University College: A chemistry graduate student's experiences, 1954-1957.

I had obtained my second class London External BSc Chemistry Degree through part-time study at Acton Technical College (later to become Brunel University at Uxbridge) while working as an analytical chemist at Glaxo Laboratories at Greenford. For the record, 760 students took this exam in June 1954; there were 5 (five) firsts, 55 seconds and 168 pass degrees. Earlier in the summer of that year, I had had an interview with Prof. Ted Hughes (acting as deputy Chair to Sir Christopher Ingold), and he assured me, that provided I achieved a first or second class degree I would be welcome as a post-graduate student. My first Postgraduate Studentship was from Middlesex for £300 p.a. plus £25 for demonstrating. (It is worth an aside here; then, "demonstrating" involved responsibility for a group of six or seven students, an unthinkably small ratio for today's hordes at my home University in Ottawa). That County Award was followed for the second and third years by a London open postgraduate studentship, again for £300. (Note that the then major source of financial support were the DSIR (Department of Scientific and Industrial Research) studentships.

And so on 20th September 1954 I was ushered into lab C16 in the "old" Chemistry building, the Ramsey Laboratories. It (the lab) looked very like that shown in the Ramsey portrait, and could well have been where it illustrated. My supervisor was to be Allan Maccoll, who made me welcome and introduced me to my lab-mates, Ken Fish (A Harry Poole student, Raman spectroscopy) and Ron Barker, doing photochemistry with Allan.

Allan Maccoll was then a short, burly and slightly stout man, with trimmed, reddish curly hair. I soon discovered his legendry capacity for beer and that he chain-smoked. Allan had grown up in Australia (the Maccoll family had emigrated when Allan was a baby) and he was one of the bright, post-war contingent of Australian academics that came back to the "Old Country" to gain experience and further their careers; especially if they went home again, full of up-to-date scientific knowledge, and thus eligible for plum positions in the expanding Australian academe. Allan visited Canada several times, the first by sea; on arrival in Montreal he noticed the big advertising sign "Drink Canada Dry", and so he obeyed this welcoming invitation to the best of his considerable ability. He became a life-long friend and mentor and I never found him unwilling to give constructive criticism, support and advice. He obviously loved what he did and it rubbed off on his students. His gowned portrait (in oils by Clive Hodgson) makes him appear somewhat curmudgeonly, as far from the truth as it is possible to get!

Allan was a patient, sympathetic research supervisor to me and his kind assistance eroded away all my (unexpressed, but perhaps visible) insecurities at having come to UCL by the inelegant route of industry, with only part-time study. I quickly found too, that my fellow students were not streets ahead of me as I had feared, but that they lacked many of the practical skills that I had learned at Glaxo and now therefore took for granted. Thus, settling in was much easier than I had expected it to be. My contemporaries were friendly and never made me feel an "outsider". Indeed many of my lifelong enduring friendships came from that period. One other aspect of UCL was that probably due to its leavening of colonial staff, it was a *classless* society.

At UCL were Allan, Ron Nyholm, Tom Dunn, Peter Delamare (New Zealand) and David Craig all from down-under and fine scientists. Ron stayed in the UK, became Professor of Inorganic Chemistry and was knighted for his services to education. Alas, he died unexpectedly in a road accident when returning from Cambridge some time in 1971. He worked intensely, at top speed all the time, and he may well have fallen asleep at the wheel, as was reported then.

David Craig, a theoretician, returned to Canberra, to the Australian National University, where he was a founding member of their excellent Research School of Chemistry. When I last saw him, he was well into his eighties and he continued to teach from time to time in the Chemistry Department and still played tennis. (He died in 2015, at the age of 95; see his Royal Society biography for his achievements). As described elsewhere I spent several happy Sabbaticals at ANU with the theoretician, Leo Radom.

During my three years at UCL I commuted almost every day by bicycle from my home in Kenton, about an eleven mile ride each way. The traffic was heavy, but a wide variety of possible alternative routes made it bearable, and better (and faster) than the train journey. I had only one accident in those years, when a Spanish onion seller opened his van door without looking.

At this time the foundations of mechanistic organic chemistry were still being worked out, chiefly in the UK and USA, and a major contribution to the detailed understanding as to how organic reactions proceed, came from the measurement of reaction rates, mostly in solution and only relatively recently had gas phase studies begun.

My research topic was to be the kinetics of the pyrolysis of alkyl iodides. The mechanisms of the alkyl chloride and bromide pyrolyses were well understood by then, the former as uni-molecular eliminations of HCl and the latter as mixed free radical and molecular decompositions producing the alkene and hydrogen bromide. The iodides presented a new problem, because the products were alkene, alkane and iodine in equimolar proportions. Thus the HI produced in the initial pyrolysis was proposed to react rapidly and completely with the alkyl iodide to create the alkane. My first task was then to study the kinetics of this secondary process, using 2-iodopropane plus HI as my example.

It is worth noting that the final outcome of this work was the excellent correlation between the *heterolytic* carbon-halogen bond strengths and the pyrolysis activation energies, showing that the transition states were essentially polar in nature: alkyl cation, halogen anion. (Many of the bond energies came from the direct measurement of free radical ionization energies, research pioneered by Fred Lossing at the National Research Council in Ottawa, and who became a friend and collaborator for many years).

The departmental support staff provided memorable characters. The resident custodian was Mr Butcher, (aka "Butch") ex-army, at least 6' 4" tall and perhaps 250 lbs, with a voice and manner to match. He was actually very good-humoured except of course, when his authority was challenged. It was rumoured that he even spoke sternly to Sir Christopher on occasion. I well recall persuading him to allow me to leave my bicycle each day in the passage near the NW door. Since he lived in the building (a flat on the top floor) he was 24-hour custodian as well, patrolling the laboratories each night, and it was from him that one had to obtaining a chit, (signed by Bill Woodings) to be posted on ones' lab door, showing that an overnight experiment was in progress.

Bill Woodings was the chief lab steward and was responsible for all laboratory management. Joe Reynolds ran the instrument store; a quiet spoken man with a moustache, he was always available to discuss what equipment he had that was suitable for one's project and to make sure that its operation was fully understood. Mr Gill was in charge of the glassware store and general laboratory equipment; he wrote down every single item that you drew in two great ledgers, one for his records and one for you, which he presented to you when it became time for you to leave the department. Thus he could show you what he expected to be returned, or at least accounted for. After you had satisfied this requirement, a note was sent to Prof. Hughes to say that your thesis could be submitted. In my time (almost) everyone obeyed this.

The glassblower, Jim Frost, was a major asset and a great personality, short, bespectacled, a non-stop talker; he ran an excellent course in glassblowing for the graduate students, once a year, just before the Christmas break. Glassblowing was a vital exercise for me, with a complex vacuum line to build and maintain. His assistants in the glass shop were Eddy Nutt and Monique, always cheerfully ready to help out, especially during an emergency. Monique was a dwarf and so a special bench was constructed for her to work securely.

Jim also gave an open lecture-demonstration once each year in the main auditorium, in which he demonstrated his skills. The highlight was the production of a quartz fibre, thinner than a cotton thread but much stronger. This was achieved as follows: a quartz rod was mounted like an arrow in a steel bow and heated strongly at its centre, just behind the (wire) bow string with a coal-gas/O_2 flame. When the rod reached incandescence the "arrow" was released and the glowing rod flew across the lecture theatre trailing behind it an almost invisible quartz fibre. The latter was extraordinarily strong, surviving its flight intact, to the audience's surprise and amazement.

The old Ramsey building was dark and smelled faintly of the accumulation of chemicals that had permeated the building over many decades. The windows were cleaned about once a year and because this was before the "Clean Air" Act of 1956, they remained clean for only a few short days. Note that there was a severe "smog" in 1957, repeating the experience of the "killer smogs" of 1952. The undergraduate laboratory floors were made of well-worn wooden planks and had suffered many a spill from the benches above. In the organic and inorganic teaching labs, the drains from the benches emptied into ceramic U-shaped runnels under removable floorboards. If a significant quantity of diethyl ether had gone down the drains, a not uncommon event, an interesting but highly frowned-upon trick, was to ignite the ether in a sink and watch the (actually cool---pour a little into your hand and ignite it) flame rush along under the floor, appearing where gaps in the old boards permitted. If one lifted the boards in these labs, droplets of accumulated mercury could likely be seen. By todays exacting (sometimes neurotic) standards the labs would be declared unsafe, but we all seemed to have survived unscathed. It is useful to record here an (in my opinion “unpredictable”) accident.

One day a notice was circulated to say that a strike at British Oxygen meant that liquid nitrogen (vital for our vacuum line traps) would not be available for some time; however a source of liquid air would replace the usual refrigerant. The coolant was contained in big conical cans, mounted so as that the refrigerant could be tipped out into the receiving Dewar flask. Ron Barker took his flasks to the basement to fill them for the day’s work. The flasks, capacity ca 1litre, were contained in wooden boxes filled with vermiculite as insulator. When Ron tipped the liquid air into the first flask there was a violent explosion, destroying the flask but fortunately missing Ron. We quickly discovered the reason for the violent reaction, our lab Dewars had over a long period, collected a film of condensed organic vapours from the lab. Unexpected, but a lesson learned.

Because my research required a complex all-glass vacuum line, Jim’s glass-blowing course was vital. It began with simple joints using soda glass; items made in this material required annealing immediately after completion, otherwise they invariably cracked due to the strains inherent in the areas of different thickness that the unskilled work produced. One heated the newly completed product in a smoky gas flame until it was covered in a fine sooty deposit; only then could it be safely laid on the ceramic bench top to cool. After this difficult introduction we progressed to Pyrex glass which did not require annealing. At the end of the course, our final exercise was to attempt a single-surface condenser, involving two internal seals. Thus briefly trained, I built most of my vacuum line myself, after the basic main-line had been set up by Eddy. I also made a complex spoon gauge, as described below.

A spoon gauge is made as follows:- a Pyrex glass tube, diameter ca 5-7mms, sealed at one end, is heated in an O_2/gas flame until the glass softens. By gentle pulling and careful blowing, a thin-walled oblate bulb can be produced in the hot zone. This is first allowed to cool and then one side of the bulb is collapsed by gentle heating, producing an object shaped like a thick, hollow spoon. Because the glass on one side of the spoon is now thicker than that of the other, a change of pressure inside the device causes the spoon to curl or bend. Next, the sealed end of the tube is collapsed, pulled out into a rod, and cut or melted to the desired length. The small movements of the spoon resulting from a pressure change therein was amplified by making the rod act as a lever, which in turn rotated a mirror incorporated with a lamp-and–scale outfit, thus greatly amplifying the spoon’s movement. My rotating mirror was mounted vertically, in contact with the rod; a small permanent magnet provided a restoring force to keep the two in contact. The device was easily able to measure pressure changes as small as 0.1 mms (Hg).

A trivial point is that the gas in London was then coal gas, the mixture produced from the destructive distillation of coal; it is worth remembering that its flame is not as hot as that of "natural" gas (methane).

Something should be recorded about the Science Library. It was possible in my time to perform a **complete** literature search for a subject such as "gas phase chemical kinetics" and the appropriate theory papers, starting ca 1920. Such a task would be very, very difficult today, computers notwithstanding,----remember that there were *no commercial journals* at all then, "Blue Bits," a.k.a. "The Journal of Chemistry and Industry" being the sole example. Tetrahedron and Tetrahedron Letters (the first) did not appear until 1959.

When library boredom set in, say after the first 3-4 hours, it was fun to look at very old texts (such as Philosophical Transactions) for the 18th century. It was amusing to read a report of the solidification of quicksilver, achieved by use of a freezing mixture of ice and sal-ammoniac (ammonium chloride). The writer, a parson, explained how he had to run to tell the squire of his achievement, and recorded the surprise and delight of the latter when he was fetched to view the marvel. Another fine example was provided by a military officer's report on the use of the expansive power of the water-to-ice transition to propel a projectile at a foe. The weapon consisted of hollow cast iron spheres or cylinders which were filled with water and a shaped iron wedge or spike was hammered tightly into the filling hole. Next, the whole object was placed within an ice/salt freezing mixture. The projectile came out with great energy, provided that the cylinder itself did not split under the expansive force, but its path was also difficult to control. The report ended inconclusively and the unreliable weapon was shelved.

By the end of November my glass apparatus was nearing completion and I set about creating a visible-wavelengths spectrophotometer, aligned with the thermostatted, optically windowed glass reaction vessel, contained in an electrically wired aluminium block thermostat. It was all contained in a light-tight box made of "Transite" with sliding shutters for access. The electronic components (photo cells, resistors, valves etc) were then easily obtainable from the many military war-surplus stores in Goodge and Wardour Streets. It took several months to create and calibrate this apparatus, with an optical bench aligned with the windowed reaction vessel; green filters selected the wave length. Allan must have been wondering when I might make the first run; however a couple of weeks were lost owing to the lab being completely redecorated; this required the construction of protective hardboard boxes to surround Ron Barker's photolysis and my apparatus; see below for the aftermath. All this experience would be very difficult to repeat nowadays where much such equipment is commercial and computer controlled; the result is a generation of scientists who do not know in any detail how their apparatus actually functions. (This is especially true of such apparatus as analytical mass spectrometers).

The hardboard for our boxes was obtained from throw-outs from the Slade School of Art (next door) and so our precious apparatus was encased in nudes and landscapes. When the work was over and the protective boards removed, there was a flood in the upstairs lab, bringing the newly painted ceiling down onto Ron's apparatus. The flood was not surprising, as the lab above was that of Albert Wasserman, a rather shy refugee scientist supported by Prof. Ingold. Albert was notoriously slow to replace old rubber tubing for gas or water supplies. He apologised profusely and accepted a cup of coffee, see below.

Next, my apparatus was calibrated, the chemicals prepared and purified. Finally, at long last, the first kinetic run for the i-C_3H_7I + HI reaction took place in early June. Everything worked according to plan and the iodine produced by the reaction was spectroscopically recorded as a function of the concentrations of sec-propyl iodide and HI. Quickly it became obvious that the reaction was autocatalytic; *any* trace of iodine would set it going:-

$I\bullet + C_3H_7I \rightarrow C_3H_7\bullet + I_2$ (rate determining);

i-$C_3H_7\bullet + HI \rightarrow C_3H_8 + I\bullet$ (fast) and $I\bullet + I\bullet + M \rightarrow I_2 + M$

Thus $d(I_2)/dt = k(I_2^{1/2})(RI)$

It is worth noting that then there were relatively few commercial sources of very pure organic chemicals etc. and so I made all my own reactants, a valuable exercise, especially as some of the substances that I needed were quite difficult to make in a very pure state. Gaseous HI, nitric oxide, alkyl halides all had to be made in house. NO is made by the reaction of copper with dilute nitric acid and the gas evolved, frozen-out with liquid N_2, is pale blue, (because of some N_2O_3). It is finally purified by trap-to-trap sublimation from liquid oxygen (-183C) to pump-down (solid; -210C) nitrogen. All good experience!

My lab (which I shared with Ron Barker and Ken Fish), happened to be the home of the "coffee club". This tradition worked as follows; each afternoon, at about 2.30, one of us would proceed to the staff refectory (just across the bridge between the Chemistry Department and the main UCL building) to collect the remaining hot coffee from the kitchen. Typically this was ca 2-3 litres, already milked and contained in an enamelled jug. We kept it warm over a low Bunsen flame. This was shared out in C16 each afternoon to a group of us. All we had to provide was the brown sugar. In those years the group also comprised Hedley Miles, John Carter, Rupert Pearson, Alec Grimison, John Morton and Peter Francis. The "coffee club" friends also enjoyed group sailing ventures, hiring boats on the Norfolk Broads in 1955 and 1956.

Occasionally we would be joined by Fishwick. The latter arrived usually in pyjamas, dressing gown, slippers and a bandaged head; he would harangue us about the failings of the department. He had, I was told, been a graduate student of Ingold' s and afterwards was involved in an explosion at a munitions factory; hence his present disturbed state, although there are indeed other, equally likely, versions of his origins. Usually we could persuade him to leave, but sometimes the authority of Butch was required.

A. J. P. Martin, the inventor of gas chromatography, GC, (his Nobel Prize with Synge in 1952 was for partition chromatography) came to give a seminar, 8/3/1955. A fellow student, Rodney Stone, was quick thereafter to construct the department's first such important analytical tool. A simple mass spectrometer was built by C. A. Bunton for studying H/D kinetic isotope effects. All organic samples were combusted completely to CO_2 and H_2O; the water was separated and reduced to H_2, HD and D_2 by reaction with heated Zinc powder. The apparatus had a fixed magnetic field and the masses 1,2,3 and 4 were separated by scanning the acceleration voltage. "Bunny" immigrated to California in 1963 and was noteworthy as an example of the then current "Brain drain". (A word about gas chromatographs: when I was at the NRC in Canada in 1960, I happened to meet Bill McFadden the younger member of the Gow-Mac partnership that produced the very fine thermal conductivity cells for gas chromatography. They then still operated out of a shared private garage in New Jersey, where they machined and assembled their devices)

Jim Millen was another inventive member of staff. He supervised my friend John Morton in the building of a microwave spectrometer for the study of gaseous nitric acid, again with the major use of war surplus klystrons, waveguides etc. John took his skills to Canada, where he worked first in the group of Herzberg (Nobel Laureate 1971) at the National Research Council of Canada in Ottawa and then in his own section as a permanent researcher in electron spin resonance spectroscopy.

My lab also contained Ken Fish's Raman apparatus. This was enclosed by a homemade block-board box, about a six foot cube. An extractor fan took away the considerable quantities of ozone produced by the powerful mercury discharge lamp inside. Before the box was equipped with an efficient extractor fan, ozone had diffused into the lab and rapidly attacked rubber tubing therein, making it brittle and hence a gas/water leak liability. The negative health effects must also have been significant. Ken's runs were controlled by a timer and usually performed overnight, but even so, the odour of residual ozone traces struck one on arrival each day after an experiment had been conducted.

One day, at about 9.00 a.m. on a singularly cold, wet and windy winter morning, I was alone in the lab and had just started the first run of the day. To my surprise, CKI came in, accompanied by a guest from Moscow, Academician Dubinin. I briefly explained what my research was about and they duly left; but not before Professor Ingold had asked me how many others I shared my lab with? It turned out that on this particularly foul day, most graduate students had yet to appear and my lab was near the end of their (remarkably early) tour! A note was sent to us all demanding that we attend an address by CKI the next day and we were duly chastised for having let the side down. His message was clearly more in sorrow than in anger and was very effective. For at least a few weeks thereafter, the building was humming with activity soon after 8.30 a.m.

Much student social life centred about local pubs and restaurants, now mostly destroyed in the widening of the Euston Road. The Orange Tree, on the NW corner of the Gower Street intersection with the Euston Road was a favourite watering hole, and staff and students would gather there often, especially after a colloquium. The Orange Tree also provided food, uncommon for a pub in those times. They specialised in cold cuts and there was a chef with a tall white hat present in the evenings, to carve thin slices of ham or beef off the bone and to provide fresh French bread and butter. Together with Professors Ted Hughes, Ron Nyholm, Allan Maccoll, Charles Vernon, Harry Poole etc we would argue way the evening on topics of every kind, in a haze of tobacco smoke from Allan and Charles' cigarettes and Harry's Pipe.

This was a period of the "cold war" when, for example, Linus Pauling had his passport seized, preventing him from travelling to conferences in Europe. In contrast, one of us distributed the "Daily Worker" on campus. The McCarthy hearings in the USA caused some ripples in UK Universities and students taking jobs that required security clearance had to be vetted for their political leanings. The Australian Professors were adamant that they would never divulge their students' political remarks, attitudes or opinions, made in private or at social meetings, to the security men who went about the College interviewing staff. Graduate student life is a great place for forming and testing ones opinions about many things and the possibility that trial balloons about religion, politics etc could be used against one at some future time by Nosey Parkers from the "authorities" was considered to be thoroughly reprehensible. Thus our discussions were always free ranging and without boundaries. My attitude to the RCMP in Canada was the same, when in the late 1960's some of my own students were being vetted for "security".

However, alas, with hindsight, today I am not so sure. Present day religion-based extremism is prone to lead to terrorism, far beyond the (relatively polite) left wing leanings, or even communism of sixty years ago.

Two last items from those days also deserve a brief airing; military service and job interviews. Then, all young men were liable for two years' military service up to the age of 26. As graduate students we had deferment. The Universities joint recruiting board for the army and the RAF would visit each campus once a year to an open session where they would extol the virtues of their Force. The Navy however did not come to these meetings because they took no general national service folk, only those with strong nautical connections (e.g. senior position in the Sea Scouts) were acceptable and their participation would have already have been worked out. The RAF was really only interested in any of us who had belonged to a school air squadron or were experienced glider pilots. That left the great majority to join the Army and the dubious pleasures of spit and polish and drill---or even military action e.g. in Korea. The sessions were conducted in a large lecture theatre and the officers sat at a table on the platform in front of the blackboard. With some justification, the engineering students could always be counted upon to provide some entertainment. One year, as we waited the presentations that opened the meeting, the doors opened with a flourish and in came a parade of engineers. They came in with T-squares at the slope and marched up and down for some minutes accompanied by a series of indecipherable orders screamed by their “officer”. The representative from the RAF laughed aloud, but the Army Brass was clearly not amused. After the officers had told us of the many (largely imaginary) advantages that could accrue from some military service, the floor was open for questions. Invariably the first question was “How can we get out of it?” It was already time to leave as the proceedings had begun to degenerate.

I certainly had no wish to join the army and so I arranged interviews for a post at the Radiochemical Centre at Amersham and for the National Coal Board Coal Research establishment at Stoke Orchard, near Cheltenham. The latter was then under the direction of Jacob Bronowski, a well-known spokesman for science on radio and TV. I also went for a “security” interview at a place near Covent Garden, a strange experience indeed. Three of us also went to Harwell for interviews, but our reception and treatment made sure that we would never consider a position there. It transpired that some current wisdom was that interviews should be aggressive (and belittling) in style. In the end I opted for the Coal Board, as did Peter Francis. We started work there in the autumn of 1957; in 1958 I was no longer required/eligible for national service and took up a post-doctoral fellowship at the National Research Council in Ottawa.

The three years at University College were among the most happy and productive years of my life. The friendships begun there have endured to the present (2020).