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Classical Analysis in the University

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At Loughborough University of Technology (LUT), analytical chemistry is taught as a coherent topic to all undergraduate courses (chemistry, medicinal chemical, chemistry and polymer science and technology, and chemistry and analytical science). During the first 2 years, analytical topics are delivered as such at the rate of one lecture per week in conjunction with tutorials and a 10 × 3 h laboratory class in year 1 and a 13 × 3 h laboratory class in year 2. Analytical topics are also taught during the spectroscopy course as “applications of atomic spectroscopy,” etc. One of the final year option courses offered to the chemistry course contains the applications of analytical topics to the chemistry of biologically active systems. The chemistry and analytical science course naturally contains more analytical topics as the courses separate, after a common first year, for a second and final year of increasing specialisation. In addition, LUT runs a 12 month MSc course in Analytical Chemistry and Instrumentation, of which the first 2 terms of 10 weeks comprise formal lecture laboratory and tutorial classes.

The so-called “Classical analysis” topics are taught in 12 lectures to year 1 (together with 30 h of laboratory work) and in 10 lectures to the MSc students (together with a small number of experiments). These topics are summarised in Table 1. It is unfortunate that this artificial division of analytical chemistry into “classical” and “instrumental” methods has arisen and is perpetuated by many text-books. Students, all too readily, are given the impression that “classical” methods are old-fashioned, obsolete and boring and that instrumental methods are modern, high-tech. and exciting. Not only that, but their experiences in the laboratory may lead them to believe that “classical” methods do not work, and that instruments always give an answer to five or six significant figures which must be right as the digits were produced as the result of a computer-based calculation.

Table 1. “Classical Analysis” topics taught at LUT

<table>
<thead>
<tr>
<th>Reactions between ions</th>
<th>EDTA titrations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Complexes</td>
<td>Redox equilibria</td>
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<tr>
<td>Stability constants</td>
<td>Non-aqueous titrations</td>
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<tr>
<td>Study of complex formation in solution</td>
<td>Dissolution methods</td>
</tr>
<tr>
<td>Acid base equilibria</td>
<td>Speciation studies</td>
</tr>
</tbody>
</table>

Buffers

The first eight lectures to year 1 at LUT are designed to present an over-all philosophy of analytical chemistry in which it is not necessary to make this distinction between “classical” and “instrumental” topics. The basis of this philosophy is shown in Fig. 1, from which it can be seen that the role of the analytical chemist is to provide information about a bulk material. This information, which usually concerns the chemical composition, is used as the basis for making a decision about the bulk material. The analytical chemist’s job is to choose the most appropriate means of providing the information, having first ascertained, (a), that the client’s perception of the problem is sensible, and (b), what constraints will be imposed by the client, and then to assess the quality of the information, i.e., provide some indication of the reliability of the result.

In addition to providing this philosophical framework for analytical topics, the introductory course aims to show the role of chemistry in the provision of information about bulk materials. Students are reminded of the basic principles of qualitative analysis (perform diagnostic chemical reaction and compare result with either memory or that of an authentic specimen, make deduction, confirm) and of titrimetry and gravimetry. Students are then introduced to the idea that chemical reactions which do not have very large equilibrium constants can be used for analytical purposes provided that a large excess of the reagent is added and that the amount of product can be measured by some means. A useful illustration of this principle is the spectrophotometric determination of chloride by use of a reagent based on mercury(II) thiocyanate and iron(III). The sensitivity of this reaction is readily demonstrated (using the overhead projector as “spectrophotometer” and a glass beaker as “cuvette”) and the concept of trace analysis is readily introduced. This paves the way for some general introductory remarks about chemical instrumentation and the lid is taken off the black box to the extent shown in Fig. 2. Part of the reasoning behind introducing chemical instrumentation at such an early stage is that it pre-empts another popular misconception about “instrumental” analysis, namely that all of the chemistry goes on in beakers outside the instrument and once a sub-sample of the pre-treated sample...
has been introduced into the instrument chemistry ceases and can thus be forgotten. This is obviously nonsense for, say, HPLC, GLC or atomic absorption spectrometry, where a good understanding of the internal chemistry is required if the instrument is to be operated properly. Even for those techniques in which no chemical reaction takes place inside the instrument, the "chemistry" of the sample material has to be understood in order that the results may be correctly interpreted (e.g., the chemical shifts observed in nuclear magnetic resonance, Auger electron and X-ray photoelectron spectroscopies).

![Diagram](image)

**Fig. 2.** Taking the lid off the "black box." A general scheme of a chemical instrument

In addition to teaching undergraduate and MSc students, universities also have the function of conducting research. The Chemistry Department at LUT firmly believes in the philosophy that the most effective teaching is done by teachers who are also active researchers, and thus the research in analytical chemistry covers most of the spectrum of research topics shown in Fig. 3. One major area of research activity is in the use and development of flow injection (FI) techniques. In its simplest form, flow injection analysis (FIA) is merely a way of replacing the beaker or graduated flask reaction vessel together with the operation of transfer of the reaction product to the measurement cell of the appropriate instrument, with the operation of loading the loop of an injection valve and actuating it. The contents of the loop are injected into a continuously flowing carrier stream containing a suitable reagent. The flow patterns in the narrow bore (typically 0.5 mm) tube produce interdispersion of the injected sample and carrier stream reagent and the reaction product is monitored at a downstream flow-through detector. Precise control over the injection and pumping processes allows quantitative analyses based on peak height to be made, and an existing analytical method is enhanced in terms of speed, convenience and economy of reagent.

![Diagram](image)

**Fig. 3.** The spectrum of analytical chemistry research which can also be viewed as developing the procedures and devices which provide the links in the chain between the "bulk material" and "decision" of Fig. 1.

However, FI techniques are capable of extending the capabilities of analytical methods in other ways. If the conditions are adjusted so that reaction is incomplete in the centre of the sample zone (for example, by injecting a large volume, say 500 µl), then two reaction zones are produced, one on the leading edge and one on the trailing edge of the dispersed sample zone. Thus, if the reaction product is monitored, two peaks will be observed (see Fig. 4). If the dispersed profiles are exponential in shape (as produced by passage through a well stirred mixing chamber), the time interval between the peaks is proportional to the log of the concentration of the injected sample.1 As can be seen from Fig. 4, there is a point on the leading and on the trailing edge where the concentrations of sample and reagent are in their stoichiometric ratios (in this instance 1:1). Thus, the measured time interval is between the equivalence points of two titrations, and a so-called "classical" technique, when adapted to an FI format, allows the working range of a spectrophotometric method to be extended by several orders of magnitude.2

![Diagram](image)

**Fig. 4.** Formation of the flow injection double peak: (a), physical dispersion of reagent, A, and sample, B; (b), concentration profiles after chemical reaction showing double peak for the product, C.

To a large extent Liebhafsky's fear3 that the chemistry is going out of analytical chemistry has not proved correct, although the chemistry has moved to locations other than reaction vessels on the bench. Titrimetry and gravimetry do not warrant the designation "classical," they are methods to be used where appropriate by the practising analytical chemist. However, as Chalmers4 has pointed out, "the gravimetric and titrimetric methods will always play an important role in the education process and must have their place in the teaching of analytical chemistry. In any chemistry course, they provide almost the only part in which physical, inorganic and organic chemistry are brought together to show how the apparently autonomous branches of chemistry really form part of the whole, which according to tradition must necessarily be more than the sum of its parts; analysis is the only unifying branch of chemistry."

**References**