole, satisfied with the standard of training of applicants for analytical chemistry posts. The main objective of the present report is to meet these criticisms with a strong recommendation for positive action. The recommendations are intended to provide graduates with a broader appreciation of chemistry and its application, and to prepare them better for available job opportunities.

The Committee has taken the standpoint that Masters' courses in analytical chemistry (under various titles) are, at present, at least in part remedial in nature.\(^3\) Consequently, if the situation were remedied at the Bachelors' level, Masterships would inevitably increase in depth and/or in specialisation to the levels which the prior preparation of the student would then permit. Such changes would not detract from the value of specialist Bachelors' degrees in analytical science or related subjects, for which an MSc course or PhD research course in analytical chemistry constitute a legitimate follow-up for those aspiring to an advanced degree. The Committee has confined its thought and effort to the basic material that needs to be assimilated by all students at undergraduate level. While shorter courses below first degree level may well need improvement as far as analytical chemistry content is concerned, the Committee has regarded as its first priority an attempt to raise to a uniform
standard the analytical chemistry subject matter taught in undergraduate courses.

The aims of the Committee in constructing the undergraduate syllabus were thus as follows.

1. To combine and condense longer established aspects of analytical chemistry with the newer, more instrumental aspects of the subject in order to produce a balanced unit reflecting the extent to which the various analytical methods are currently utilised.
2. To produce a course that takes a relatively small fraction of the time allocated to undergraduate training.
3. To produce a course that integrates other branches of chemistry (and physics) and exemplifies their application.
4. To provide a well balanced lecture scheme which, when combined with a substantial and realistic practical schedule, should allow students to develop positive practical skills and confidence in analysis, and to maximise their potential as chemists, whatever their intended career specialisation might be.

**General Plan**

The proposed course falls conveniently into two main parts. The first is a coherent basic course of approximately one conventional “course unit” in duration, a total of 173 hours, which may be taught in two sections (phases) if this is desirable. The second part is an optional, more advanced unit of 85 hours. The distribution of time to be devoted to analytical chemistry is thus:

<table>
<thead>
<tr>
<th>Basic unit—</th>
<th>Lecture hours</th>
<th>Practical hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase 1—Analytical Reaction Chemistry</td>
<td>18</td>
<td>49 + 67</td>
</tr>
<tr>
<td>Phase 2—General Analytical Chemistry</td>
<td>46</td>
<td>60 = 106</td>
</tr>
<tr>
<td><strong>Total 173</strong></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Optional unit—</th>
<th>Lecture hours</th>
<th>Practical hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase 3—More Advanced Topics in Analytical Chemistry</td>
<td>30</td>
<td>55 = 85</td>
</tr>
<tr>
<td><strong>Total 85</strong></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The Committee has specified the lecture topics and the assigned hours in considerable detail below. The amount of time assigned to practical classes, 173 and 85 hours, are, in the Committee’s judgement, sensible amounts, appropriate to a well balanced undergraduate course. The basic unit amounts to no more than one-ninth of a typical undergraduate course, and successful completion of the basic unit should enable the graduate to start useful employment in industry as an analytical chemist. However, students who also complete Phase 3 should regard themselves as well on the way to becoming analytical specialists.

The Committee has recognised that the analytical chemistry course must be co-ordinated carefully with other parts of an undergraduate chemist’s normal training and education. This co-ordination should be updated each Session so that there are no significant gaps in the transition from “basic” chemistry to analytical chemistry. It is not the Committee's purpose to dictate the precise manner in which this might be done, but the following two approaches readily suggest themselves, the second of which is preferred.

(i) The basic analytical chemistry unit could be placed in time after the undergraduate has completed basic units of descriptive inorganic and organic chemistry and also spectroscopy, thermodynamics, kinetics, electrochemistry, optics, electronics and the mathematics of elementary statistics. In this instance the analytical unit would appear best placed in the latter part of a three-year course. The basic 173-hour unit might be run, for example, for one six-hour working day per week for 25 weeks, the approximate length of the university undergraduate's in-college working period per year, with one additional lecture per week on another day during the same period (or otherwise, as suits the particular establishment). Any fragments of the proposed analytical chemistry lecture course (such as the theory of certain instrumental methods) which have been well covered in an earlier part of the three-year period, need not be repeated unnecessarily, in which case the appropriate analytical chemistry lecture time so released might
best be used for additional analytical chemistry practical work.

(ii) The second, preferred, approach that suggests itself stresses how important it is for the undergraduate to have the opportunity to specialise in analytical chemistry and to be committed to it early in his undergraduate life if he so desires, just as he is able to do with the other branches of chemistry. The proposed syllabus is thus presented in three phases so designed that the first phase can be taken in the first year of an undergraduate course, with phases two and three following in years two and three, respectively. This procedure should facilitate the integration of the analytical units with the other parts of an undergraduate course.

While the Committee believes that a course such as the one proposed is necessary if the student is to have a thorough grounding in analytical chemistry, it is realised that some universities and polytechnics will require a shorter basic or "core" course and will be reluctant to allocate time for more than, say, 30 lectures and 60 hours of practical work. A short course of this type, intended to cover the bare essentials of analytical chemistry and to enable the institution concerned to claim that the student has reached a "level of competence" in analytical chemistry, could be constructed by judicious selection from the course recommended below. The content of such a course would depend to some extent on whether or not the institution had any specialisation in analytical chemistry.

The Committee wish to stress that the core course must, together with topics covered in other courses, form a cohesive whole, and should not be merely a collection of unrelated sets of lectures utilising the time allocated.

No attempt has been made to design a set of lectures and practical work suitable for a "service course" in analytical chemistry, intended to satisfy the requirements of non-chemists, e.g., metallurgists, geologists or biologists, as it would be extremely difficult to establish the level of background knowledge in chemistry of the students concerned.

THE LECTURE COURSE

Basic Unit

Phase I—Analytical Reaction Chemistry

A. Scope of analytical reactions

<table>
<thead>
<tr>
<th>Lecture</th>
<th>Title</th>
<th>Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>Introductory review</td>
<td>Scope and applications of analytical chemistry. Chemical equilibria, quantitative reactions and solution chemistry. Coordination, complex formation and chelating. Selectivity, masking.</td>
<td>5</td>
</tr>
<tr>
<td>B. Titrimetric analysis</td>
<td>Acid - base, redox, complexometric, non-aqueous, indicators.</td>
<td>5</td>
</tr>
<tr>
<td>C. Gravimetric analysis</td>
<td>Theory of crystal formation, co-precipitation, elementary gravimetry, weighing forms, amplification reactions; precipitation from homogeneous solution, titrimetric precipitation reactions.</td>
<td>4</td>
</tr>
<tr>
<td>D. Colorimetry</td>
<td>Spectrophotometric reagents, elementary visible spectrophotometry, spectrophotometric titrations, turbidimetric methods.</td>
<td>4</td>
</tr>
</tbody>
</table>

Phase 2—General analytical science

A. General procedures in analytical science

(i) The several steps in analytical procedures, accuracy, propagation of errors, precision, limit of detection, heterogeneity of samples, sampling technique.

(ii) Pre-treatment of samples to obtain homogeneous solutions, statistical treatment.

(iii) Rejection of outliers, Student's-t function, F-test.

(iv) Regression lines, calibration graphs, method of standard additions.

(v) Systematic errors, standard materials and procedures, standard and official methods, pharmacopoeias.

B. Separatory analytical methods

(i) Distillation, partition theory and solvent extraction.

(ii) Principles of chromatography; theoretical plates, resolution and band spreading. Paper,
thin-layer, ion-exchange, gel-permeation and gas-liquid partition methods.

(iii) High-performance liquid chromatography.

(iv) Electrophoresis.

C. Instrumental methods

(i) General principles of spectrometers.

(ii) Analytical application of ultraviolet-visible absorption spectroscopy. Fluorescence and phosphorescence.


(iv) Infrared spectrophotometry; basic theory; solid, liquid and gas samples; group frequencies, quantitative uses.

(v) Potentiometry (pH and ion-selective electrodes), polarography, coulometric, potentiometric, conductimetric, titrations.

(vi) Radiochemical methods.

(vii) Immunoassay.

(viii) Elementary mass spectrometry of organic compounds; determination of molecular formulae and partial structure.

(ix) X-ray methods.

D. General application of analytical science

(i) Qualitative analysis of unknown samples. Schemes of wet qualitative analysis for inorganic ions, organic compounds and functional groups.

(ii) Automation of quantitative analytical procedures; applications of microprocessors; on-line determination; flow injection; continuous flow analysis; data processing.

Optimal Unit

Phase 3—More-advanced Topics in Analytical Chemistry

Lecture hours

(i) Optical activity in analytical chemistry; rotatory dispersion; dichroism. 5

(ii) Nuclear magnetic resonance spectroscopy, quantitative applications. 2

(iii) Auger electron spectrometry, ESCA. Applications to surface analysis, other surface techniques. 1

(iv) Advanced mass spectrometry; various analysers and sources; inorganic and organic analysis; isotope methods. Gas chromatography - mass spectrometry. 3

(v) Advanced electroanalytical techniques; anodic-stripping voltammetry; pulse polarography. 3

(vi) Advanced immunoassay and electrophoretic techniques. 2

(vii) Radiochemical methods of analysis; neutron activation analysis; γ-ray spectrometry. 4

(viii) Thermal analysis; thermogravimetry; DTA; DSC. 1

(ix) Kinetic methods of analysis and use of bio- and chemiluminescence in analytical chemistry. 1

(x) Statistical comparison of results from different laboratories; sampling and correlation of analytical data with origin of samples, significance of correlation by "t" statistic. 2

(xi) Advanced atomic spectrometry; electrothermal atomisation; arcs, sparks and plasma emission. 3

Schedule of Practical Work

Illustrative, useful practical work is a vital part of a chemist's training. It is intended that the following schedule be used as a suggestion pool from which the tutors concerned can select experiments best suited to their facilities and inclinations. The experiments are listed in sections which refer to the foregoing lecture syllabus. Most of these exercises and many alternatives can be found clearly described in standard texts on chemical analysis and also in
reference works like British Standard Specifications and the British Pharmacopoeia. The following two publications on recognised methods of analysis are also excellent sources of analytical exercises for students:


Phase 1A  Selected library exercises.

PHASE 1B

1. Use of sodium carbonate as a primary standard in acidimetry.
2. Standardisation of sodium hydroxide solution with potassium hydrogen phthalate and conventional indicator.
3. Titrations with permanganate, dichromate and vanadium to illustrate oxidation - reduction.
4. The cerium(IV), arsenic(III) oxidation-reduction reaction catalysed by iodide.
5. Saponification value for a dark-coloured specimen of fat or wax by potentiometric titration.
6. Iodine value of unsaturated fatty acid standards and of given unknown fat or wax.
7. Iodine - iodide coulometric titrations.
8. Determination of iodide in iodised table salt by microtitration of iodine with thiosulphate (back-titration with iodine).

Phase 1C

1. Gravimetric determination of sulphate as BaSO₄.
2. Determination of calcium by EDTA.
3. Determination of phosphate in a fertiliser by gravimetry.
4. Examination of dithizonates of various metals.
5. Determination of aluminium and nickel.
7. Determination of molybdenum by spectrophotometric estimation of molybdenum thiocyanate extracted into 3-methylbutanol.
8. Gravimetric determination of piperazine adipate following reaction with 2,4,6-trinitrophenol (British Pharmacopoeia 1980).

PHASE 1D

1. Photometric titration [e.g., arsenic(III) with triiodide ion].

PHASE 2A

1. Literature search problem, e.g., what methods are available for the determination of sulphur dioxide in the atmosphere?
2. Repeated use of pipette and determination of statistics on the results of individual and class efforts.
3. Acid treatment of gelatin and hair to obtain clear solutions of trace cations.
4. Wet and dry combustion - oxidation to prepare a sample of foodstuff for analytical determination of trace metals. Illustration of loss of volatile metals by atomic-absorption spectrophotometry.
5. Determination of sodium chloride in selected samples from a (heterogeneous) finely powdered salt - sand mixture. Determination of the heterogeneity of the mixture by the 99%-point of the "t" distribution, and examination of a representative sample.

PHASE 2B

1. Chromatographic identification of dyes used in coloured, boiled sweets.
3. Thin-layer chromatography of phosphates and phenylhydrazones.
4. Chromatographic purification and spectrophotometric determination of aspirin.
5. Ion-exchange separations of zinc and cadmium, and of sodium and copper.
6. HPLC analysis of analgesic tablets.

PHASE 2C

1. Determination of copper and nickel in a cupronickel alloy by controlled potential electrode position.
2. Characterisation of surfactants in commercial detergent preparations by infrared spectroscopy.
3. Atomic-absorption spectrophotometric determination of calcium and magnesium in tap water. Determination of sodium and potassium in plasma or urine by flame atomic emission.
5. Ion-selective electrode determination of sulphide.
6. Preparation and de-gassing of solution for polarography and determination of cadmium.

7. Determination of sulphur dioxide in the air through formation of potassium dichlorosulphitomercurate(II) and estimation of this spectrophotometrically through conversion of acid-bleached $p$-rosaniline to $p$-rosanilinemethylsulphonic acid (West-Gaeke method).

8. Checking an ultraviolet-visible spectrometer.


10. Determination of calcium in barium nitrate by flame emission photometry on an ethanol extract versus a further extract blank.

11. Quantitative analysis of dimethylbenzene for the proportion of ortho, meta and para isomers by infrared spectrophotometry.

12. Determination of vitamin A by ultraviolet-visible spectrophotometry.

13. Determination of quinine (and interferences therewith) by spectrofluorimetry.

14. Potentiometric titration of iron(II) with cerium(IV).

15. Determination of iron in an ore by reaction of iron(III) with tin(II) and potentiometric back-titration of this with cerium(IV).

**Phase 2D**

1. Qualitative analysis of $n$-radical mixtures.

2. Qualitative analysis of a commercial detergent preparation.


**Phase 3**

For the practical work of Phase 3 the lecturer should demonstrate a selection of the more advanced techniques dealt with in the lectures, and the student should carry out entirely with his own hands some of the more lengthy and searching experiments in the foregoing lists for Phases 1 and 2.

**References**


