The Kipp generator

Transforming chemistry teaching in the 1950s

Tom O'Donnell

Until the 1950s, University of Melbourne science lecture courses at first-year level were largely general and descriptive, with some content of physical chemistry (concerned with the application of the techniques and theories of physics to the study of chemical systems).[1] Under Professor Ernst Hartung (chair of chemistry from 1928 to 1953), first-year lectures were characterised by brilliant lecture-demonstrations. Organic chemistry (broadly speaking, the chemistry of materials that contain carbon atoms) was introduced in second year, with very formal physical chemistry but *no* inorganic chemistry (the study of salts, metals, substances made from single elements, and any other compounds that do not contain carbon bonded to hydrogen). Even at third-year level, physical and organic chemistry predominated over inorganic chemistry, which rated only a single topic, covered by about eight lectures.

At this time, the entire first-year *practical* course was based on analytical chemistry: quantitative (both volumetric and gravimetric) and qualitative (based on the sulphide separation scheme for studying and identifying cations and anions—species with negative and positive electrical charges respectively—in aqueous solution). Second-year 'prac' consisted of two terms of analytical chemistry and one term of organic chemistry. The organic component was larger at third-year level.

The sulphide separation scheme was based on the use of hydrogen sulphide gas (H_2S), which was generated each day by laboratory assistants in a large gas-holder on the roof of the Chemistry Building, and piped into the laboratories through fragile vulcanite pipes and valves. This was an extremely risky process, because H_2S is comparable in toxicity to, and even faster-acting than, hydrogen cyanide (HCN). Each of the 200-odd students in the first-year laboratory saturated their gram-scale test solutions in a 250 millilitre Erlenmeyer flask (a conical glass container also known as a titration flask). The department reeked continuously of foul-smelling H_2S (often dubbed 'rotten-egg gas'), as it was the essential reagent for their experiments in qualitative chemical analysis.

Early in 1950 in his PhD project, Tom O'Donnell was separating radio-isotopes on the semi-micro scale (0.1 gram or less). In 1951 he succeeded the great analyst Gustav Ampt in running the second- and third-year analytical courses. He realised that the introduction of semi-micro techniques, which were then unknown in Australia, could greatly increase the efficiency of conducting qualitative analysis. For example, separation of precipitates by centrifugation is far more rapid and effective than the paper filtration method that had been used in all laboratory work to date. Furthermore, there was potential for significant monetary savings on costs of chemicals used by students.

In 1952 Professor Hartung authorised construction (in the department's stores and workshops) of the necessary kits and items of apparatus for the introduction of semi-micro techniques to the third-year students. On visiting the laboratory, Hartung, himself a very skilled experimentalist, observed all of the 30-odd students saturating solutions in 10 millilitre test tubes with H_2S that flowed through six glass capillary taps from a single Kipp H_2S generator, fixed for safety to a wooden base-board.

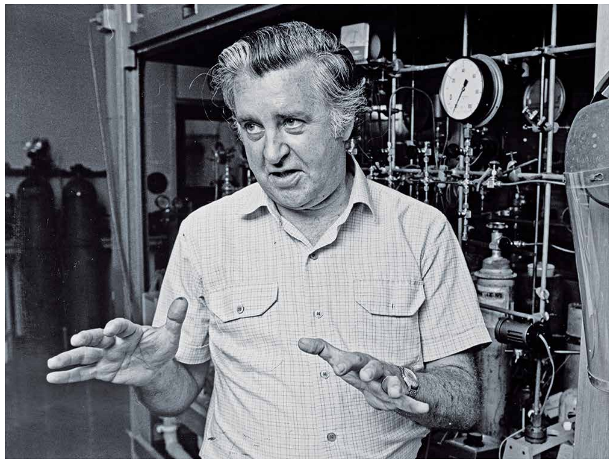
The Kipp generator (or Kipp's apparatus) for preparing small volumes of gases was the invention of a Dutch pharmacist in the 1840s. It consists of three main chambers: in the lowest chamber is placed a solid material, such as pellets of iron sulphide (FeS), and in the topmost chamber is placed a liquid, such as hydrochloric acid (HCl). When the

Kipp's apparatus for H_2S generation, used in the School of Chemistry teaching laboratories, 1950s, glass, approx. 63 × 30 × 20 cm. Gift of Professor Tom O'Donnell, 2009, School of Chemistry Collection, University of Melbourne. Photograph by Ben Kreunen, University of Melbourne.

HCl reaches the FeS, hydrogen sulphide gas (H_2S) is formed and rises to fill the middle chamber. The H_2S is under slight pressure from the column of HCl. Opening the tap in the middle chamber to draw off H_2S allows more acid to descend and generate further H_2S as required. The double bubbler at the very top of the apparatus minimises release into the atmosphere of any H_2S dissolved in the HCl.

Having seen the limited and safe dispensation of H_2S on the semi-micro scale, and noting the elegance of the techniques, Hartung immediately authorised construction of several hundred kits for the introduction of semi-micro qualitative analysis, firstly at second-year level and then first-year.

But demonstrating the small-scale and intricate manipulative techniques of semi-micro analysis to hundreds of undergraduates in a lecture theatre was difficult. So in 1954 Ron Brown (senior lecturer in general chemistry, later to become foundation professor of chemistry at Monash University), Robert Craig (a senior demonstrator in physical and inorganic chemistry) and Tom O'Donnell (now a lecturer) made a 35-minute film demonstrating these techniques.[2] Around this time, the newly appointed head of department,



Professor Tom O'Donnell, c. 1970s–80s, photograph by Norman Wodetski, black-and-white photographic print, 16.5 × 21.5 cm. 2003.0003.00137, University of Melbourne Media and Publication Services Office, University of Melbourne Archives.

Professor J.S. Anderson, turned over virtually all first-year teaching and practical courses to Brown and O'Donnell, who in their lectures developed the theoretical concepts from the practical courses and introduced some inorganic chemistry, while retaining much of the physical and organic chemistry. In 1955 Brown and O'Donnell collaborated on a textbook that added to and updated several of the exercises in quantitative and qualitative analysis and provided a much deeper theoretical background than had been a feature of earlier courses.[3]

The printed notes used by first-year students into the 1950s presented acid–base reactions in terms of the Arrhenius concept, which classifies a substance as an acid if it produces hydrogen ions H^+ or hydronium ions in water, and as a base if it produces hydroxide ions OH^- in water. This way of defining acids and bases works well for aqueous solutions. Oxidation-reduction in solution was represented by equations such as:

$$2FeO + O_2 \rightarrow Fe_2O_3$$

Qualitative analysis was presented largely as a puzzle, to identify cations and anions in given samples called 'unknowns'. Brown and O'Donnell introduced the Lowry-Bronsted concept of acids and bases and oxidation-reduction reactions in terms of electron transfer. They switched the emphasis in qualitative analysis from identifying components of 'unknown' mixtures to a consideration of competing ionic equilibria in the sulphide separation scheme and the accompanying reactions. The 1957 edition of their textbook added to the previously exclusively analytical program preparation of some inorganic compounds using semi-micro techniques, and introduced practical exercises in qualitative and preparative organic chemistry, also using semi-micro procedures.[4]

A case can be made that Professor Hartung's decision, after seeing bulk reticulation of H_2S replaced by Kipp generators, to make semi-micro techniques available in practical courses at all levels was the forerunner of major changes in the teaching of first-year chemistry at the University of Melbourne in the 1950s. We are fortunate that one of those early Kipp generators has been preserved in the School of Chemistry Collection.

Professor Tom O'Donnell (1923–2010) was an undergraduate in the School of Chemistry at the University of Melbourne in the 1940s, and completed an MSc and then a PhD in 1954. He was promoted to lecturer (1951), senior lecturer (1957), reader (1963) and finally professor of inorganic chemistry (1979–88). He spent time at Cambridge, Chicago, Toronto and Berkeley, published numerous scientific papers and several books on inorganic chemistry, and served as head of the School of Chemistry and on many important committees and boards.[5] Professor O'Donnell donated the Kipp generator to the university in 2009.

The School of Chemistry Collection comprises more than 300 items associated with the first century of teaching and research in chemistry at the University of Melbourne, from the 1850s to the 1960s. Selected items can be viewed as part of a series of rotating exhibitions on the ground floor of the Chemistry Building. Plans are underway to create a permanent museum to house and display the entire collection. For more information, or to search the collection catalogue, see http://museum.chemistry.unimelb.edu.au/.

1 This article is an edited version of a text written by the late Professor Tom O'Donnell in 2009. Many thanks to Professor Ken Ghiggino and Rob Ennis-Thomas of the School of Chemistry.
2 R.D. Brown, R.A. Craig and T.A. O'Donnell, *Semi-micro analysis*, 1955, 16 mm film. 51b, School of Chemistry Collection, University of Melbourne.
3 R.D. Brown and T.A. O'Donnell, *Manual of elementary practical chemistry*, 1st edn, Melbourne University Press, 1955.
4 R.D. Brown and T.A. O'Donnell, *Manual of elementary practical chemistry*, 2nd edn, Melbourne University Press, 1957.
5 Terry O'Donnell and Valda McRae, 'Master of dangerous chemicals: Thomas Aloysius O'Donnell, scientist, educator, 28-11-1923 – 21-2-2010', *Sydney Morning Herald*, 7 May 2010, www.smh.com.au/national/master-of-dangerous-chemicals-20100506-ugz3.html.