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Celebration of Inorganic Lives
Interview with Donald C. Bradley

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Professor Don Bradley (born 7th November 1924) received his university education at Birkbeck College (University of London) where he obtained a B.Sc. (1st Class Honours in Chemistry 1946), Ph.D. (Physical Inorganic Chemistry, 1950) and D.Sc. (1959) degrees. His first University appointment was at Birkbeck College where he was Lecturer in Inorganic Chemistry (1949–1959) and his first Professorial appointment was at the University of Western Ontario, London, Ontario, Canada (1959–65). In 1965 he was appointed to the Chair of Inorganic Chemistry at Queen Mary College, University of London and served as Head of the Department (1979–83). Since 1965 he has served on many committees of Queen Mary College and the University of London and has been chairman of the University Board of Studies in Chemistry, member of the University Academic Council and the Senate. He has also played an active role in the Royal Society of Chemistry, being a member of Council and serving successively as Honorary Secretary/Treasurer, Vice-President and President of the Dalton Division. He has also been a member of Council of the Royal Institution and served as Honorary Secretary (1988–92). In 1987 Professor Bradley was made Emeritus Professor of Inorganic Chemistry of the University of London and became a Fellow of Queen Mary College in 1988.

During the past 50 years his research work has involved pioneering studies on the synthesis and characterisation of novel metallo-organic compounds such as metal alkoxides, oxoalkoxides and trialkylsiloxides, metal dialkylamides, metal dithiocar-

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(Donald C. Bradley.)

bamates, metal organo-imido complexes and metal bis-trimethylsilylamides of the transition metals and lanthanides. A highlight of this work was the establishment of the very lowest coordination numbers for transition metals and the lanthanides.

His early work on the metal alkoxides laid the foundations for subsequent developments with special reference to recent applications such as the deposition of films of metal oxides by MOCVD and the synthesis of ceramic materials including high temperature superconductors by the sol–gel process. Similarly the metal dialkylamides are being used as precursors for the deposition of metal nitride films.

This work on the metal alkoxides and metal dialkylamides demonstrated unequivocally that the physicochemical properties, such as oligomeric nature, solubility and volatility, of these metallo-organic molecules could be controlled by the steric effects of the bulky organic ligands. This principle was subsequently exploited by others to great effect in generating novel species of the p-block elements.

Recently his research has involved the synthesis of volatile organometallic compounds of aluminium, gallium and indium as precursors for the deposition of III–V semi-conductors by chemical vapour deposition. In collaboration with the late Professor M. Faktor and Dr Halina Chudzynska he devised a highly efficient process for the purification of trimethylgallium and trimethylindium by complexation with Diphos (bis-diphenylphosphinoethane). This was patented by the MoD and licensed to Epichem Ltd. who have successfully exploited the process commercially achieving world-wide sales of ultrapure precursors for the deposition of a range of III–V semiconductors. The use of fluorinated tertiary alkoxide ligands has led to the synthesis and characterisation of novel mononuclear volatile complexes of yttrium and the lanthanides, which have potential as precursors for the deposition of metal oxides or metal fluorides.

Over 250 original papers have been published by Professor Bradley and his work has been recognised by his election to the Fellowship of the Royal Society (1980) and the award of the Ludwig Mond Medal and Lectureship of the Royal Society of Chemistry (1987). In 1998 he was awarded a Royal Medal by the Royal Society. In addition to giving plenary lectures at International conferences he has given invited courses of lectures in Australia, Brazil, The People's Republic of China, Hungary and India.

P.O'B How did you first get interested in chemistry?

Watching a coal fire burning and seeing all the tarry stuff bubbling out of the hot coal and spurting yellow flames and wondering what was going on and what this was about. I'd just taken the school certificate, which was the forerunner of O Levels; in fact I didn't take sciences until the sixth form so I was really a late adherent to science.

I had a lot of ground to make up in my Higher Schools Certificate, chemistry, physics, maths and biology. I was very lucky to be in a brand new school on the Sussex Downs with excellent teachers, there were only three of us in the upper sixth science so I virtually had individual tuition; a bit of luck.

P.O'B. So you were born in Sussex?

No, I was born in London. My parents managed a pub in the Earls Court area right in the centre of London, but living there didn't suit my health. I was really brought up by my aunt and uncle with my cousin in Southhampton. I was sent down there because I was very skinny in my youth and they thought I had TB or something. So I was sent down to the South coast for my health and I flourished. I stayed on and then my uncle moved to Hove and I eventually got a scholarship to go to this new Hove County School for Boys, up on the Downs. The school has now merged and is a comprehensive. When I left school in the middle of the war, I was directed into a reserved occupation — at a research establishment on war work.

P.O'B You studied part time at Birkbeck while working on war work?

Actually Birkbeck during the war, was weekends only, because of the bombing. The College was itself bombed, quite badly damaged in fact. It was just across the road from here (the Barbican Centre) in Fetter Lane.

P.O'B Birkbeck was quite a lively place in those days, I always think of Bernal

Professor J.D. Bernal — he was out with Lord Mountbatten during the war. He did a lot of original work on wave motion, especially for the beach landings at Normandy — Bernal was never given proper credit for things he did because he was allegedly a 'fellow traveller'. In London the only Colleges operating were IC, who did a special 18 month degree for people to go into the services and Birkbeck with a part time degree. All the other colleges were evacuated. QM was at Cambridge (King's), UCL was at Bangor or Aberystwyth or somewhere. They were exciting times. There were various interesting people at Birkbeck, Sir Graham Hills and I graduated together. The year before that, Vernan Ingram, who is a Professor at MIT (Biochemistry) — and lots of other people who went on to become research directors at Kodak and May and Baker etc.

P.O'B. So you spent your days working, weekends at Birkbeck and evenings?

Fire watching — so we could cut out a bit of the travelling, we did this at the weekend. I went everywhere by bike — 21 miles per day round trip from Feltham to Alperton, at the research establishment. I came back to London during the war and returned to my parents in Feltham.

P. O'B. So what did you do during the war at the establishment?

This was the British Electrical and Allied Industries Research Association, it was funded partly by DSIR and the electrical industries, GEC etc. and I was in the chemistry lab (a one man and his dog operation) servicing physicists and electrical engineers. I was the lab boy termed the student apprentice. The work was quite varied; first of all I was involved in slopping mustard gas onto various electrical component relays and things to see if the telephone system would be affected if the Germans used mustard gas in their bombs. I got blisters for my efforts.

Then there was solving problems in radio communications in tanks. When new tanks went out to the Western desert to reinforce the 8th Army, all the power packs failed in Egypt. It turned out to be quite an interesting story. The capacitor's paper was impregnated with polychlorodiphenyl wrapped with aluminium foil and the electrodes made a sandwich and what happened was that impurities in the paper set up an electrolytic reaction under the conditions of humidity and temperature, and of course you got a short circuit. The physics chap in charge really could have discovered paper chromatography while we were doing this! We used to treat the paper with various acids and leaches and washes and then exchange it with various cations. We used to dip the paper in various solutions and we saw things eluting. Eventually we found by exchanging the paper with calcium ions the lifetime of the capacitors went up by several orders of magnitude.

It was 1946 when I graduated with a first class honours — that was a good year — we had about 8 and 3 or 4 were interviewed for the Neil Arnott Award; but they declined it because they didn't want to do Ph.D.s! One of my friends Bill Bartley, who obviously got a slightly better first than me, got the prize!

P.O'B So in 1946 you decided to study for a Ph.D. with Professor Wardlaw?

I was taken on the staff at the research association and I was told I could do research there that could be used for an external Ph.D. However, I was warned that this wasn't a very good way to do it. Then the boss, the graduate chemist in charge, said that Professor Norrish was advertising Ph.D. studentships at Cambridge. He advised me to apply and I did. I was invited to Norrish's place. It turned out the project was to study the kinetics of polymerisation, which didn't really interest me. I was given first refusal; the chap who took it on became the professor of polymer chemistry at the University of Lancaster.

By then I was thoroughly committed to full-time research at Birkbeck, which I was able to do with a DSIR grant. Although, I didn't actually start my Ph.D. until February 1947.

P.O'B. And this was fortuitous in that it led to your lifelong interest in alkoxides.

Yes. I started on zirconium alkoxides under the alias of esters: zirconium esters. The idea was to make organo-zirconium compounds as analogues of the silicones. Gilman, of course, had tried for years to make organo-compounds of transition metals. Apart from a few notable exceptions they weren't stable. So I decided there wasn't much of a future in this idea. However, I hit on the idea of using bulky ligands to control molecular association and volatility; ending up with the really great thing about my Ph.D. namely making the zirconium tetra-*t*-butoxide. This is a very volatile compound, so volatile, I lost most of it coming over in the benzene. I wondered what this stuff was, precipitating out of the benzene (zirconium hydroxide). The alkoxide hydrolysed, I was really a coordination chemist, these associated alkoxides were really coordination polymers involving alkoxide bridging.

P.O'B So what was inorganic chemistry like in the UK at that time?

There was so little relevant literature I could remember it all, page numbers and journals. Well, it was the Cinderella subject, there were very few chairs of inorganic chemistry, it was either physical, organic or both. Then Ronald Nyholm came over from Australia to work at UCL with Ingold, and then crystal field theory appeared. We were just coming to terms with the Schrödinger wave equation, the application of wave mechanics to chemical bonding. There were all these new bond theories, with Pauling's the Nature of the Chemical Bond. I think the renaissance in inorganic chemistry was sparked by Pauling's book and development of molecular orbital theory and Hückel approximations. Then crystal field theory that really came out of the physicists, was taken over by the chemists particularly by L. Orgel who was my contemporary; he was at Oxford doing a D.Phil. with Lesley Sutton. Suddenly you could see some sense in transition metal chemistry/coordination chemistry and could understand oxidation state, the relative stabilities of different oxidation states, why some metals had higher coordination numbers, why some were square planar, why some were tetrahedral etc. All this suddenly started to make sense with wave mechanics and crystal field theory and then ramifications for mechanisms and bioinorganic chemistry. In fact, working on early transition metals, which were d^0 species, we weren't actually involved in crystal field theory then because there are no d-electrons. It was only when Malcolm Chisholm did his Ph.D. at Queen Mary that we got into d^1,d^2, d^3, chromium, molybdenum and tungsten paramagnetic species. The early 1950s were, of course, marked by the famous coordination chemistry symposium organised by Joe Chatt at the ICI Laboratories at the Frythe, Welwyn Gaden City that which is held to mark the beginning of the International Conference on Coordination Chemistry that has its 50th anniversary in 2000. Ron Nyholm, Lesley Orgel and I attended the meeting. I attended because Wardlaw was one of the few professors of inorganic chemistry and was invited by Joe Chatt and he took me along. I had just finished my Ph.D., but I was just listed as Mr. D. Bradley!

P.O'B. So the Birkbeck days continued, at least 2 or 3 influential people passed through your group, Ram C. Mehrotra and Mark Faktor spring to mind

Ram Mehrotra was our first Indian Ph.D. student, he came over as a British Council Scholar and he already had an Indian D.Phil. when he came to work with Professor Wardlaw. At that time Wardlaw was busy being president of the Chemical Society and he left me to run the research group. I was his right hand man. I had quite a lot of research experience through the war, so he could rely on me.

P.O'B. What did Mehrotra do?

Mehrotra came just as I cracked the problem of alkoxides and we exploited the area. In my Ph.D. I had established that the branching of the alkyl group was crucial in reducing the molecular association and volatility of an alkoxide. We weren't sure whether it was purely a steric effect or an inductive effect. π-bonding instead of σ-bonding could satisfy the electrophillicity of the metal without increasing its coordination level. We designed a series of alkoxide groups with varying steric and electronic factors to see which was the predominant effect. Mehrotra had to make a lot of the complicated alcohols; you couldn't buy them. We did all the isomeric butyl alcohols, all the isomeric pentyls and some of the hexyls. I suppose the neopentyl was the crucial one, it is a primary alcohol so the electronic effects are different to a tertiary but it has a steric effect as well, it still has bulkiness. Mehrotra did all these isomeric alkoxides; we included silicon as well, because it's a non-transition metal. He did titanium, zirconium, and hafnium for his Ph.D. As successive students came over to work with Professor Wardlaw, I put them on to different metals, thorium, cerium etc. We didn't have any modern spectroscopic equipment so all we could do was prepare the compounds and analyse them, measure boiling points, and do molecular weights. Molecular sieves did not come in until 1957 or so!

P.O'B A few more words about your students in the early days?

Yes, when Wardlaw retired I took on my own Ph.D. students, Ian Thomas, Mark Faktor and Harry Holloway were all very good. Mark Faktor studied the thermal decomposition of the alkoxides. He pointed out that this was an excellent way of depositing the oxides! That came out of a project with Harwell to see if we could separate hafnium from zirconium by fractional distillation. However, the alkoxides tended to decompose, we wanted to find out why? This gave us a clue, later on, as to MOCVD processes. This was Mark's Ph.D., then he went to the Post Office Research Establishment (as did Harry Holloway). Harry did his Ph.D. research on the hydrolysis of metal alkoxides under controlled conditions, which led to sol–gel processing. Ian Thomas did work on siloxides that was related to some industrial sponsorship, my first from the Bradford Dyers Research Association, who had an interest in water repellent treatment for fabrics. There was an American patent that said that if you mixed a titanium alkoxide with a silicone they form a water-repellent treatment. So I wrote an application saying you could make what we now call a single-source precursor treatment by combining the siloxy part with the metal to form a trialkyl siloxide which would then hydrolyse forming the coating.

Eventually I was running out of steam……. But then the US Air Force came along and offered me a grant on inorganic polymers. They were looking for stable materials for rockets and missiles. For me it was a lifesaver. The grant gave me postdoctoral support for Ian Thomas to start metal dialkylamido work, which eventually we took with us to Canada.

P.O'B. You have a good story about travelling across the Atlantic on a flight in the early days?

In 1958, Geoff Wilkinson and I travelled to an ACS meeting to which we both had been invited, and we both had USAF contracts, so had free flights on military transport. We flew from Mildenhall to McGuire A.B. (New Jersey). Geoff had a glass ampoule in his pocket of a blue solution of potassium dispersed in a polyether — to my horror!

P.O'B How did you decide to go to Canada?

I'd been to the 1958 ACS meeting and an old school friend, who was a physics professor at the University of Western Ontario, heard on the grapevine that I was touring in the US and invited me to visit. He arranged for me to give a talk in the Chemistry Department, which they obviously liked, because on my return to the UK they invited me to go back as a Visiting Professor. I'd just arranged a year's study leave to go to Canada when I got another letter from Canada saying that they were now setting up a graduate school (a Ph.D. programme) and would I like to go to a tenured position, as Associate Professor. My wife wasn't happy about the idea of emigration so I wrote back saying 'no' — and received a letter by return saying it would now be a Full Professorship. An offer I couldn't refuse — so I persuaded my wife to go to Canada for at least 5 years. As soon as I left (in 1959) inorganic chemistry in the United Kingdom grew and lots of new jobs came up under Robbins' expansion. However, in Canada, we had great facilities IR and NMR. That was the main reason for going there — we had modern everything! (IR, NMR, mass spec.).

P.O'B Any notable people in Canada?

I had a hard job getting people, everyone was going to MIT, Harvard. etc... Elmer Alyea did well, became Professor at Guelph. John Charalambous came over as a post-doc. and met his wife there. (I brought about one or two marriages!). We carried on with the alkylamides, which were much more easily made volatile by piling on the steric effects. Our first three-coordinate species was chromium tris(diisopropylamide). Jagtar Singh Basi got me into three-coordination. The funny thing you see was that in the literature there was a brief paper by Burger and Wannagel on chromium and iron tris-bis(trimethylsilylamide). We tried other metal diisopropylamides but they were not very successful. Then we tried silylamide, at just about the time I was coming back to UK. At QM we decided that silylamide was the ideal bulky ligand for stabilizing low valent low coordination number transition metals. Eventually Joginder Ghotra went on to do the lanthanides the first examples of three-coordinated lanthanides.

P.O'B What about the invitation to come to Queen Mary College?

Joe Chatt was the first professor of inorganic chemistry at Queen Mary–he was planning to have his nitrogen fixation unit built there adjacent to the then new chemistry building. There were problems with the building project (expense) and he went to Sussex to found the ARC unit there. At that time I was already on my way to be interviewed for the Head of Department job at Brunel and on the way was interviewed for the vacant chair at QMC. I didn't really want to be a Head at that stage and Brunel chemistry was still at Acton Tech. The move to establish the new base would have taken 2/3 years and it would have been the end of my research. However I'd put in for it because of the promise I'd made to my wife.

The other thing that we found when studying the effect of bulky ligands on chromium(III) was that the *t*-butoxide disproportionated to the very volatile chromium(IV) compound presumably leaving the chromium(II) behind, but we had a bit of trouble establishing all this. I told Henry Taube who found all this rather surprising "Henry once you get into non-aqueous systems, chemistry is different". We could explain this reaction by crystal field theory: tetrahedral d^2 chromium(IV) and presumably an octahedral d^4 chromium(II) was a more stable set up than two molecules of tetrahedral chromium(III). Then Geoff Wilkinson found the same thing with alkyls of chromium; but years later.

At around that time we also got into dithiocarbamato species. We had started that in Canada; when we added carbon disulphide to metal dialkylamides there was a vigorous reaction to form dithiocarbamate. The discovery had actually been made at Birkbeck. We had no IR but UCL did! Jack Lewis kindly agreed to collaborate on alkoxide IR work. Colin Barraclough, working with Jack, collected the samples. One day he looked at a metal dialkylamide that Ian Thomas had made, and he was using CS_2, as a suitable window/solvent for IR, but on mixing it almost exploded.

We didn't take it up at that time. I mentioned it to Melvin Gitlitz doing his Ph.D. with me in Canada who said maybe it's an eight-coordinated titanium. Robin Clark and Nyholme later showed that titanium could be eight-coordinated in the $TiCl_4(diarsine)_2$ complex. When I was back at QM with Mike Hursthouse, we collaborated on a crystal structure with Sandro Vaciago in Rome, who was also interested in crystal structures of metal dithiocarbamates and found indeed it was eight-cordinated. We could make transition metal dithiocarbamates easily in non-aqueous solutions by adding CS_2 to the metal dialkylamide; it was a big break through that was never really exploited (chemistry not accessible in aqueous solution). However, grants became hard to obtain even though the work was going well, it was so frustrating……. Don't remind me!

So back to London, UK, Queen Mary College, we had a new building and the latest equipment, except we only got a second hand manual diffractometer to start crystallographic work with Mike Hursthouse — who was great! We had to have structures. We then did a lot of low coordination work all across the transition metals series from scandium right through to the end and with additional ligands like phosphine with bivalent metals and univalent metals. We found univalent nickel, we stabilised nickel(I) silylamide with two phosphines, unusual cobalt(I) species too, and so on. We did other work on five-coordination and eight-coordinate zirconium.

P.O'B. Lets reflect on what Queen Mary College was like when you went there?

I was part of the big expansion. Keble Sykes was building up the Department. We took on five or six lecturers. I think I was the only professor appointed at that time. Initially they had had Gordon Stone as Reader; he was there a year or two when he came back from Harvard. (He didn't get tenure at Harvard, but then Geoff Wilkinson didn't either). Gordon was involved in planning the new chemistry building. Then he got the chair at Bristol — which I'd actually applied for as well! They then thought (at QMC) if we're going to keep anyone good we need to make them Professor, and that's when they got Joe Chatt, but he did not stay for a year; then they got me. At the first Inorganic group meeting, I attended, Alan Massey (then a lecturer) said we're making a book on how long you are going to stay! At that time the inorganic group was myself, Alan Hart, Alan Massey, that's about it. We started expanding, we got Mike Hursthouse and Peter Thornton came along, Mike Mingos took his first faculty position as a lecturer at Queen Mary. Things really got going with these people. Oh! Ed Randall was there of course, we were building up our NMR and ESR with Keith Sales, mass spectrometry with Trevor Toube. Things were really bubbling in the late 1960s, early 1970s.

P.O'B. London was very exciting in the late sixties, Nyholm was still alive, Wilkinson was at the height of his powers and you!

Steve Mason was at King's, Jack Lewis briefly came back from Manchester to UCL. London really was a great place to be. Then everything started to go off in the late seventies, research grants were thin on the ground but we still managed to trudge along. Then Mark Faktor got in touch from British Telecom Research, he had by now reached a high position, and said it's time you inorganic chemists got involved in this exciting electronic materials area — chemical vapour deposition (CVD).

So that's how I changed from purely academic research to what I will call applied — it was very nice to be doing something that was useful in the short term. So we got involved in depositing III–V semiconductors and then metal oxides. We got involved in the Joint Opto-Electronic Research Scheme (JOERS). Eventually you joined the group and got involved in II–VI semiconductors; and the rest is history. There were again great days with Eric White and Mark Faktor as Visiting Professors, sadly they passed away and we lost two key people. Lets face it I'm just an ordinary inorganic structural chemist not a real crystal grower.

P.O'B. Lets talk about the diphos route for the purification of metal alkyls

Mark Faktor and Halina Chudzynska and I were discussing ways of purifying trimethylgallium and trimethylindium. I said what we need is something to coordinate and satisfy their higher coordination number and to stabilise the compounds; they are horrendously air-sensitive (pyrophoric). Hopefully something that would crystallise and could be used to purify them — I came up with the idea of using triphenylphosphine and so Halina made the triphenylphosphine adduct of trimethylgallium and it crystallised. The other thing we wanted was a coordination compound that wouldn't be too stable. We knew that by using a fairly bulky phosphine, phosphorus wouldn't be such a strong donor as nitrogen and the phenyl groups are very steric, so you probably wouldn't have a very strong coordination compound. Then it would dissociate very readily to release the volatile trimethylgallium; it just worked. We didn't realise how well it would work! We purified triphenylphosphine by vacuum sublimation and that was going to contaminate things so we needed a less volatile phosphine, bis-diphenyl phosphinoethane (dppe, diphos). Although it chelates with transition metals, it doesn't chelate to Group III alkyls. We got 1 mol of diphos to 2 mol of trimethylgallium and the great thing was that with one crystallisation, the impurity levels dropped by 10 000. This was a great development for the purification of the materials. This work was done in conjunction with the MoD (RSRE at Malvern) and was then taken up in the JOERS project which came later, the indium phosphide project. So we tried triphenyl phosphine, diphos, triphos, tetraphos, they all worked. Diphos was commercially available, very pure, but the others were very expensive. So we settled for that. The MoD took out a patent and a small British company Epichem licensed it; the rest is history. Epichem were successful in export and got a Queen's award for industry and British Telecom also got a Queens Award. So I ended up doing something extremely useful, after all this academic research.

P.O'B. Your work has had a lot of influence on those interested in materials especially your early work on oxides, a lot of Americans think you were there 20–30 years ago?

The papers with Mark Faktor were a benchmark. Mark showed what was happening. Although the zirconium tertiary alkoxides are thermally unstable the zirconium oxygen bond is really very strong, over 100 Kcal mol^{-1}, so it wasn't breaking down by fission of the zirconium–oxygen bond, it was the organic part that was going. It was a high-temperature hydrolysis, you only need one molecule of tertiary alcohol to dehydrate on the glass surface to give you isobutylene and water, and then that water molecule immediately hydrolyses off the two molecules of tertiary alcohol. You have two molecules that split off, which give you four and you've got a chain reaction. Mark worked this all out — Brilliant! Gas chromatography, etc. confirmed all this. He used a spoon gauge, which he had to make himself. Douglas Swanwick, another Birkbeck Ph.D. student did all the accurate vapour pressure measurements — that was tremendous work. He was a wonderful glass blower; he made the spoon gauges. It was basic fundamental data — all the alkoxide vapour pressure work was extremely accurate. Mark took over from Douglas and used the technique. He would vaporise a gram or so of the zirconium *t*-butoxide in a globe attached to a spoon gauge — so you can monitor the pressure. He then gradually raised the temperature and the pressure stayed steady for a long time and all of a sudden it went up and you got a deposit of zirconium oxide. Mark could see the pressure rise was something like sixfold. One molecule of zirconium *t*-butoxide gave 4 molecules of isobutylene and two molecules of water (vapour at this temperature). He did chromatography to check this. There was an induction period and sooner or later the thing would decompose. The decomposition had the characteristic sigmoid curve of a chain reaction. Classical Hinshelwood/Semenov kinetics and he was able to determine a rate constant. It turned out to be the rate constant for the surface catalysed dehydration of the tertiary alcohol. So alkoxides were inherently unstable. To prove this idea, we used Ian Thomas's trialkylsiloxide, although a trialkylsilanol condenses to hexaalkyldisiloxane and water and it can't give a chain reaction because you only get one molecule of water from two molecules of silanol. So even if the siloxy compound broke down, which it did not seem to, it wouldn't give this catastrophic decomposition. We found that the siloxy compounds were indefinitely stable at the high temperatures at which the tertiary metal alkoxides just decomposed. This gave a clue as to what was happening in the thermal decomposition of alkoxides.

P.O'B. What about Queen Mary College days and Malcolm (Chisholm)?

This is what got us into crystal field theory, which was fantastic. Malcolm was working on niobium(IV), chromium(IV), molybdenum(IV) and tungsten(IV), alkoxides and dialkylamides, doing ESR and mass spectroscopy. He dragged me into crystal field theory. He got the tungsten hexa(dimethylamide), a six-coordinate species, which was very stable. A crystallographer dropped a sample on the bench exposed it to air and nothing happened. The steric crowding with all those methyl groups! He also got tantalum(V), a five-coordinate dialkylamides. That was a funny business about the dropped crystallographic sample. Malcolm just scraped up the sample redissolved it and re-used it. Malcolm also introduced the group to the local pub; he was a great socialiser. He had a very vigorous student life. He didn't take chemistry very seriously until after his B.Sc. He just scraped an upper second and then did a final year project on iron enneacarbonyl and that set him off. I happened to have a grant (he would have worked with Allan Massey) but I persuaded him to work with me. Allan did some very good work; he had Dave Fenton working with him at QMC.

You mentioned the lanthanides, to get a three-coordinated lanthanide was incredible, it was done by Ghotra a post-doc. from the Punjab, India, he's now at Kingston University (London). Interesting that in Canada, Basi also from the Punjab, did the first three-coordinated chromium.

When Malcolm finished his Ph.D. he went to post-doc. with Howard Clark at Western Ontario on organoplatinum compounds, very air-stable. Malcolm made about 20 in a week. Then he went to Princeton and assembled an impresive research group and exploited the triple bond stuff he did at the end of his Ph.D. (I lost out on it because my next Ph.D. student after Malcolm was a bit mad and wouldn't work on my ideas!).

P.O'B. Anything else you'd like to say?

Ian Thomas after working with a big glass manufacturer in Toledo, went to Lawrence Livermore laboratories to work on the big laser fusion project. There he used his skills for depositing oxide films on mirrors for the laser project, another area where our work has been used in a spectacular way.

The American, Chuck Ewing, from Owens-Illinois in Toledo worked on chromium(II), very tricky work, he survived an explosion. Another one who survived an explosion was Ken Aitcheson who got liquid oxygen into gallium trimethyl — he was lucky! One of the bolts was embedded in the framework of his bench, he was very lucky!!

P.O'B. If you were starting out now, would you find it a challenge or intimidating, e.g. the information mountain?

Going back to Malcolm's hexa(dimethylamide) — the crystal structure we did at QMW — it turned out that we had a solid solution with the hexa(dimethylamide) ditungsten. Because the triple bond is so short it almost looked like it was just a normal monomeric hexa(dimethylamide). Also, it fitted into the lattice, it was later on that Malcolm found that the analysis wasn't quite right. The dinuclear compound is reactive if you add an alcohol whereas the hexa(dimethylamide), the monomer, is not, so he was able to isolate authentic hexa(dimethylamide).

Another interesting compound Malcolm made was niobium tetra(diethylamide) (a d^1 system) but the analysis was always a little bit out, although he'd purified it by distillation; lovely EPR spectrum. Then I had this Brazilian co-worker, Claudio Airoldi, he did some work on this. One day, he came to me and said he had got the NMR spectrum — "what do you mean, you can't, it's paramagnetic" but he had. I immediately recognised he had got a niobium(V) species. It turns out that when you start with niobium pentachloride you get an impure penta(diethylamide), which breaks down to give you the tetra(diethylamide) plus another imino alkylamide which contains a three membered metal–nitrogen–carbon–metal ring and this has a chiral carbon with a characteristic NMR signature. Ian Thomas had already found a similar reaction with tantalum in his earlier work and so we realised that this is what had happened to Malcolm's synthesis. You can't separate it and it is EPR silent and we'd never looked at the NMR because there shouldn't have been one. So I looked back in Malcolm's thesis and found tungsten diethylamide did the same thing. A good example of things unravelling in time and people doing things they shouldn't do.

There's also a lot of luck!

P.O'B. Would you like to comment on the Mond Medal and the recent Royal Society Royal Medal

The Royal Society of Chemistry's Mond medal was a tribute to my co-workers. It was very nice because the Mond process was the first industrial use of chemical vapour deposition — nickel carbonyl for nickel. It's a bit embarrassing that many Ph.D. students don't seem now to know what the Mond process is!

The Royal Medal is quite remarkable; not many chemists get it. The nice thing is that this year Malcolm has got the Davy medal. It's a nice way to round things off. The Royal Medal is great recognition of a lifetime of research. I'm hoping to complete the new book on alkoxides soon, and I'm writing up research work that hasn't seen the light of day for ages. I still go to College and share an office with Bernard Aylett and Peter Thornton.

I suppose the most dramatic thing I've seen over the years is the application of computers to physical science. First of all, automated diffractometers and routine crystal structure determination, incredible! and then NMR spectroscopy. Fourier transform, made a fantastic difference.

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Appendix A. Addendum

Explanation of less well known acronyms in this article: ARC = Agricultural Research Council; CVD = Chemical Vapour Deposition; DSIR = Department of Scientific and Industrial Research; IC = Imperial College; MIT = Massachusetts Institute of Technology; MoD = Ministry of Defence; QMC or QMW = Queen Mary College later Queen Mary and Westfield College — following the merger of the two institutions; RSRE = Royal Signals and Radar Establishment; UCL = University College London; USAF = United States Air Force.