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Enric Casassas Memorial Lecture 2002

Some highlights and perceptions of analytical chemistry*

J. D. R. Thomas**
Emeritus Professor of Chemistry and Applied Chemistry, University of Wales, Cardiff, Wales

Abstract

Analytical Chemistry is perceived in terms of the author's experiences in industry, the public service, teaching, research and consultancy. Views are expressed on the evolution of Analytical Chemistry in the last half century, and also on aspects of its significance and role in decision-making, matters of curricula development (including student projects) and *'Continuing Professional Development'*, and research.

Although chemical sensors figure highly in the author's research contributions, the focus here has been more on his other work in the analytical field, including separations by electrophoresis, ion-exchange and foam chromatography, and of polarography. Finally, there are reflections on the miscellaneous challenges made of Analytical Chemistry, and of demands of the future as offering exciting prospects.

Keywords: Analytical Chemistry, image, education, teaching, research, prospects.

Dedication

This paper is dedicated to the late Professor Enric Casassas (1920-2000). A central figure of his beloved Catalonia, Professor Casassas was revered for his wisdom in guiding colleagues, for his reasoned ideas, and for his contributions to analytical chemistry. He promoted university education in Catalonia. Stimulated by two years (1954-55) in the USA with Professor I. M. Kolthoff at the University of Minnesota, USA, Professor Casassas went on to produce almost 200 publications and he supervised the research for around 30 doctorates.

Catalonia is in an ideal setting for analytical chemistry in terms of water and air quality. Professor Casassas used this to advantage by applying electrochemistry and spectroscopy for speciation studies and calculations of stability constants of metal complexes, particularly of macromolecular ligands of environmental and biochemical interest.

The life and work of Professor Casassas have been well documented in an Obituary by Professors Miquel Esteban and Cristina Ariño [23], and in an earlier biography by Professor Salvador Alegret [1]. The tributes to their esteemed mentor highlight facets of a momentous half century in Analytical Chemistry during when *'wet chemistry'* laboratories have been almost overtaken by instrument halls, principally for chromatography and various spectroscopies. In diverse ways the life and work of Professor Casassas over this exciting period relate to many of my experiences and views, as reflected by the following account of an activity that impacts heavily on human endeavour and well-being.

It has been a privilege to have witnessed developments in Analytical Chemistry in Catalonia. This was by invitations from Professor J. Albaiges (CSIC – Institute of Bio-Organic Chemistry, Barcelona) to successive *International Congresses on Analytical Techniques in Environmental Chemistry* in 1978 and the 1980s, and then in 1984 as a guest of Universitat Autònoma de Barcelona (UAB) at Bellaterra in association with Universitat de Barcelona and the Universitat Politècnica de Catalunya. Furthermore, by Professor J. Albaiges being editor of the *International Journal of Environmental Analytical Chemistry*, of which it has been an honour to have served on its Advisory Board, Catalonia has gained further prominence in Analytical Chemistry.

Chemical and bio-sensors

It is with chemical– and bio-sensors that I am often associated. But there is much else besides in a lifetime spent as a chemist, first in industry, and then in public service, teaching, and in research and consultancy. The Analytical Chemistry part sets the theme of this paper, modelled as it is on the author's L.S. Theobald Award Lecture [102] at the Royal Society of Chemistry's (RSC) Analytical Division (AD) meeting at the University of Bradford on 28 March 2001.

As we have some common ground with the Sensors and Biosensors Group at UAB, Bellaterra, it is appropriate to first highlight some of our work on sensors, i.e., before taking a wider view of Analytical Chemistry, ranging from concepts and practice on to aspects of education and to its development by research and consultancy.

The torrent of interest in chemical sensors was stimulated by ion-selective electrodes (ISEs), which stemmed from glass electrodes for hydrogen and sodium [51]. By 1967 the field had opened out to membranes based on crystals, liquid ion-exchangers and neutral carriers [51].

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** Author for correspondence: JDRTHOMAS@aol.com

Despite the demand for selective ion measurements, particularly in the biomedical field, the nature of the then liquid membrane devices was a hindrance. This was eased by trapping liquid ion-exchanger in a PVC matrix [16,59]. PVC electrodes were widely acclaimed, and ISE manufacturers have since used the PVC technique [25]. In fact, it made liquid ion-exchanger and neutral carrier type ISEs commercially feasible.

By the almost universal use of ISEs for blood electrolyte measurements in hospitals for clinical diagnoses[25], we can claim to have a hand in monitoring human health! This derives from our work at the University of Wales in Cardiff, helped by developments elsewhere, and by standardisations of the *International Federation of Clinical Chemistry (IFCC)*. The calibration of blood electrolyte systems by potentiometric ISE technology, as an attractive alternative and indeed as a replacement of flame photometric measurements, are set out in a review by a former Cardiff co-worker [14].

The Cardiff researches on chemo– and bio-sensors flourished, year by year from 1967 until my retirement, when the UK's RSC (Analytical Division) held its three-day *'International Symposium on Electroanalysis'* of April 1994 in Cardiff. Professor Salvador Alegret of UAB at Bellaterra was at the Symposium with a paper on *Application of an ISE for the Automated Titration of Anionic Surfacants*. This matured an earlier link between Catalonia (Universitat Autònoma de Barcelona), Portugal (Universidade do Porto) and Wales (UWIST, i.e., the present University of Wales, Cardiff which is recognised by its public name of *'Cardiff University'*). The paper was published [2] in the Symposium Special Issue of *The Analyst*.

[*Interestingly, the links between Catalonia and Wales are on a high plane, as in April 2001 Catalonia's President Jordi Pujol was the recipient of an honorary doctorate (LL.D. – Doctor of Laws) of the University of Wales. This was* «***in recognition of his role in Catalonia's development and in the promotion of regional partnerships within the EU***»*. President Pujol took particular pleasure in receiving the honour from the University of Wales as the nation of Wales has much in common with Catalonia, not least in its national character*.]

Our sensor work covered many aspects of ISEs [88,90,94,95,97], including using the MINIQUAD computer programme for speciation and chemometrics for calibration, both of them matching some interests of Professor Casassas. We explored new sensors, including those for neutral non-ionic surfactants. Attention was later given to amperometric enzyme electrodes for food-based components and other analytes, and to catalytic metal oxide membrane electrodes for hydrogen peroxide. As these are well covered in earlier reviews [41-43,49,53,56,81,85,87,88,90-92,95,97] electrochemical sensor studies will not be discussed further.

Prospects for piezoelectric quartz crystal sensors for gases and vapours have been assessed, including a range for determining aromatic vapours with sensors of macrocyclic materials [21]. Near to this area is recent interest by others in acoustic sensor technology in the roles of *quartz crystal microbalance methods (QCM)* as 'next step' techniques for studying unlabelled molecular interactions in real time, and of applications in chemistry and the biomedical areas.

Analytical chemistry is a significant subject

Analytical Chemistry is sufficiently important for various bodies to express concern about it from time to time [82]. A 1981 analysis of the activity and its conduct in the United Kingdom gave reasons for low emphasis and its consequences [78]. These have been rectified to some extent by the institution of new Chairs in the area, by additional research grants and studentships, and by a change in focus of interest by some researchers (as bees are attracted to a honeypot!).

Analytical Chemistry has considerable significance in its impact on our lives, in health and food analysis, and in servicing our democracy, economy, and environment. It has stimulating challenges in terms of *'fit for purpose'* data for establishing the constitution and nature of materials and in exercising judgement and interpretation.

Analytical Chemistry is important enough to be linked to Nobel prizes, fairly recently by the 1993 chemistry half-prize to Kary B. Mullis *'for inventing the polymerase chain reaction method'*.

Preparing for analytical chemistry

The subject and pursuit of Analytical Chemistry calls for appropriate education and experience for developing its skills. Here, there are similarities as to how Professor Casassas and I were stimulated to become chemists. Also, there are some common aspects to our later lives, as may be deduced by comparing statements here with the above-cited biography [1].

My fascination for chemistry was kindled and set alight by the skilled demonstration experiments of Henry J Griffiths at Llandovery in rural Wales [89,93]. The fascination and a university course interrupted by war service in blood transfusion at medical units – mostly in India, led to a BSc in chemistry and mathematics, with honours in chemistry.

The Cardiff BSc course, more by accident than design, had links to modern Analytical Chemistry. There was classical reaction analytical chemistry and various underpinning fundamentals, including Aston's *mass spectrograph* (mentioned in the 1922 Nobel Prize in Chemistry); the quantum theory and wave mechanics were there – leading to Raman and other spectroscopies. Incidentally, C.V. Raman received the 1930 Nobel Prize in Physics *'for his work on the scattering of light and for the discovery of the effect named after him'*. Papers in analytical chemistry now often relate to Raman spectroscopy, e.g., there were five papers in the February 2002 issue of *The Analyst* by UK and Australian authors on various Raman applications. These papers were linked respectively to Romano-British wall-painting fragments [20], detection of amphetamine sulfate [24], monitoring an esterification reaction [38], cinnabar from Herod's palace [26], and contaminants on polymer coated beverage cans [35].

Electrochemical analysis came via Faraday and Nernst, and we had polarography – a decade before Heyrovsky's 1959 Nobel Prize *'for his discovery and development of the polarographic methods of analysis'*. There were gems, like the urease-catalysed hydrolysis of urea and other enzyme reactions – the basis of future chemical (and bio-!) sensors.

Microscopy we had as an analytical tool for sugar osazones. And there were RA Zsigmondy's ultramicroscope and T Svedberg's electron microscope, recognised by Nobel Prizes in Chemistry, respectively in 1925 and 1926. In 1986 the electron microscope came up again with half a Physics Nobel Prize to E Ruska *'for fundamental work in electron optics, and for the design of the first electron microscope'*!

Chromatography, a mainstay with spectroscopy of today's instrumental analysis, was yet to cross the divide from biochemistry into chemistry curricula, but adsorption chromatography, as *'adsorption analysis'*, and *'electrophoresis for studies concerning serum proteins'*, brought the 1948 Nobel Prize in Chemistry to AWK Tiselius.

'The invention of partition chromatography' by AJP Martin and RLM Synge gave them the Chemistry Nobel Prize of 1952, the year of the Physics Prize to F Bloch and EM Purcell for *'developments of new methods for nuclear magnetic precision measurements'*. 1952 was also the year of the description of gas-liquid chromatography, by which the challenges of petroleum analysis were greatly eased. It led to derivatisation for volatilising analytes. Then in the late 1960s HPLC (high performance/pressure liquid chromatography) came to meet biomedical challenges, and for which the linking of chromatography to mass spectrometry has been most fruitful for analyses that were previously just pipe dreams.

Analytical data for decision making

Sound analytical data are of the utmost importance, as they are used for making decisions. But *Valid Analytical Measurement*, was a priority long before today's VAM activities. Over fifty years ago we had monthly inter-laboratory checks between a company's central and branch laboratories (today's *'proficiency tests'*). A salutory experience occurred when the data of one laboratory were out of line – due to supplied mis-calibrated pipettes.

Valid data demand vigilance, so that calibrations and intralaboratory testing and standards have special roles. On reference standards, there are those of Risdale, NIST, BCR, LGC, etc. Pure electrolytic silver was the ultimate standard in an ICI scheme, published in *The Analyst* of November 1950 (Volume 75, page 577), for standardising volumetric solutions. The scheme included primary and secondary standards.

Participation in setting a BCR standard of Bovine Skim Milk Powder [3] was a stimulating experience, as was producing recommendations on ISEs [48] which became a basis for drafting *Recommendations for Nomenclature on Ion-selective Electrodes* for the International Union of Pure and Applied Chemistry (IUPAC) [27].

The RSC book on *'Referring to the Facts: Application of Reference Materials in Analytical Chemistry'* by VAM authors [9] has good guidance on the correct use of certified reference materials (CRMs). Of course, CRMs are as much for instrument calibrations as they are for more routine use. The book discusses CRM production, and many other intricacies relating to them.

Without entering into detail, statistical tests can evaluate the quality of analytical data. As data need to be precise, accurate and unbiased, it is usual to express tolerances in terms of confidence limits for precision and accuracy. There is also *'uncertainty'*, a peculiar term for *'reliability'* [99].

Of course, analytical chemists need to adopt a formalism in terms of harmonisation of procedures and practices for analytical method development and validation. The new Burgess text [13] of the RSC's Analytical Methods Committee of its AD is a timely *'best practice approach'*.

Neither sampling nor statistical quality are in most undergraduate courses, but I set both in curricula of the University of Wales at Cardiff and at Swansea. Related is simplex optimisation, which with statistics and chemometrics [37, 64-66] are creeping into curricula. Simplex [100], aims for the best response from a system for desired criteria. In simplex optimisations of enzyme electrodes for glucose [10], we used *'modified'* and *'super modified simplex'*, and we also devised an improved *'additional modified simplex'* [11,12].

Analytical data are central to information for decision making. They must not be compromised, and tolerances must not be exploted to the detriment of product quality.

Sampling and method development can be tricky. The variety of 'Universes' or 'Lots', i.e., of material in bulk, *or* in motion, *or* in antibiotic ampoules, etc. offer problems, but few can be more challenging than one given by a medical colleague on determining iron loss by women during the mentrual cycle [22]. Integral to this was developing the analytical method of determining the iron with thioglycollic acid, but after checking the ashing stage by thermogravimetry [74]. The challenge led to 110 data sets for menstrual iron loss – from a cohort of 44 women. The data were used to assess the association between such iron loss and the level of circulating haemoglobin [22].

Evolution of analytical chemistry

Much of the impetus for the evolution of analytical chemistry in the second half of the 20th Century came from IUPAC's *Congress on Analytical Chemistry* of 1952 held in Oxford, and fully reported in *The Analyst* at the end of that year. There were eight sections, and the papers bridged theoretical principles and analytical roles of solvent extraction, square wave polarography, equivalence point determination (Gran's plots), chelating ligands, infrared spectroscopy, gas-liquid chromatography, ion-exchange, and statistics.

On entering teaching in 1953 I brought instrumental analysis into BSc and other curricula. Semi-micro and micro methods went into inorganic and organic analysis. [Pregl's Nobel Prize *'for his invention of the method of micro-analysis of organic substances'* was in 1923, but it took the 1940s Royal Institute of Chemistry lectures of Ronald Belcher for micro-analysis to move forward.] Also, I introduced project work – well before it became fashionable. Projects freshen teaching, and they are now stipulated by the UK's Quality Assessment Authority (QAA).

The projects I set in the late 1950s and 1960s on X-ray determination of crystal stucture, building a gas chromatograph [68], separations by electrophoresis [19], building an atomic absorption spectrometer, flame photometry of calcium in saliva [34], and interferences in reaction analytical chemistry [70,71] were challenging to students. The scope is unlimited for interferences, especially by using various and different kinds of unwelcome contamination.

Does crystallography relate to analytical chemistry?. The 1962 and 1964 Chemistry Nobel Prizes, respectively to Max Perutz and John Kendrew for the X-ray *'structure of globular proteins'* (heamoglobin), and to Dorothy Hodgkin for *'determinations by X-ray techniques of the structures of biochemical substances'* are certainly relevant, by information on *What is its profile and shape?* and of *Where is it?* for individual atoms. The information may even go as far as *What is it?* and to *How much?*.

Max Perutz who died on 06 February 2002, had trained as an inorganic chemist in Vienna. According to an Obituary he saw himself as «*a chemist studying a biological problem in a physics department*». But, as *'everything in the world is chemistry'*, crystallography is near to being analytical – with physics tools. Analytical Chemistry is multidisciplinary in the variety of tools and agencies (heat, diffraction, spectroscopy, electricity, microscopy, computing, etc) used in the pursuit of *Where is it?, What is it?*, and *How much is there?*, etc.

Food analysis is wide-ranging and encompasses many measurements during the lifespan of a product. It is rich in project lines. We used the effect of metal ions on the visible spectra of food dyes [32], chloride ISE studies on vegetables, fruits, juices and oils [52,54]; and fluoride in coffee and tea [60].

Case studies were linked to matters like lead pollution of road verges, and to interdisciplinary *'group'* projects on water quality involving sampling, bacteriological assessments, and measurements of BOD, COD, pH, hardness and other parameters. These express other proposals of today's currency for undergraduate case studies.

A good class project turned out to be the Andrews' titration of iodide [I(-1)] with iodate [I(+5)] in concentrated hydrochloric acid. This occurs in two stages, iodide being oxidized first to iodine [I(0)] followed by iodine oxidation to cationic iodine [I(+1), i.e., ICl_2^-] [44,45]. A Chemical Education panel in the USA selected our exercise [44] for a class experiments book [40]. Such exercises and similar are adaptable to *'proficiency testing'*, and to the concept of *'Z-scores'* as featured in some of the LGC's activities for the VAM Programme of the UK Government's Department of Trade and Industry.

Course/curricula development

Analytical Chemistry was promoted by the facility of *Higher National Certificate Endorsements*. I devised the first of these for Analytical Chemistry in 1957 at the then SE Essex Technical College (now University of East London) [80]. In the 1960s they were adapted to LRIC (*Licentiate of the Royal Institute of Chemistry*), and the contents also went into undergraduate curricula at the then burgeoning technological universities and polytechnics, all of which are now universities.

Several of us contributed tried class experiments to workshops and exhibitions of scientific societies in Sheffield and London in the 1960s to 1980s. Other initiatives were the *'Why teach a topic?'*, *'The teaching of a topic'*, and *'Training requirements for a topic'* series of the Education and Training Group of the RSC's AD [77,101]. These are consistent with the current RSC initiative of *Workshops for New Lecturers*. An extension is the teaching of chemical principles from a base of Analytical Chemistry, e.g., by and in the teaching of chromatography and electrophoresis [98].

The 1960s saw MSc type courses in Analytical Chemistry introduced in the UK, some with studentships from the Department of Scientific and Industrial Research (DSIR) – now EPSRC. We pioneered an early version in 1962, but as the number of entrants was small, we changed priorities and strengthened analytical chemistry in undergraduate curricula instead – both in chemistry, and in areas, like biology, medicine, pharmacy and instrument engineering.

Practical work and projects complement lectures. Students need experience in searching for and in applying information. Library projects are also helpful, such as:

- What method can be used for determining atmospheric fluorine near an aluminium plant?
- A plastics sheeting contains magnesium and zinc, each in the 10 to 100 mg kg^{-1} range. How can the metals be quantitatively determined?
- How can the level of catechol in cigarette smoke be determined?
- How can a molecular recognition site be introduced into a matrix for molecular imprinting?

We sought reports (~250 words) to the following guidelines:

- Statement of the problem.
- Proposed solution (with attention to instruments, limitations, and precision).
- Practical significance of the solution / method.
- Most recent significant reference found.
- Most helpful reference(s).
- Most fruitful search approach.

Communication skills are promoted by student lecturettes (5 to 10 minutes), e.g., on *'Why do analytical methods have to be validated?'*; *'What is 'speciation'?'*; *'Effective sampling'*; *'Dimethyl-glyoxime as a reagent'*; *'Operating principles of an analytical instrument'*; and *'Determination of Vitamin A'*. The possiblities are many. The principle extends to a «*One* *minute please*» type of *'game'* exercise, especially for students of limited attention span. The object was for participants to speak for one minute on a topic, such as *'mercury drops'*, *'dithizone'*, *'ion-exchange'*, and, *'gravimetry'* written on a piece of paper, which was picked from a hat or a box.

Short courses and symposia

It is important to keep up to date and to learn new and modern skills. Short courses and symposia/conferences are popular for these purposes. They are regular features of academic institutions, learned societes and other bodies. They must be well planned, and they are good for testing organizational skills. An impetus for Short Courses in Analytical Chemistry was the instrumentation and methodological advances of the 1950s – and today's initiatives cater for the newly coined *'Continuing Professional Development'*.

Also in the United Kingdom is an *Open Learning Scheme*, known as «*Analytical Chemistry by Open Learning: ACOL*». Set up in the early 1980s by Government funding [101], ACOL gives knowledge of the principles, practice and instrumentation of analytical chemistry. It is based on textbooks for 'distance learning', and supported by workshops (for practical work, etc.) at designated centres [103].

Short courses at Cardiff during the 1960s and early 1970s, often with practical work, were of the evening type, e.g., *Sampling and Statistics, Spectroscopy*, *Separations*, *Radiochemistry* and *Electroanalysis*. The thirst for knowledge, in order to catch up with the then latest developments had participants travelling by up to 100 km or more for the ~3-hour evening sessions.

The several more ambitious type of Cardiff-based international type symposia of 2 to 5 days duration, usually in association with learned societies, attracted up to ~250 participants for *Instrumentation in the Chemical Industry (1962), Thin-layer Chromatography* (1965), *Atomic Absorption Spectroscopy* (1967), *ISEs* (1969), the IUPAC-sponsored *ISEs* (1973) [75], *Electroanalysis and Sensors* (1981, 1983 and 1987) [79,83,84], *Polymerase Chain Reaction* (1990), and *Gas and Headspace Vapour Analysers* (1991).

Symposia (with attendances of ~100 or more) for the RSC's AD in London include those on *Free Radicals* (1990), *Fungicides, Herbicides and Insecticides (1992)* and *Alternatives to Chemical Solvents Restricted by the Montreal Protocol* (1995), with the next in the series being on *Recovery Factors: Aspects of Analytical Measurement* (2003). There have been many other events, some continuing, both of the AD's Programmes Committee and of other organisations.

Public promotion of analytical chemistry

It has been a refreshing experience to promote Analytical Chemistry among lay people at evening lecture events (*'Our Daily Bread'* was one theme, but it is easy to dream up others, such as *'Clean Water'* or *'The Clothes we Wear'*), and also to young people, such as by *'Schools' Lectures'*. The AD's *'Schools' Lectures'* which ran from 1983 until 1997 was a significant 'national' UK and Republic of Ireland initiative by the RSC's Analytical Division. Sponsored by the RSC's Analytical Chemistry Trust Fund [101], they are now localised – to lessen the burden on lecturers. There is no longer a named 'national' lecturer for the year as previously, as lecturers are now by *ad hoc* nominations from participating local centres who arrange audiences of young people of >15 years old. Examples of lectures titles are *'Trace Analysis', 'Potential Sense in Analytical Chemistry'* (the author's theme), *'Chemical Analysis of Food', 'Pink Panthers and Ozone Holes or Colour and Light in Analytical Chemistry'* and *'Mass Spectrometry – Molecules in Flight'*.

Helping with the erstwhile AD's Education and Training Group / Committee initiatives on promotional brochures, posters and videos (*'Chemical Detectives'* and *'ANALYSTS – Analytical Science in Action'*, and with Schools' Analyst Competitions was an informative privilege [101]. The UK National Schools Analyst Competitions are based on teams of three ~16 year old pupils carrying out practical tasks [86,101]. The 'Final' stage has up to 16 teams, each team having been winners of preliminary competitions at various UK local centres. An example of a practical task is the characterisation of a coloured polymer compound. This was by infra-red spectroscopy to identify the plasticiser, thin-layer chromatography to identify the dye, and gas chromatography and spectrophotometry to quantify the plasticiser and dye, respectively.

Research and consultancy

It can be regarded as a special favour and privilege to have had the opportunity to contribute to the UK's ~6 to 7% share of the world's annual research in Analytical Chemistry; and as a result to have lectured, advised, consulted, and to have had merit in the citation index (top centile). Professor Casassas contributed similarly, and he witnessed an impressive input by Catalonia into Spain's phenomenal increase to knowledege in Analytical Chemistry since the 1970s, and especially in the last 20 years.

Lecturing tours plus visits for cooperation, advising and consulting at centres in the UK and in various countries (30 in all, with six times to Catalonia), including the periods as Japan Society for the Promotion of Science (JSPS) Visiting Professor in Japan (1985), as Distinguished Visiting Fellow of Australia's La Trobe University (1989), as Foreign Expert in China (1983, 1985), and various other privileges have been rewarding and much appreciated.

Research was commenced during spare-time (there wasn't much!) in my first teaching post – for an MSc under HB Watson, a doyen of physical-organic chemistry and author of *'Modern Theories of Organic Chemistry'* – the first textbook in its field. There was much teaching, covering all branches chemistry, ~6000 h in the first 8 years, out of ~15 000 h over 40 years. This broad base was an enrichment of the know-how and experience for conducting and directing research on analytical chemistry, as was also felt by Professor Casassas. Direction of research was geared to giving a wealth of broad experience to researchers by planning projects in terms of *'aims and objectives, background, projected programme, research training, and expectations'*.

Progress of graduate co-workers was monitored by:

- **Weekly seminars** (usually during 09.00 to 10.00 h on Mondays): A 20 minute talk by a co-worker, followed by discussion prior to a summing-up conclusion by a Supervisor.
- **Monthly written reports** (~2 pages) from each coworker, set on achievements of the previous month and a work programme for the next month.
- **Periodic progress meetings with Sponsors**, where appropriate. These hinged on the co-worker's oral presentation, which was set against a pre-submitted written report. This experience was consistent with 'consultancy' exercises that might be faced in later life.
- According to progress, co-workers would **draft papers** for suggested amendment and approval by supervisors and research director prior to submission for publication.
- **Thesis plan** was required at a project's half-way stage, and a start made on the introductory literature review. Periodic reviews would ensue.
- **Termination of Projects** was aimed for the end of the projected 3-year term, including reports to sponsors and thesis writing. This emphasised the need for promptness in meeting deadlines.
- **Performance**: Discounting withdrawls within 6 months, >95% of graduate researchers produced successful theses– all except one were produced within 6 months of the end of the project term.

The following outlines our research, other than chemical sensors, which have been mentioned above.

Separations by Electrophoresis

The research on electrophoretic separations, conducted between 1958 and the early 1970s focused on improving the method, with advances as follows as covered in our book [50] and elsewhere [102]:

- **roles of support matrices** (thin-layer types based on alumina, kieselguhr and silica gel, cellulose acetate, and gels of agar, polyacrylamide and polystyrene);
- **explanations of electrophoretic migrations**: steric, polar and other characteristics
- **scope in applications areas** (food colours, carbohydrates, blood serum and oil additives);
- **deleterious thermal effects** (quantifying heating and refrigeration of supports); and
- **refrigerated air cooling** with a mini-electrophoresis tank and low-sorptive supports on microscope slides, for rapid separations of improved resolution.

A huge challenge was ***'zone migration'*** of soluble carbohydrate-borate complexes – leading to haphazard visualization. This happened with low sorption supports – kieselguhr G and cellulose acetate. The effect (shown with dyes), was by separated zones 'slipping over' one another. The solution was to promptly '*freeze-dry*' the electropherograms. [17,18].

Non-sorptive supports, like cellulose acetate and kieselguhr G, gave complex zones for blood serum well beyond the anticipated for both normal and kidney disease screening patterns [72]. In retrospect, we were close to current DNA profiling and polymerase chain reaction methods, but lack of resources led to discontinuance of this project in the late 1960s.

Electrophoresis has expanded and some practical difficulties circumvented since our work of the late 1950s to early 1970s on separative electrophoresis, as reviewed in our book of 1975 [50]. For example, *capillary electrophoresis* (CE) with buffer-filled capillaries has developed apace [4], and is now routine in many hospitals. Also, CE has dramatically increased throughput for DNA profiling, and it has wider uses in areas similar to those of HPLC.

Ion-exchange In Non-aqueous and Mixed Solvents

Interests in ion-exchange stemmed from seeking a teaching exercise on non-aqueous anion-exchange, which led on to rewarding research. Thus, nitro-phenols of similar pK_a were separated by gradient elution anion-exchange from methanol solutions buffered with triethylamine –acetic acid mixtures [67,73]. Buffering with diethylamine– acetic acid gave separations of high pK_a cresols and xylenols.

Anion-exchange with amine-acetic acid buffers and infrared monitoring, yielded aliphatic esters, aromatic esters and phenols from methanolic extracts of peat [69]. This gave me a good industrial consultancy, and many years of research support on industrially relevant projects. [*For financial support I am indebted to the petroleum, surfactants, instrumentation, environmental, energy and biomedical industries, and also to charities and public bodies – including those of health-care.*]

Our other ion-exchange studies showed:

- salt-form resins to be the more resistant to solvent and temperature effects, [39];
- organic and mixed aqueous-organic solvent media to yield interesting separations and selectivity reversals of inorganic ions [33,46,47];
- a linear relation for log[selectivity coefficient] (between cations and hydrogen ions) vs. reciprocal of dielectric constant for methanol-water, but did not reach to methanol-rich media [33]; and
- concentration isotherm studies, by batch anion-exchange resin experiments, showed prospects for selecting conditions for the ion-exchange of anionic surfactants [61].

Role of Polyurethane Foams

Foamed cellular materials seemed attractive for extractions and separations by their favourable aerodynamic properties [55,57]. Most extractions and separations have been on commercially available polyether type polyurethane foams [55,57].

We established conditions for extractions of metals on treated foams [36,57,62,63], and explored *'Chromofoams'* of preloaded chromogenic chelates, like dithizone [62] – concentrations were linked to lengths of coloured bands, i.e., red for iron(III) and blue for cobalt (II).

Polarography/Voltammetry

My research interests in polarography/voltammetry arose from a 1977 brief to review developments in electroanalytical chemistry [76]. This complemented a 1961 experience of lecturing on polarography at an *inspection* by the then Ministry of Education of the UK Government which fortunately resulted in a confidence boosting «*glowing report*». Many will be aware that much external appraising, inspection, and prescription by the QAA has by now befallen UK universities.

Our researches on polarography include

- **polarography of imidazoles**, which are used in the pilocarpine iontophoresis generation of sweat for chloride and sodium determination in diagnostic screening for cystic fibrosis [15];
- **voltammetry** (cyclic voltammetry, differential pulse polarography, d.c. polarography, and linear sweep voltammetry) of copper O,O'– dialkyl-phosphoro-dithioates (dialkylthio-phosphates – DDP oil additives), i.e., CuDDPs [29-31]; and
- a step away, a **four-electrode potentiostatic study** of facilitated ion-transfer of alkali metal cations from water to a nitrobenzene phase containing various crown ethers [28].

Miscellaneous challenges

Analytical chemistry is exciting by the immense variety of miscellaneous challenges, and problems never cease. Not least among these have been controls of the *Montreal Protocol of Substances that Deplete the Ozone Layer* on the production and supply of chlorofluorohydrocarbons (CFCs) and carbon tetrachloride – as they have necessitated replacements or modifications to various analytical procedures [96].

There are also the unexpected problems – the lifeblood of many consulting practices. Examples we have encountered include:

- severe burns by a *'home bleach'* compounder when mixing components delivered by a supplier (a container 'mistakenly' contained *'battery'* sulfuric acid!);
- quantification of cadmium in the urine of patients *poisoned* by welding cadmium-plated bolts in an enclosed atmosphere;
- sulfate content of soil samples and sulfur content of steels (there are BSI methods);
- hydrocarbon (kerosene) contamination of a consignment of distilled / deionised water; and
- the already cited mentrual iron loss case.

Environmental, and vehicle and driving legislation have led to many new analytical methods and challenges, as have Green and health issues. Many of these are *chemistry in action*, as happens with effluent streams from disued and old mine-workings, and from old and distressed industrial sites. Such seepages are of the kind that can entice those with speciation skills, as exist in Barcelona.

Price differences between petrol and kerosene create temptations to adulterate – this is rampant in Malaysia, but national borders of the EU also lead to temptations, as indeed do the colouring of diesel for differentiating between different excise duty rates. But they all offer challenges and livelihood to analytical chemists. The above matter of distinguishing between petrol and kerosene led us to a propan-1-ol based *'phase-titration'* determination of kerosene adulteration in petrol [5,6]; and on to an alternative approach with platinum-based fuel cells [7,8].

Conclusion

These are just a some of the highlights and perceptions of Analytical Chemistry as derived from my experiences of its challenges and exciting issues over more than half a century. The advances, by the march of technology through research, application and experience, have been phenomenal. These have imposed great changes in the scope and concepts of Analytical Chemistry, as indeed has happened for most human activities and human endeavour. By now, for Analytical Chemistry:

- even the laboratory is frequently dispensed with, as in process monitoring, analysis in space, road-side breath alcohol measurement, the use of ISEs for sweat chloride analysis, by enzyme electrodes for monitoring blood glucose, and by smoke detection devices in households; and
- new diseases, new materials, new processes, sophistications in crime, lead– and sulfur-free fuels, and other features create new challenges.

Even with ~20 Chairs in the United Kingdom devoted to research in Analytical Chemistry the future remains as exciting as in the rest of the world. But, there is the widespread matter of analytical chemists all too often having doubts about the value of their work. However, an 'editorial' of the journal, *Analytical Chemistry* [104] emphasised that analytical chemists have many reasons to be proud about what they do, as «*Analytical methods and measurement science are of fundamental importance, and without them, chemistry would not be an exact science today*». Nor would there be benefits in quality of life and its activities.

Bluntly, Zenobi, author of the editorial, says «***Chin up, chest out!***».

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About the author

J.D.R. Thomas, born in 1926 at Gwynfe in rural South Wales, there was local schooling until 1943 followed by university, with a break for army service in the Royal Army Medical Corps, and graduation from the University of Wales, Cardiff in 1950. Then came industrial, professional and teaching appointments with some research until 1967, followed by university teaching and research (7 books, >300 articles) during 1967-93, and Emeritus Professor of Chemistry and Applied Chemistry of the University of Wales. Other details are available in The International Who's Who *(Europa Publications) and in* People of Today *(Debretts Peerage). Awards include RSC Electroanalytical Chemistry Medal 1981, J. Heyrovsky Centenary Medal 1990, L.S. Theobald Lecture 2001, and various Honour appointments: China 1983/85, Japan (JSPS) 1985, Australia 1989, Hong Kong, 1991–, Romania 1996– etc.*

Learned Society Appointments: Council Member of the Royal Society of Chemistry during 1977-80 and 1990-to date, President of the RSC's Analytical Division during 1990-92, and Honorary Editor of AD NEWS (news-sheet of the Analytical Division) during 1994-to date. Public appointments: Member, Court of Governors of the University of Wales during 1989-present, and of its Council (1997-2001), and Council of the University of Wales College of Medicine during 1993-99.

After early work on kinetics and energy relations in chemistry, research has focused on analytical chemistry in terms of (i) potentiometric ion-selective electrodes (ISEs), which led to them becoming an essential component of the almost universal blood electrolyte measuring systems; (ii) amperometric type enzyme and metal oxide catalyst membrane electrodes for sugars, hydrogen peroxide and food components; (iii) piezoelectric quartz crystal sensors for gases and vapours; (iv) separations (electrophoresis, polyurethane foam extractions and ion-exchange); and (v) miscellaneous analytical determinations.

Some highlights and perceptions of analytical chemistry: a post-script

J. D. R. Thomas

The presence of Senyora Casassas, widow of Professor Enric Cassasas, and daughter, at the *Enric Casassas Memorial Meeting* on 7 March 2002 lent an appropriate aura to the event. This was especially the case for recollections of stages in the early life of Professor Casassas. The presence also promoted appreciation of references made to Professor Casassas's research contributions, and for the way they had impacted on the development of analytical chemistry and on the researches of others. Special reference was made to those contributions linked to the cathodic displacements by ligands, such as dihydroxyazobenzene, when L. Eek was principal coworker of a long series of papers covering spectrophotometric and polarographic studies [1-7]. There was an exciting stir in the audience when it was realised that, although Dr. Eek himself had by now retired, there was a niece present in the gathering (i.e., a daughter of his wife's brother), and who is a member of staff at the Polytechnic University of Catalonia.

Professor Casassas would have been delighted that his broad theme of studies in the area of complexation should continue. Indeed, this is to be seen in a paper in *The Analyst* of the month of the Memorial Symposium, i.e., on the complexation of Cd^{2+} by glutathione fragments studied by differential pulse polarography [8].

Finally, the author is grateful for the invitation to present the *Enric Casassas Memorial Lecture 2002*, and he also expresses sincere appreciation and thanks to all concerned with the associated care and hospitality which were to his benefit and comfort during the visit to the beautiful city of Barcelona.

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