



Big and little Meccano

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ABSTRACT

The emergence of the mechanical bond during the past 25 years is giving chemistry a fillip in more ways than one. While its arrival on the scene is already impacting materials science and molecular nanotechnology, it is providing a new lease of life to chemical synthesis where mechanical bond formation occurs as a consequence of the all-important templation orchestrated by molecular recognition and self-assembly. The way in which covalent bond formation activates noncovalent bonding interactions, switching on molecular recognition that leads to self-assembly, and the template-directed synthesis of mechanically interlocked molecules—of which the so-called catenanes and rotaxanes may be regarded as the prototypes—has introduced a level of integration into chemical synthesis that has not previously been attained jointly at the supramolecular and molecular levels. The challenge now is to carry this level of integration during molecular synthesis beyond relatively small molecules into the realms of precisely functionalized extended molecular structures and superstructures that perform functions in a collective manner as the key sources of instruction, activation, and performance in multi-component integrated circuits and devices. These forays into organic chemistry by a scientific nomad are traced through thick and thin from the Athens of the North to the Windy City by Lake Michigan with interludes on the edge of the Canadian Shield beside Lake Ontario, in the Socialist Republic of South Yorkshire, on the Plains of Cheshire beside the Wirral, in the Midlands in the Heartland of Albion, and in the City of Angels beside the Peaceful Sea.

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I was born in Scotland's capital, Edinburgh—often referred to somewhat derogatorily as Auld Reekie and, at other times, significantly more reverently, as the Athens of the North—on Victoria Day in 1942, slap-bang in the middle of the Second World War. I was only six months old when my father decided to forsake the comparative comfort and relative security of being a farm manager, overseeing a couple of the University of Edinburgh's farms in the vicinity of Balerno on the west side of the city, to take up the tenancy, with my mother and myself in tow, of one of Lord Rosebery's farms—namely Edgelaw Farm—beside the reservoir of the same name about a dozen miles more or less south of the capital.

This move on my father's part in his early 30s could be likened to that of a young academic leaving the somewhat sheltered existence of a postdoctoral position for an assistant professorship—that is, he went from being a salaried man, tackling a limited number of different projects in a highly supportive environment, to being an entrepreneur running many different lines of business, some of which would pay off handsomely and others which would fail rather miserably and so have to be subsidized as part of an overall

business venture that had to somehow, come what may, keep the wolf from the Stoddarts' door.

During my formative years, attending what I now often look back upon as the university of life, I was to learn how to keep many balls in the air at once for such was the art and practice of mixed arable farming in the Lowlands of Scotland in the middle of the 20th century. It was a time of rapid development in agriculture in that part of the world and my parents had no choice but to embrace change as if there was no tomorrow. The horse and cart gave way to the tractor and trailer almost overnight. The binder and threshing mill yielded to the combine harvester and baler in a matter of a few years. Hand milking of cows in byres (cowsheds) was replaced by machines in milking parlors in next to no time at all. Hens were denied free range for deep litter or battery environments, except that my parents resisted this particular change and became known as the suppliers of organic (bio) eggs long before their time in name at least!

I was to learn early on from these experiences that one can never afford to believe, for very long at least, that things were better in the good old days—and to hold on doggedly to old practices when new ones are staring you in the face. Indeed, it soon became evident to me that, if one chooses to be the leader in innovation, then one stands a pretty good chance of stealing a march on all and

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sundry. While competition was invigorating, winning competitions was the spice of life. In the midst of the daily routine, dealing with crises, sometimes verging on disasters, was commonplace. Indeed, people in the farming community were greatly valued for their problem-solving capabilities. Most of the contraptions that came to fore during my first 25 years spent down on the farm could be stripped down to their basic parts and put back together again. The personal satisfaction that could be drawn, either from getting a machine to work that was kaput or from enticing some piece of machinery to perform at something like its optimal efficiency, was endless. Also, in an environment where keeping a slightly sick machine going—and hence making sure that people were gainfully employed—was not unimportant, one learnt that, if a key piece of machinery was not actually broken, then there was no need to fix it! Wear and tear went hand in hand with toil and trouble.

Edgelaw Farm (Fig. 1a) up until 1959, when I was 17 and all but ready to leave school for university, had another unique challenge to put before its tenants. It had, during our first 17 years there, remained unconnected to the national electricity grid, an anachronism which meant that we were competing with all around us, barring those on one of the neighboring farms, with our hands tied behind our backs. This particular situation was to teach me another important lesson in life—namely, how to turn an unavoidably negative experience to my advantage. All it means is that one has simply got to be much more creative in one's thinking and actions in order to make progress against overwhelming odds. The experience was to become useful to me in a completely different context in later life. That occasion came when my late wife was to fall foul of breast cancer for, as it turned out, during one-third of our married lives and one-fifth (1992–2004) of her own life, we had to continue somehow to function as scientists and do science with a whole array of impediments, and an endless, it seemed, supply of distractions in the foreground, and yet somehow, in my case, be able to survive the horrific experience in a professional, as well as a personal, context.

There was another feature of this close-knit farming community that I was to find could be transplanted into the arena of scientific

research to my considerable advantage. The community was a highly supportive one where networking and collaboration were the order of the day. It was, for example, common practice for one farmer to lend his machinery and assign his workforce to another farmer whose crop of hay, oats, barley, wheat, etc. was ready to be harvested before his own: the gesture would then be returned automatically when the time was ripe, so to speak! Later on, I was to call on this practice during my own scientific career for, just as farming back in Midlothian half-a-century ago was much more important than the farmers and the farmhands, science is much more important than the scientists—be they principal investigators, postdoctoral scholars, or (post)graduate students. Sinking pride and tempering egos are just as effective today in doing good science, as they were in farming 50 years ago. I am in no doubt at all that, by networking and collaborating, not only can a piece of science be greatly enhanced, but also the scientists become enriched with new knowledge and practices.

Another piece of good fortune that stemmed from my upbringing on the farm was the fact that my relationship with my parents was not too dissimilar from that of a graduate student, blessed with two outstanding joint mentors, offering complementary skill sets. In addition, I was surrounded by farmhands who, in many ways, could be equated with the postdoctoral fellows in a well-balanced research group. I was to pick up a lot during 25 years from these rich and colorful characters who came and went, bringing and taking their families, sometimes quite large, with them. Being an only child, their children were my playmates, much like one's fellow graduate students in a research laboratory. Our parents were all too busy attending to their daily tasks and chores and so we were left to run wild through the many nooks and crannies of the farmstead, to roam on foot far and wide into the countryside with its abundance of paddocks and fields, not to mention woods and hedgerows, and to take our buggies and bicycles along tracks and paths by burns (streams) and ditches. Then, there was always the reservoir when we wanted to bring a pond with rafts and boats into our lives. We lived a carefree existence courting considerable danger climbing roofs, burrowing through

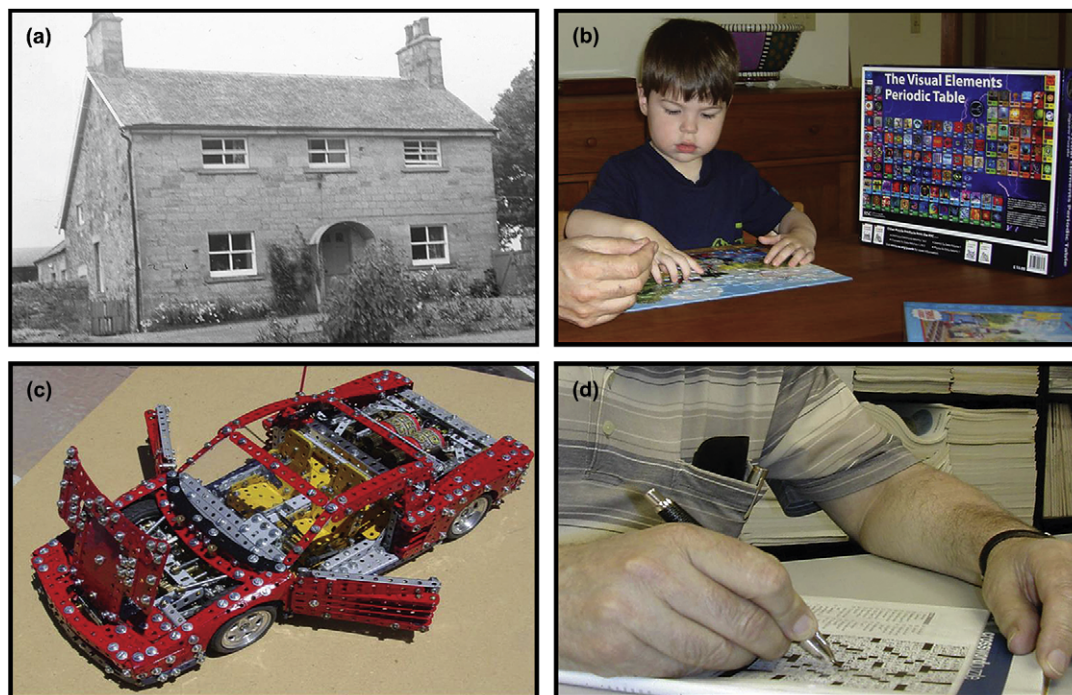


Figure 1. (a) Edgelaw farmhouse where the author spent the first 25 years of his life from 1942 to 1967. (b) A 50-piece jigsaw has just been completed while a 550 piece one waits in the background. (c) A product of Meccano. (d) Solving a crossword.

passages between bales of hay in the haysheds, and speeding down hillsides on carts with two, three, or four wheels in the summer and on homemade sledges in the winter. There were occasional accidents but we learnt to see them coming and so take the necessary precautions to avoid getting hurt. On reflection, however, it continues to be a source of amazement to me that no one got killed or even badly maimed. We were a resilient bunch. I had survived the mishap, when I was three years of age, of tumbling head over heels out of the family car doing around 30 mph. I was positioned in the passenger's seat, sitting on the lap of a friend of my mother, who was driving the car at the time. My hand or foot must have come in contact with the door handle, opening the door, and out I went onto the side of the road. I got off with no more than a grazed upper lip and have no memories of the episode, which could have changed my life!

Both indoors and outdoors play, like work, often times was a highly organized activity. From a young age, I was addicted to solving jigsaw puzzles (Fig. 1b), a pursuit which was surely some kind of preparation for coming to terms with molecular recognition later on in my education as a stereochemist. From about aged 10 onwards, I was to spend many an hour with my Meccano set (Fig. 1c), building progressively more and more sophisticated working models in miniature of the types of constructions, gadgets, and machines that were rapidly invading the rural and urban landscapes in the 1950s. The urge to make something I had designed myself and get it to work after a fashion was to find expression later on when my passion for chemical synthesis, in the broadest of senses, of unnatural products began to develop. There is no doubt that my Meccano set whetted my appetite 40 years later for constructing artificial molecular machinery. My fascination for tinkering with mechanical devices had also been increased considerably by the servicing of tractors and motor cars on the farm, for my childhood coincided with that time when you could take engines apart, clean them, replace worn parts, and put them back together again. In my later teens, my father introduced me to the art of solving crossword puzzles (Fig. 1d), invariably in fierce yet friendly competition with him. I was always struggling with his flow of words that fitted together for he had an extensive vocabulary and was a fountain of knowledge into the bargain. My father's elder sister, who had graduated with a degree in English from Glasgow in the 1930s, was a writer of short stories, some of which were broadcast on radio by the BBC. I must have been at least 20 before I could even begin to follow a conversation in the extended Stoddart family.

At various stages in my youth I reared bantams, kept bees, and practiced gardening, all with varying degrees of success. Team playing was also the order of the day both indoors, where card playing was a favorite and frequent family pastime, and outdoors, where I graduated progressively from hide and seek, to kick the can, to rounders, and, of course, ultimately to football. As the years went by on the farm I was expected to do my fair share of the work and sometimes more. I learnt to feed hens, lamb ewes, milk cows, and drive tractors, all of which I was doing proficiently by the time I entered the senior school at aged 12. Eventually, I was able to plough (plow) to my father's satisfaction and could reverse a tractor and trailer into almost any gap at any angle at the first go—a counterintuitive exercise if ever there was one! I could not have had a better training in multitasking than was provided at the university of life on the farm.

My formal education began with my going to the local village school in Carrington when I was four. Although when I joined the school in the autumn (fall) of 1946 there were only four other children in attendance, by the time I left in 1950, the number of pupils had risen sharply to 28. The arrival in the village of a couple of large families could change overnight the dynamics in the school quite remarkably. In this rapidly changing environment, where the older pupils looked after the younger ones to a great extent, I was to learn quickly from Miss Morrison who did not hesitate to use the

tawse (belt) for poor performance, let alone for bad behavior. At one period during my four years at Carrington I was the only boy in the school and so I was duly taught to knit along with all the girls. Knitting is like learning to ride a bicycle. Once you know how to do it, you never forget! Much of my early training in the three Rs—reading, writing, and arithmetic—was carried out on slates with chalk and dusters. At some stage, however, I was introduced to the use of an ink pen with a replaceable nib that was fed from an ink well buried in my desk. I guess this is when my love affair with pens—now fountain in nature—and paper began. I find it difficult even to this day to draw the same satisfaction from using a ball-point (biro) pen. My learning experience at Carrington was certainly one that introduced me to the attributes of empowering young people with responsibilities just as soon as they are ready to shoulder them. In many ways, my research group has run itself quite successfully after the model of the village school in Carrington for the past 30 years or thereabouts.

At aged eight my mother decided that I should go to a fee-paying, all-boys, day school in Edinburgh. She chose Melville College—formerly called the Edinburgh Institution and now part of Stewart's Melville College—because she liked its red (predominantly) and black uniform, which I was then obliged to wear for the remainder of my school days. I had to take an entrance examination to get into Melville College. Apparently, I passed with flying colors, mainly because of all that I had learnt at the village school from Miss Morrison. I was at least one year ahead of the boys who had started their education at Melville, and so a couple of weeks after I arrived as a new boy I was moved up a class. I was blessed with some really outstanding teachers in the primary school. On my most recent trip back to Edinburgh this spring, I renewed my acquaintance with Miss Pratt (now Mrs McKelvey) after more than half a century. She recalled a very shy little boy, shyness being something that has taken me the best part of a lifetime to overcome!

Until I was 16 and could do the journey independently to school in Edinburgh on a Lambretta—with no end of spills on black ice during the winter months—my mother would drive me three miles in the family's Hillman Minx to the nearest bus terminal at Rosewell. We picked up other boys and girls bound for Edinburgh schools on the way. Her consideration for them generated lots of friends for me and she seemed quite content to be running a small school bus for the local community. The double-decker bus journey, on the top deck—where the atmosphere could be cut with a knife and the socializing could become quite intense, if not sometimes intimate—took 45 min, leaving Rosewell at 7:40 a.m. and arriving at Saint Andrews Square just before eight-thirty. That left a one-mile walk, usually in the company of a friend or two, along George Street to the school on Melville Street. When time was short, for school started with assembly at ten-to-nine, or if it was raining cats and dogs, I could take a tram most of the way for an old penny. In retrospect, I now realize that my senior school education was a uniquely special experience that no doubt still reflected in the 1950s some of the flavor and hallmarks of the period of the Scottish Enlightenment two centuries previously. I guess I was only about half-a-dozen generations removed from that remarkable time in Edinburgh's and Scotland's history. I was taught Latin, English Language, English Literature, French, History, Geography, Mathematics, Music, Physics, and Chemistry by teachers, most of whom could have been university professors. Exercise and sport was also a major part of the school curriculum, with rugby, cricket, hockey, and swimming all being pursued on a compulsory basis at some time during the school year. One thing that happened every school day, come rain, hail, or shine, was 'the walk'. At 10:50 a.m. a bell would ring and the whole school would empty, in a highly organized manner by classes, starting with the juniors and ending with the seniors, into Melville Street and walk round the block in the

ensuing 10 min. Given the very bright uniforms we were all wearing, it must have been a pretty sight for the local residents to behold from indoors. In my final year at Melville, I was appointed by the Headmaster to be the second prefect of the school. This executive-style position gave me adequate opportunities to develop leadership skills. It also left me with responsibilities for planning and running school and extracurricular events, an experience which was to become invaluable when I found myself thrust into leadership roles at Birmingham University as the Head of the Chemistry Department and then subsequently at UCLA as the Director of the California NanoSystems Institute (CNSI). Somewhat ironically, these all-male (and all-female) schools in Britain in 1950s were still preparing young men and women to go out and run an empire that was rapidly disintegrating and disappearing during their schooling!

My mother was a terrific baker and cook, knowing instinctively how and when to mix ingredients—rarely, if ever, weighing anything out—and when to stop beating and heating mixtures. She could bake and cook effortlessly while, at the same time, feeding hens, mucking out henhouses, rearing chickens, gathering eggs, and selling them. My early prowess as a practical chemist in the laboratory undoubtedly owed much to my watching the remarkable time and motion machine she was in action. In my third year as an undergraduate student in the Chemistry Department at Edinburgh, one of the lecturers who ran a particularly demanding practical analytical chemistry laboratory put the fear of death into me and my fellow students in the class by informing us that we would be handling enough cyanide in one experiment to kill the whole of Edinburgh. He was also foolhardy enough to boast that no student had ever come anywhere near finishing all the experiments in his practical course during its 10-week duration. Little did he know that he had wound me up sufficiently and issued me with a challenge to rob him of that boast in future years. In the event, I completed his lab class in seven weeks, a feat that was to earn me a paid position in his research group over the following summer. I became addicted to doing research almost overnight and that addiction has remained with me to this day.

My father, by far the best educated and most well-read farmer in the community, had two vices. He was addicted to gambling—particularly backing horses over the phone if not at the racecourses themselves—and almost brought the family to rack and ruin at one point. With my mother's encouragement, he accepted an offer to become an elder of the Church of Scotland in Carrington Parish. I think she hoped it would quell his addiction to gambling but, of course, it did nothing of the kind. For a while, I followed his example by doing the football pools until I learnt that any short-term gains were more than squandered over a prolonged period of time. Gambling was not for me and nor was smoking cigarettes, my father's other vice. In the event, his chain smoking was to take him to an early grave. My repugnance of this habit was so extreme and severe that I have still to put a cigarette to my lips to this day, having smoked more of them passively as a young boy in the family home than I care to contemplate.

One good habit I did acquire from my father is fastidiousness when it comes to presentation. Whether he was showing heifers or selling lambs, he would spend hours—even working well into the night or wee small hours before a big event—bathing, titivating, and manicuring these animals so that when they entered the show ring or the sale pen, they were specimens to behold. I have tried throughout my scientific career to apply these same principles to the presentation of my science—whether it be producing grant applications, writing reports, or preparing manuscripts for publication. My father was highly innovative when it came to turning out his produce—whatever he was selling—for consumption. Animals and crops were most definitely sold at a premium above the going rate as

a result of his attention to detail, together with his flair for doing something uniquely different that caught the eye. In introducing, very early on, color into our presentations of molecular structures and in persevering with the use of graphical representations (cartoons) to convey concepts and illustrate principles—occasionally in the face of considerable opposition—I was merely following in my father's footsteps.

Come 1 March 1967, I was on my way to Canada to take up a postdoctoral fellowship with Ken Jones at Queen's University in Kingston, Ontario that had been arranged—somewhat after the style of an arranged marriage—by Sir Edmund Hirst, the first Forbes Professor of Chemistry at Edinburgh University. I had begun my research career working on *Acacia* gums^{1,2} in his world-renowned carbohydrate school and it was his expectation that I would remain a carbohydrate chemist³ for the rest of my life. In the event, as soon as I set foot in Jones' laboratory, Ken informed me that he was shortly to depart for Curitiba in Brazil and would be spending a whole year there on sabbatical leave. This totally unexpected development was soon to work to my advantage. My free farm-boy spirit was aroused immediately when I read Pedersen's first communication⁴ in the *Journal of the American Chemical Society* in the spring of 1967 on crown ethers. I quickly realized that these macrocyclic polyethers shared some of the constitutional features (OCO repeating units) with the sugars and so I set out at Queen's University—and then subsequently at Sheffield where I was appointed to a lectureship in organic chemistry in 1970—to pursue what I called lock-and-key chemistry⁵ by marrying Pedersen's crown ethers to Emil Fischer's carbohydrates. In the course of 70s, which took me on a journey from carbohydrates to enzyme analogues,⁶ my research was completely overshadowed and eclipsed by that of Cram on host–guest chemistry^{7,8} and that of Lehn on supramolecular chemistry.^{9,10} I did not stand a cat's chance in hell of being able to compete with either one of these giants of chemistry, let alone with the two of them simultaneously. As it turned out, my progress in research was forestalled and frustrated by all the many antics that pervaded the extremely hierarchical academic system in Britain at that time. As the 70s wore on, I yearned to return to North America and eventually I got the chance with a Science Research Council (SRC) Senior Visiting Fellowship to spend the first three months of 1978 with Cram at the University of California, Los Angeles (UCLA). This short sabbatical was a real breath of fresh air. Little did either of us appreciate that I would be returning two decades later to succeed him as the holder of the Saul Winstein Chair in Chemistry. While I was at UCLA, I was to receive some more good news. I heard from the SRC that they were ready, willing, and able to support my secondment to the Imperial Chemical Industries (ICI) Corporate Laboratory in Runcorn, Cheshire under the auspices of a brand new Cooperative Research Scheme for three years. Finally, I could look forward to breaking loose from the shackles of the British academic system. I was over the moon. I was free at last to be myself and do my own thing. When I arrived in Runcorn in the summer of 1978 I met up with a bright young chemist, Howard Colquhoun, now a Professor of Chemistry at Reading University. I will let Howard take up the story now in his own words, written in June of last year just before a symposium in Edinburgh in honor of my 65th birthday.

Fraser at the Corporate Lab (1978–1981)

In 1978, while a Lecturer at the University of Sheffield, Fraser was awarded an SRC Cooperative Research Grant to work 'on secondment' at the ICI Corporate Research Laboratory in Cheshire, where he was based in the Catalysis Group led by Warren Hewertson. This sabbatical enabled Fraser to work more or less full time on research at ICI between 1978 and 1981, and he and his family moved to Chester to

be nearer to the Lab. However, I do recall that Fraser was still teaching at Sheffield, on a day-visit basis, and that somehow he also continued to maintain a research group there. (Fraser—then as now—always seemed to be doing about two-and-a-half normal people's jobs quite satisfactorily, as was once also said of Sir John Cockcroft.)

At the Corporate Lab he worked in a windowless, broom-cupboard sort of an office at the top of the building, from which he supervised a group of PhD students, including David Holland and Tom Crawshaw, who were working mainly on carbohydrate and crown ether derivatives as potential chiral auxiliaries for catalysis. This group was based in lab C04, where I also happened to be working (on di-nitrogen–transition metal chemistry), having joined the Corporate Lab in 1977 after an MA in Cambridge, a PhD in London with Bernard Aylett, and a postdoc at Warwick with Malcolm Wallbridge.

One day in early 1980, Fraser and I were discussing crown ether chemistry in the context of Tom Crawshaw's project, and the idea gradually emerged that ammonia, when coordinated to a transition metal center might form three strong(ish) hydrogen bonds to [18]crown-6 in exactly the same way that an alkylammonium ion does. Neither of us can recall this key conversation in detail—Fraser seems to think that I suggested the idea, whereas I am quite convinced that he produced it—but in any event it probably arose from our thinking that (i) most catalysts are transition metal based, (ii) ammonia binds as a ligand to all sorts of transition metals, and (iii) a chiral crown ether derivative, if it did indeed hydrogen bond to coordinated ammonia might act as a sort of supramolecular chiral ligand and thus be able to influence the enantiomeric selectivity of a chiral-catalytic reaction.

Fraser was also aware of instances where attempts to complex [18]crown-6 directly to transition metal cations had instead produced crystals in which the crown ether was found to be hydrogen bonded, in the solid state, to metal-coordinated water. So, as the postulated crown–ammonia–metal interaction seemed a quite reasonable and potentially very exciting idea, I said I would go away and have a look in the literature. This survey produced lots of evidence, going back many years, for coordinated ammonia being able to form hydrogen bonds in a general sort of way, e.g., to solvents and to counterions in cationic complexes, but nothing at all about its possible interactions with crown ethers.

An especially pretty technique for detecting hydrogen bonding to coordinated ammonia had been described by Joseph Chatt (a particular scientific hero to us and often an unknowing godfather to our work—see below) who had found that the infrared stretching frequency of the carbonyl ligand in the cationic complex $[\text{Ru}(\text{NH}_3)_5(\text{CO})]^{2+}$ was highly dependent on the nature of the counterion. This frequency was much lower for chloride or bromide than it was for hexafluorophosphate or tetrafluoroborate, and Chatt had shown that this difference reflected the strength of hydrogen bonding between the anion and coordinated ammonia in the complex cation. Hydrogen bonding was much stronger for the small halide ions than for the larger counterions, and this fact led to increased electron density on the metal and so in turn to a reduction in the CO stretching frequency.

I knew about Chatt's work and, as a test of our proposed crown–ammonia–metal interaction, I suggested we should use a complex with just one ammonia ligand, $[\text{CpFe}(\text{NH}_3)(\text{CO})_2][\text{PF}_6]$, in which the CO ligands would act as infrared probes. As a control, we used the corresponding complex in which pyridine replaced the NH_3 . Addition of dibenzo[18]crown-6 to a solution of the ammonia complex indeed produced a marked shift of the two carbonyl bands to lower frequency, but the same experiment with the pyridine complex left the carbonyl bands unmoved, entirely consistent with strong hydrogen-bonding between the crown ether and the ammonia ligand. (I don't remember needing to ask anyone's permission to start this work: the Corporate Lab was an amazingly flexible research environment in those days.)

However, I was not able to isolate any specific adduct from the iron system, and so we next decided to look at an even simpler platinum complex $[(\text{Me}_3\text{P})\text{PtCl}_2(\text{NH}_3)]$, which had no charge and so no counterions to worry about. It was also more symmetrical than the iron complex and so potentially more likely to form crystals as an adduct. More persuasively still, Chatt and Venanzi had shown a quarter of a century earlier, using infrared methods, that strong intermolecular hydrogen bonds existed in the solid state between the ammonia and chloride ligands in this type of complex.

I was fortunate in being able to synthesize $[(\text{Me}_3\text{P})\text{PtCl}_2(\text{NH}_3)]$ very quickly by making use of ICI's unique store of research materials in the 'Chatt Archive.' This collection of transition metal complexes dated from the early 1950s, when Chatt had led a pioneering ICI research group, which effectively laid the foundations of modern coordination chemistry. One of the compounds we used even carried a penciled label 'L. Venanzi'! The archive, which then still survived in the basement of the Corporate Lab, had been moved from Chatt's former laboratory at The Frythe in Hertfordshire. This was a country house research establishment, which, before ICI set up its research center there after the war, had been home to the Technical Section of the Special Operations Executive (SOE). The Frythe was then known as 'Station IX' and specialized in the design of secret weapons for the French Resistance.

Our new platinum–ammonia complex quickly gave crystalline adducts with both [18]crown-6 and dibenzo[18]crown-6, and David Williams at Imperial College—subsequently, if not already by then, one of world's great crystallographers—was able to solve their structures and so prove conclusively the existence of multiple hydrogen bonds between the two components. I think I still have those crystals somewhere. I rescued them when David was clearing his lab a few years ago.

Fraser and I decided to try and extend these results by working with larger crown ethers, up to dibenzo[36]crown-12, and by using different co-ligands as ^1H NMR probes for adduct formation. We had worked out by this time that, when the crown ether interacted with coordinated ammonia, the aromatic rings of the crown produced significant ring-current shifts in resonances associated with adjacent ligands. This ring-current shift in fact proved an exquisitely sensitive tool for detecting such interactions and it has played an absolute key role in much subsequent research. We had also realized that the metal–ammonia–crown binding phenomenon was really a special case of 'second-sphere coordination', a concept going back to the earliest days of coordination chemistry.

Fraser's group in Sheffield (specifically I think John Wolstenholme) was co-opted to synthesize the larger crown ethers, and I designed a series of transition metal complexes containing both multiple ammonia ligands (to increase the number of potential hydrogen bonds) and also suitable probe ligands for ^1H NMR work. These ligands included 2,2'-bipyridyl, which I chose because I suspected—probably wrongly—that it could act as a π -acid ligand and so perhaps have the capacity to increase the hydrogen-bonding potential of any ammonia co-ligands. We despatched these compounds to Sheffield (I think Fraser must have taken them in his car) for NMR experiments with the larger dibenzo-crown ethers.

One day shortly afterward, coming back from lunch, I met Fraser who excitedly told me that one of the larger crown ethers, dibenzo[30]crown-10, when added to a solution of the cationic bipyridyl platinum complex $[\text{Pt}(\text{bipy})(\text{NH}_3)_2]^{2+}$, was producing absolutely enormous shifts (several parts per million) in the bipyridyl proton NMR resonances, implying the existence of an extremely strong set of interactions between the two components. Obviously, we needed to try and crystallize something to see what was going on, and after Fraser brought some dibenzo[30]crown-10 back from Sheffield I eventually managed to isolate some goodish crystals of a 1:1 adduct with $[\text{Pt}(\text{bipy})(\text{NH}_3)_2][\text{PF}_6]_2$. The crystals were sent off to David Williams, and a couple of weeks later he

produced the structure, which, sure enough, showed an awful lot of hydrogen bonds. To our astonishment, however, it also showed a perfect triple π -stack between the electron-deficient bipyridyl and the electron-rich aromatic rings of the crown ether. We only needed one look at the structure to see it was a charge-transfer complex! Regrettably, neither of us had noticed that that the adduct was a rather deeper shade of yellow than its parent platinum complex (to be fair, the difference was not that great) but we soon showed that this additional depth of color was due to a clearly identifiable charge-transfer band in the visible spectrum.

A year or so later, after we had filed patents and then published these results, it was time for Fraser to return to Sheffield. He gave a final seminar in the Corporate Lab conference room, setting out all the results achieved during his time there. At the end of the talk my one-time ICI boss, Eric Goodings (an outstandingly far-sighted chemist who had actually retired by then, but had come in specially for Fraser's talk) pointed out that ICI's bipyridyl-based herbicides paraquat and diquat were well known to form charge-transfer complexes, and why didn't we see if dibenzo[30]crown-10 would bind to these herbicides in the same 'sandwiching' sort of way as we had found for coordinated 2,2'-bipyridyl?

Since diquat was based on the same 2,2'-bipyridyl unit as our platinum complex, we thought Eric's idea well worth a try, and we quickly obtained a sample of diquat from ICI's Plant Protection Division at Jealott's Hill. I converted it to the hexafluorophosphate salt (more soluble in organic solvents than the commercial bromide salt), dissolved this salt up in dichloromethane and tried adding an equivalent of dibenzo[30]crown-10. The solution immediately changed from colorless to deep red. This observation strongly suggested formation of a charge-transfer complex, but I was unable to isolate anything interesting from this solution (the two components always tending, in my impatient hands, to crystallize separately). This was I think the last experimental work I did with Fraser, as by this time I was heavily involved in ICI's new research programme on high-temperature aromatic polymers and had little free time left for what was still a very academic topic. However, some months later John Maud—working with Fraser in Sheffield—did manage to isolate the dark red diquat-crown ether complex which I had seen briefly in solution. David Williams solved its structure (a particularly difficult crystallographic problem), and showed that it contained a triple π -stack entirely analogous to that of the platinum bipyridyl adduct: the resulting papers on diquat complexation carried, *inter alia*, the name of Eric Goodings!

Fraser, David Williams, and I pursued research on second-sphere coordination for a few more years, mainly through a collaborative Ph.D. studentship (Simon Doughty, who did a very nice job), and in 1986 we wrote a comprehensive review of our work in this field for *Angewandte Chemie*.

I spent some 20 years in high-performance polymer research, both industrial and academic, before returning almost accidentally a few years ago to work on the interactions of electronically complementary π -systems. This current work is now in the context of using tweezer-type aromatic molecules to 'read' polymer sequence information (perhaps ironically, at the University of Reading). Once again, a key result leading into this research—the complementary structure of a tweezer-macrocycle complex synthesized by my colleague Zhixue Zhu—emerged from the outstanding structural work of David Williams, by then Professor of Structural Chemistry at Imperial College.

Meanwhile, Fraser, still working closely with David, had managed to raise ARC funds to design and synthesize crown-ether-based receptors for paraquat, and from 1982 it was this extremely difficult and challenging work (in which I was not involved), which led Fraser, first in Sheffield, then in Birmingham, and now at UCLA, into the world of the mechanical bond and on to the now-famous catenanes, rotaxanes, molecular knots, rings, and functional

molecular machinery, which have come to define the Stoddart Era in Chemistry.

H.M. Colquhoun (5 June 2007)

If perchance the reader would like to refer to some of the publications from this 'blue period,' then with reference to the following topics, they are:

- (1) Key communications and papers on the second-sphere coordination of neutral and cationic transition metal amines by crown ethers.^{11–19}
- (2) Three reviews on second-sphere coordination of transition metal complexes by crown ethers.^{20–22}
- (3) Key communications and papers on the complexation of the diquat cation by dibenzo[3*n*]crown-*n* ethers.^{23–30}

Figure 2 portrays a collection of images that captures the significance of the research carried out in Runcorn, and then subsequently at Sheffield, that paved the way for the template-directed synthesis^{31,32} of donor–acceptor catenanes and rotaxanes.

On my return to Sheffield in 1981, I determined that we should also uncover a good crown ether receptor for the 'cousin' of diquat, namely paraquat, also known as methyl viologen. These two compounds have been sold by the ton in admixture by ICI now for half a century as a 'wipe-out' weedkiller. They are, however, not only bad news to plant life, they also interfere pretty dramatically with our own bodily functions if ingested either by accident or by design. And so, we reasoned that if we could contain paraquat in some kind of molecular container, then we might be within reach of an antidote for paraquat poisoning. This line of thought was, however, to lead us to grossly overdesign receptors for paraquat. We were to learn like many before us trying to do lock-and-key chemistry and, undoubtedly many after us, that months can be devoted to a multistep synthesis of a potential receptor for a particular substrate only to learn that the potential of the receptor to complex with the substrate is nil or pretty close to it. And so, after some years squandered trying to be too clever, we decided to let the molecules show us the way forward. We had noted that diquat's receptor, dibenzo[30]crown-10 (DB30C10), had also some affinity for paraquat and so we decided to make the slightest and most subtle of changes in an incremental manner to the constitution of DB30C10. This strategy proved to be very much more rewarding and led³³ quite quickly to the realization that a constitutional isomer, namely bisparaphenylene[34]crown-10 (BPP34C10), is an excellent binder of paraquat. BPP34C10 had been made by the Cram group³⁴ at UCLA several years previously for completely different reasons. It was the X-ray crystal superstructure, presented to us by David Williams, of the 1:1 complex formed between BPP34C10 and paraquat that got us all thinking outside the box. David's unique knack, as a structural chemist, of seeing the big picture by X-ray crystallography, and of illustrating an emerging phenomenon in plain and uncluttered pictures, was crucial at this juncture—as it was at so many others along the pathway of discovery and invention with regard to the mechanical bond in the final analysis. If anyone ever proved that old adage—'a picture is worth a thousand words'—David did so over and over again. The solid-state superstructure revealed that the rigid paraquat dication is threaded centrosymmetrically through the middle of the BPP34C10 ring in the crystalline 1:1 complex (Fig. 3a). A mere glance at the picture that David laid before us told us that the mechanical bond was already in the making. Subsequently, David and I were to refer to this complex as an example of a [2]pseudorotaxane³⁵ after it and its analogues were found to serve as templates in the synthesis of rotaxanes—and also of catenanes.^{36,37}

Much of the remainder of 1980s was spent trying to gain a better understanding of the nature of the interactions between π -donors

and π -acceptors so that we could employ their molecular recognition in self-assembly processes and ultimately in the template-directed syntheses^{31,32} of catenanes and rotaxanes. With paraquat and BPP34C10, we had established that we could thread a π -acceptor through a ring containing two π -donating units.

Our next task was to reverse this recognition motif and show that we could locate a π -donor inside rings containing two π -accepting units—for example, two bipyridinium units. Hence, we targeted cylcobis(paraquat-*p*-phenylene) (CBPQT⁴⁺), shown in Figure 3b, as a π -accepting receptor. We had little or no experience in the handling of organic salts and so we were on a steep learning curve for some time. Postgraduate student Mark Reddington eventually isolated and characterized this tetracationic cyclophane after a two-step synthesis, starting from 4,4'-bipyridine and 1,4-bis(bromomethyl)benzene. The very best yield he could obtain in the ring closure step, however, was 12%. When we submitted two communications to *Angewandte Chemie* in 1988, we were to find out quite quickly that Siegfried Hünig at the University of Würzburg had also synthesized the same tetracationic cyclophane for reasons that were rather different from ours. Subsequently, he and I became very good friends and years later in 2001 my wife and I were hosted in style in Würzburg by the entire Hünig family on the occasion of his 80th birthday.

The publication of our two communications^{38,39} alongside one⁴⁰ from the Hünig group was to be something of a watershed for me as

it marked our first use of color in primary papers, the only previous time being in our review²⁰ in 1986 on second-sphere coordination. We settled on the use of pillar-box red for π -donors and royal blue for π -acceptors. As a result CBPQT⁴⁺ became known as the little blue box and gained considerable notoriety as a promiscuous receptor for a whole range of π -donors. Although we met some criticism for our use of color (and cartoons), our critics had to concede ultimately that we used both in a consistent manner—and have done so to this day! Mark went on to show that the yield of the little blue box from its precursors could be enhanced three-fold by tagging diethyleneglycol chains onto the two phenolic hydroxyl groups on hydroquinone and used this compound, after methylation of its primary hydroxyl groups, as a template for the reaction. Nowadays, the use of a better template, in which a 1,5-dioxynaphthalene ring system replaces the 1,4-dioxybenzene unit, in addition to employing an ultra high-pressure reactor (the Menschutkin reaction undergoes a negative change in its molar volume at the transition state), has raised⁴¹ the yield of the little blue box to being near quantitative after a liquid–liquid extraction to remove (and recycle) the template.

Although the next step—that is, using BPP34C10 as the template in a reaction to produce the CBPQT⁴⁺ ring and having the π -accepting tetracationic cyclophane interlock mechanically with the π -donating crown ether—was obvious, it was several months before this historical reaction was performed in our laboratories.

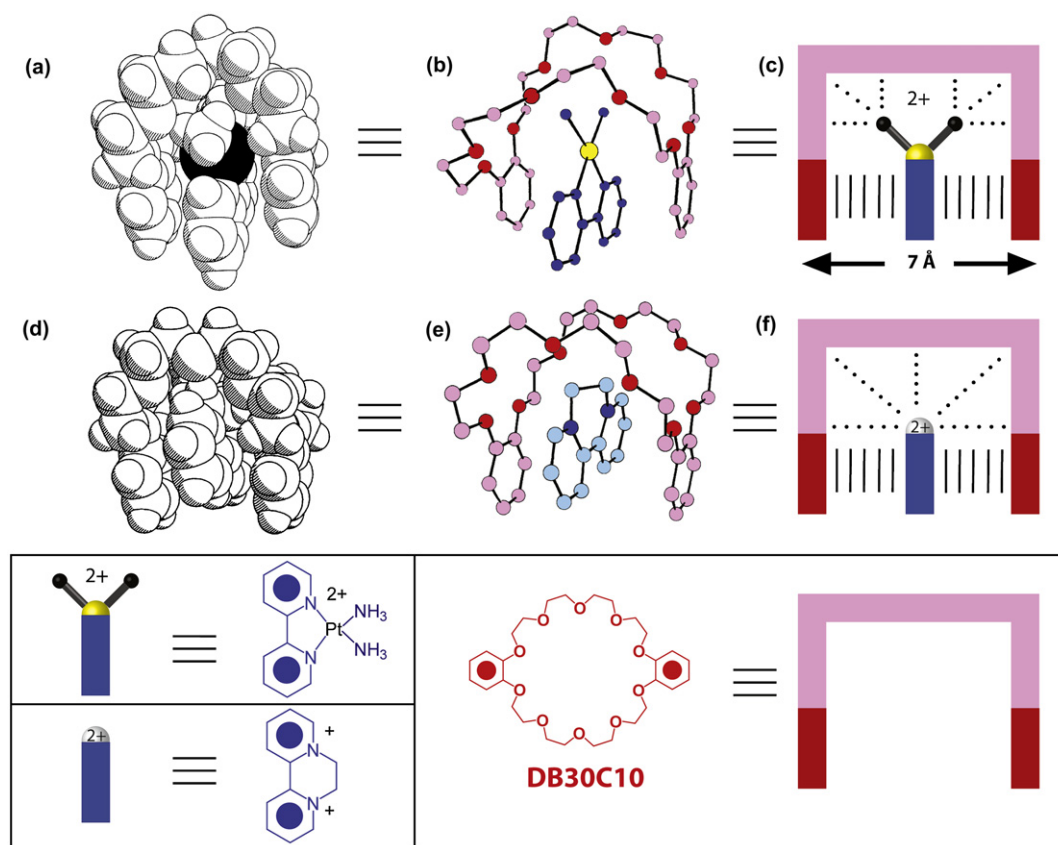


Figure 2. Space-filling (a) and ball-and-stick (b) representations of the 1:1 adduct formed between $[\text{Pt}(\text{bipy})(\text{NH}_3)_2]^{2+}$ and DB30C10 in the solid state. (c) A graphical representation of the 1:1 adduct showing the $[\text{N}-\text{H}\cdots\text{O}]$ hydrogen bonds and pole-dipole interactions between the dicationic transition metal ammine and some of the oxygen atoms residing in the polyether loops of DB30C10. The π -donating catechol units are shown in red and the π -accepting bipyridyl ligand in blue. The donor-acceptor interactions are indicated by parallel vertical lines. Space-filling (d) and ball-and-stick (e) representations of the 1:1 complex formed between diquat and DB30C10. (f) A graphical representation of the 1:1 complex showing $[\text{C}-\text{H}\cdots\text{O}]$ and pole-dipole interactions between the diquat dication and some of the oxygen atoms residing in the polyether loops of DB30C10. The π -donating catechol units are shown in red and the π -accepting bipyridinium unit in blue. The donor-acceptor interactions (3.5 Å separations, plane-to-plane) are indicated by parallel vertical lines. The progression in terms of molecular recognition and self-assembly from the 1:1 adduct to the 1:1 complex is an example of the kind of incremental development that makes for sure and steady progress in science. It is, by far, the most prevalent manner in which much chemical science moves forward into the unknown. Doing chemistry by analogy in increments can be extremely productive and immensely rewarding.

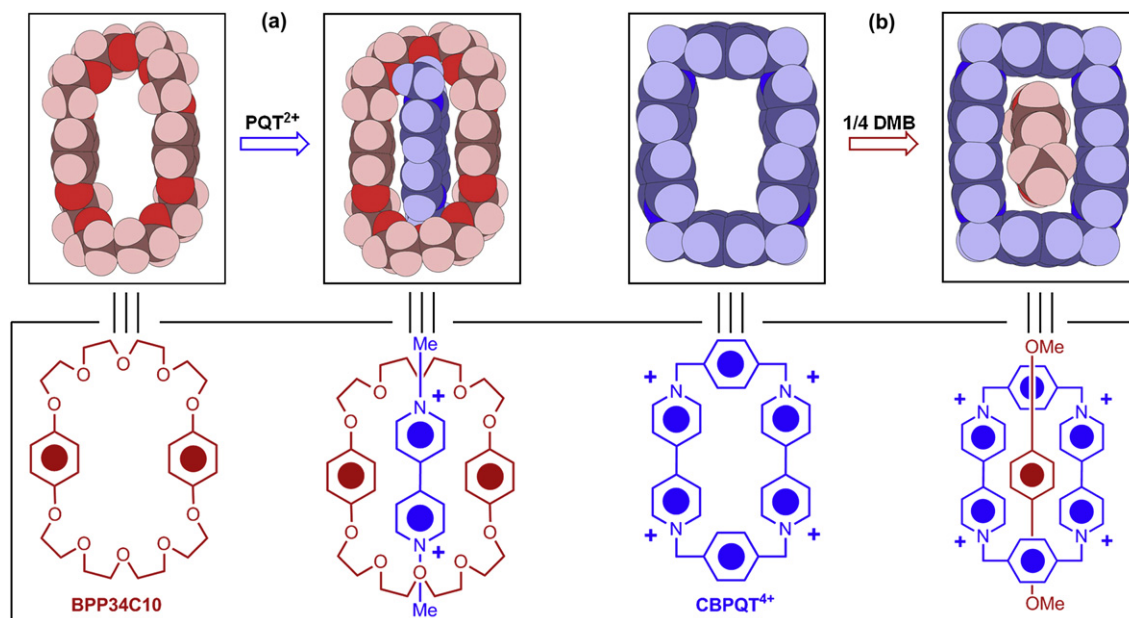


Figure 3. A comparison of the space-filling representations of two free receptors (BPP34C10 and CBPQT⁴⁺) and their 1:1 complexes obtained with paraquat and 1,4-dimethoxybenzene (1/4DMB), respectively, in the solid state by X-ray crystallography. Their constitutions are related to their structural formulas in the box below. The pre-organization of BPP34C10 and CBPQT⁴⁺ is reflected in the fact that these receptors bind paraquat and 1/4DMB, respectively, rather well in solution. Although the binding of the paraquat dication by BPP34C10 involves π - π stacking between the π -electron-deficient bipyridinium dication and the two hydroquinone rings of the crown ether, most of the energy of complexation comes from [C-H...O] interactions involving the methyl groups and two of the hydrogen atoms on the pyridinium rings α to the nitrogen atoms carrying formally one positive charge each, as well as pole-dipole interactions. The sources of binding between the tetracationic cyclophane (CBPQT⁴⁺) and 1/4DMB are (i) aromatic (face-to-face) interactions between the π -electron-rich hydroquinone rings and the two bipyridinium units and (ii) aromatic (edge-to-face) interactions between two of the hydrogen atoms on the hydroquinone ring in 1/4DMB and the faces of the bridging paraphenylene rings in CBPQT⁴⁺. This donor-acceptor complex is DNA-like (n.b., π - π stacking interactions that are also prevalent in DNA) in the horizontal direction and protein-like (n.b., there are edge-to-face [C-H... π] interactions reminiscent of those found in proteins, which are rich in aromatic amino acids, e.g., Phe, Tyr, Trp) in the vertical one. Both 1:1 complexes, as represented by the space-filling diagrams, are just more than a nanometer tall and just less than a nanometer wide.

During the summer of 1988, Mark was insistent that he should characterize his hard-won receptor rather fully, if not too fully for my liking! Finally, an opportunity to attempt the reaction came our way in the autumn with the arrival of Cristina Vicent from Madrid on a three-month postgraduate fellowship. Mark agreed that she, under the guidance of postdoctoral fellow Neil Spencer, should try to form the CBPQT⁴⁺ ring starting from its precursors in the presence of 3 equiv of BPP34C10 as the template. We were awestruck as this reaction changed color and produced a precipitate within minutes in front of our very eyes. This template-directed synthesis (Fig. 4) proceeded with the remarkable yield of 70% under kinetic control. Although the mass and ¹H NMR spectra left us in no doubt that we had made a [2]catenane, the crystal structure (Fig. 5a), obtained virtually overnight by David Williams, gave us that crucial picture, which sealed the outcome and was to become key in keeping the skeptics at bay. One reviewer of the subsequent communication that was published⁴² in *Angewandte Chemie* in 1989, while accepting the evidence for catenation in the solid state, was less convinced by our solution-state evidence!! With this first synthesis of a degenerate [2]catenane behind us, I knew that it was only going to be a matter of time before we had a non-degenerate, potentially switchable [2]catenane in our hands. It was my realization that function is being progressively built into this donor-acceptor catenane during its template-directed synthesis that got me really excited. Figure 6 outlines, in qualitative terms, how covalent bond formation activates the emergence of noncovalent bonding interactions and so progressively ramps up the molecular recognition—the intramolecular forces, if you like—inside this programmed molecule. For it is clear, from spectroscopy and electrochemistry performed in solution, as well as from X-ray crystallography in the solid state, that in the final product—the [2]catenane—the weak interactions which have been accumulated in an incremental way during the synthesis ‘live on’ inside the

molecule afterward. It seems to me that this close coupling of function to the templated synthesis of mechanically interlocked molecules has already acquired sufficient promise to have potential applicability to other areas of chemistry.

With this entry into the chemistry of the mechanical bond under our belts, I challenged every member of my research group to come up with 39 different ideas, emphasizing that we were standing at the entrance to a ‘goldmine’. One of these ideas was simply to take CBPQT⁴⁺ and BPP34C10 in admixture and see if we could perform the equivalent of conjurer’s magic ring trick—that is, to get them to interlock mechanically in a spontaneous manner under thermodynamic control. We had to wait, however, for the best part of two decades for this simple experiment to be performed at UCLA by postdoctoral researcher, Ognjen Miljanić. He reckoned that nucleophilic attack at one of the benzylic methylene groups in CBPQT⁴⁺ should be facile, given the fact that the positively charged bipyridinium unit is a good leaving group and that relief of considerable ring strain would accompany the cleavage of the C-N⁺ bond. He pointed out that the nucleophile should only be present in catalytic amounts in order to reduce the likelihood of multiple attacks on the CBPQT⁴⁺ ring. Under equilibrium conditions, the intermediate formed in step A in Figure 7 will become bound (step B) by BPP34C10, forming a 1:1 complex and setting up a situation where reverse nucleophilic attack by the pyridyl nitrogen lone pair can occur, expelling the original nucleophile as a leaving group and generating (step C) the [2]catenane. Since the entire process would occur under thermodynamic control, the [2]catenane should be formed in good yield because of the inherently higher stability of the mechanically interlocked structure relative to the individual structures of the collective noncatenated rings. Ognjen set about testing this hypothesis with a number of different nucleophilic reagents until he discovered that tetrabutylammonium iodide (TBAI) is the perfect catalyst for the job in

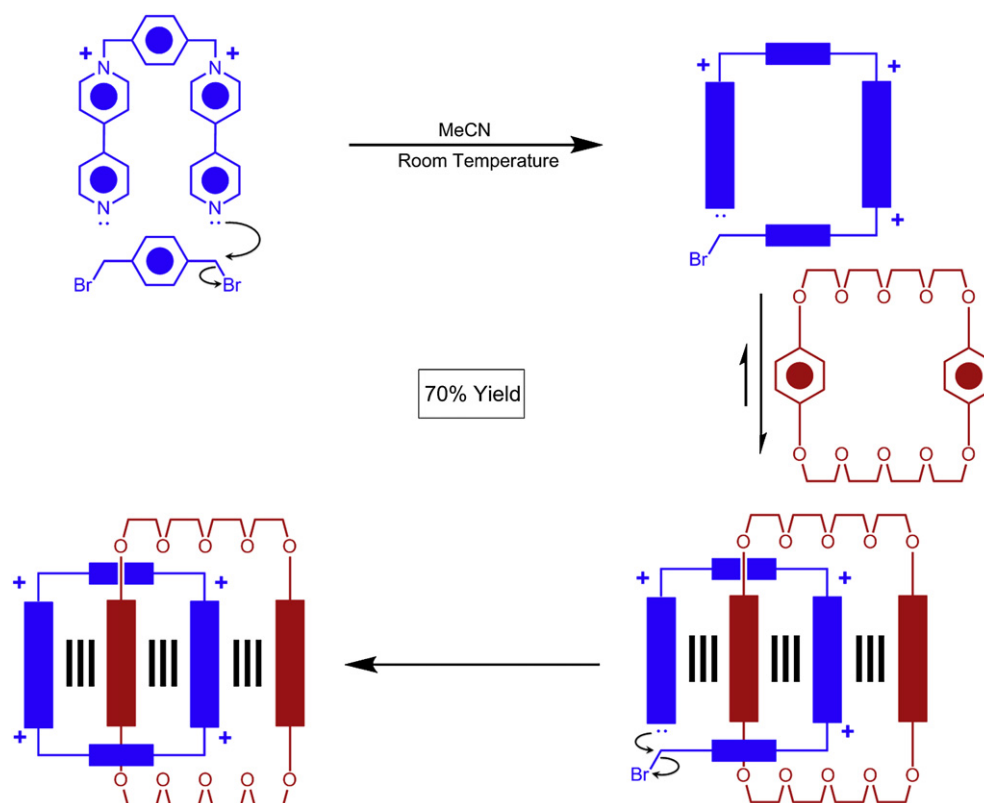


Figure 4. The template-directed synthesis of the first donor–acceptor [2]catenane under kinetic control in acetonitrile at room temperature. BPP34C10, which is present in excess (3 equiv), acts as the template for the stepwise formation of the mechanically interlocked tetracationic cyclophane. This [2]catenane, which is isolated as a crystalline salt in 70% yield after treatment with aqueous ammonium hexafluorophosphate, occupies roughly one cubic nanometer.

acetonitrile. Performing the reaction⁴³ with an excess of BPP34C10 led to a 100% conversion of the [2]catenane as estimated by ¹H NMR spectroscopy. Dr. Miljanić and graduate student Kaushik Patel have subsequently shown⁴⁴ that the reaction is of general scope and

proceeds in high yields without giving rise to by-products. It strikes me that despite its long history, nucleophilic substitution has been underutilized as a thermodynamically controlled catalytic reaction. In fact I could go further and point out that there is a dearth of

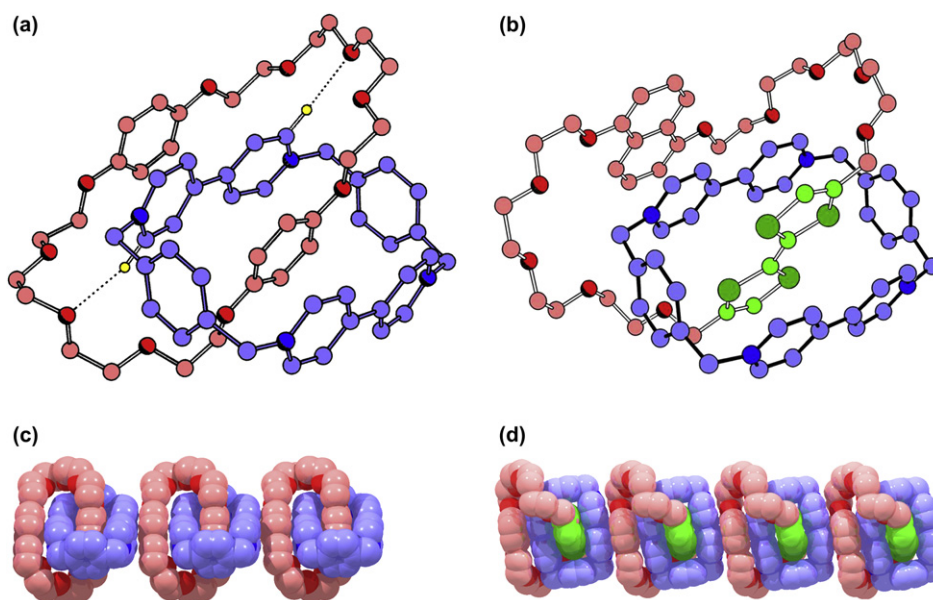


Figure 5. The solid-state structures of (a) the degenerate donor–acceptor [2]catenane and (b) the switchable [2]catenane. Note the presence of the π – π stacking interactions in both [2]catenanes between aromatic donors and acceptors with planes that are parallel, one with the other and separated by 3.5 Å. In the π – π stacking direction, the molecules are just over a nanometer wide. In the degenerate [2]catenane, note the presence of both [C–H···O] and [C–H··· π] interactions. The switchable [2]catenane occupies roughly one cubic nanometer. The fact that the packing of (c) the degenerate and (d) switchable [2]catenanes in the solid state constitutes, in both cases, an extended donor–acceptor stack encouraged us to use the Langmuir–Blodgett technique to fabricate memory devices.

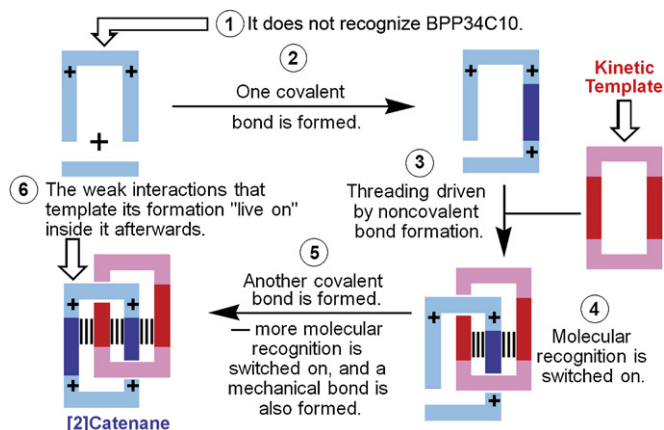


Figure 6. A blow-by-blow summary (1–6), using a graphical display, of the formation of the first donor–acceptor [2]catenane under kinetic control. It represents a fascinating interplay between covalent bond formation, progressively switching on non-covalent bonding interactions until, in the final step, a covalent and a mechanical bond are made simultaneously. The fact that the noncovalent bonding that is acquired during the template-directed synthesis ‘lives on’ in the [2]catenane afterward renders it a prototype for the designing and producing of two-state (bistable) and four-state molecular switches.

reactions that proceed under thermodynamic control, with or without a catalyst being present. What I often refer to as billiard ball chemistry still dominates the conceptual aspects and practical outcomes of organic synthesis, particularly in relation to natural products. Perhaps it has to be that way but perhaps not? Under kinetic control, you can usually get what you want, but you are nearly always lumbered with by-products, which have to be removed somehow from the reaction mixture. There is no going back, whereas under thermodynamic control⁴⁵ going back as well as forward is happening all the time. In terms of chemical synthesis, it is somewhat counterintuitive that, as a number of covalent bonds are being made, almost just as many are being broken! The mental reasoning when conducting synthesis under kinetic control—reactions routinely occur with acceptable conversions in a matter of hours—is very different from that required when performing reactions under thermodynamic control where patience is often a virtue—it might take a month or more for a reaction to come to equilibrium yet deliver a near-quantitative yield of a close-to-pure product.

The community in the early 1990s was, to some extent, lukewarm about our new mechanically interlocked compounds.⁴⁶ Did they really exist? If they did, were they good for anything? I determined that both questions would be answered in the affirmative. While the second question was to take another decade for us to address, the first one was easier to tackle with the wizardry of David Williams, producing a continued stream of solid-state structures of catenanes—and the occasional rotaxane. In the case of the catenanes, we resolved to show that the template-directed synthetic protocol^{31,32,47} was capable of delivering higher order catenanes with relative ease. In Birmingham, postdoctoral fellow David Amabilino was to take up this challenge, amidst many more, with a zeal that was breathtaking to behold. Working closely with another two postdoctoral researchers, Anatoly Reder and Ju-Young Lee, he brought into being a [5]catenane^{48,49} (Olympiadane) in 1994 and a branched [7]catenane^{49,50} in 1997. The solving of the crystal structures was a tour de force on both occasions by David Williams. Given the enormous experimental difficulties that are associated with the handling of single crystals, highly solvated with volatile organic solvents and containing 12 and 20 disordered counterions (PF_6^-), I am convinced that no one else in the world at that particular time, other than David, could have cracked these two problems (Fig. 8) in structural chemistry. While his contribution to establish the chemistry of the mechanical bond has been monumental and leaves me and others greatly enriched, the extent to which his life-long institution and his native country have failed to recognize his contributions leaves me saddened. Being of an optimistic disposition, however, I live in hope that I will not have to take this travesty of professional justice to my grave.

Before we left Sheffield for the University of Birmingham in 1990, visiting researcher Pier-Lucio Anelli had made our first degenerate [2]rotaxane using a very similar templated-directed protocol, which afforded the first degenerate [2]catenane. Dynamic ^1H NMR spectroscopy revealed (Fig. 9) that the CBPQT^{4+} ring moves back and forth between the two hydroquinone stations like a shuttle round about 500 times per second at room temperature. As a consequence, I found myself calling the compound a molecular shuttle. The following conclusions were drawn at the end of a communication⁵¹ employing the title ‘A Molecular Shuttle’ in the *Journal of the American Chemical Society* in 1991.

The opportunity now exists to desymmetrize the molecular shuttle by inserting nonidentical ‘stations’ along the polyether ‘thread’ in

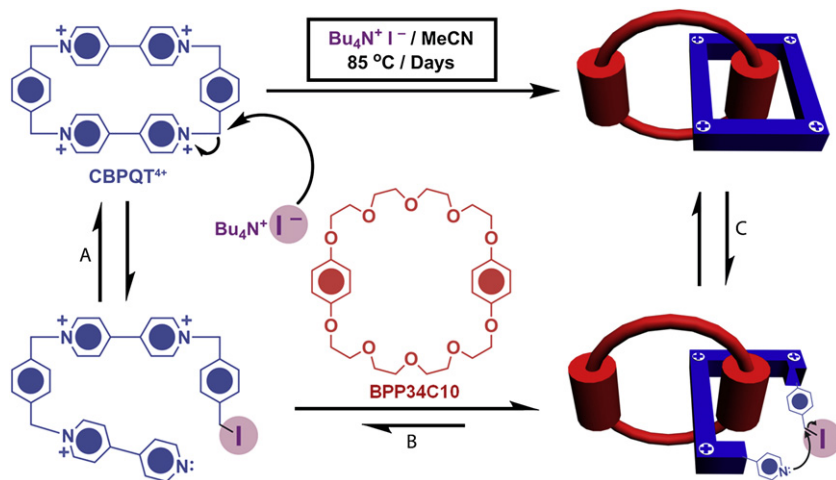


Figure 7. A proposed mechanism of [2]catenane formation involving dynamic nucleophilic substitution. Step A: nucleophilic opening of the CBPQT^{4+} ring by iodide ion. Step B: threading of the π -acceptor through the BPP34C10 ring. Step C: nucleophilic closure to give the tetracationic cyclophane around the crown ether to form the mechanically interlocked [2]catenane. Note that the iodide ion, which is consumed in the first step (A), is regenerated in the third step (C), that is, it is a catalyst. Since the reaction occurs under thermodynamic control, it is important to keep every component soluble in acetonitrile, hence the presence of the Bu_4N^+ cation. KI does not work.

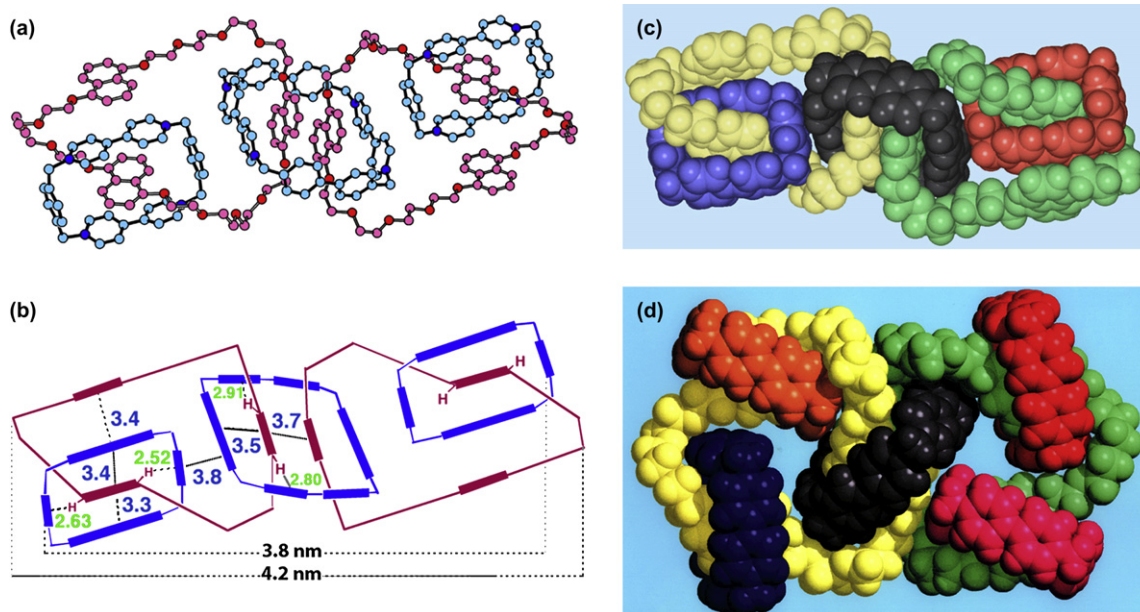


Figure 8. The ball-and-stick (a), skeleton (b), and space-filling (c) representations of a donor-acceptor [5]catenane, which has been called Olympiadane. The $[\pi \cdots \pi]$ face-to-face (blue) and $[H \cdots \pi]$ edge-to-face (green) distances are shown on the skeleton representation. Note that Olympiadane is centrosymmetric. Not so the branched [7]catenane, the space-filling representation of which is shown in (d).

such a manner that these different 'stations' can be addressed selectively by chemical, electrochemical, or photochemical means and so provide a mechanism to drive the 'bead' to and fro between 'stations' along the 'thread'. Insofar as it becomes possible to control the movement of one molecular component with respect to the other in a [2]rotaxane, the technology for building 'molecular machines' will emerge.

The molecular shuttle described in this communication is the prototype for the construction of more intricate molecular assemblies where the components will be designed to receive, store, transfer, and transmit information in a highly controllable manner,

following their spontaneous self-assembly at the supramolecular level. Increasingly, we can look forward to a 'bottom up' approach to nanotechnology, which is targeted toward the development of molecular-scale information processing systems.

For the next three years, in our idyllic new surroundings on top of the Haworth Building on the campus of the University of Birmingham, a small team within my research group worked extremely hard to make controllable molecular shuttles. A trio of back-to-back communications^{52–54} on this topic in *Synlett* bears witness to the fact that the task was far from an easy one.

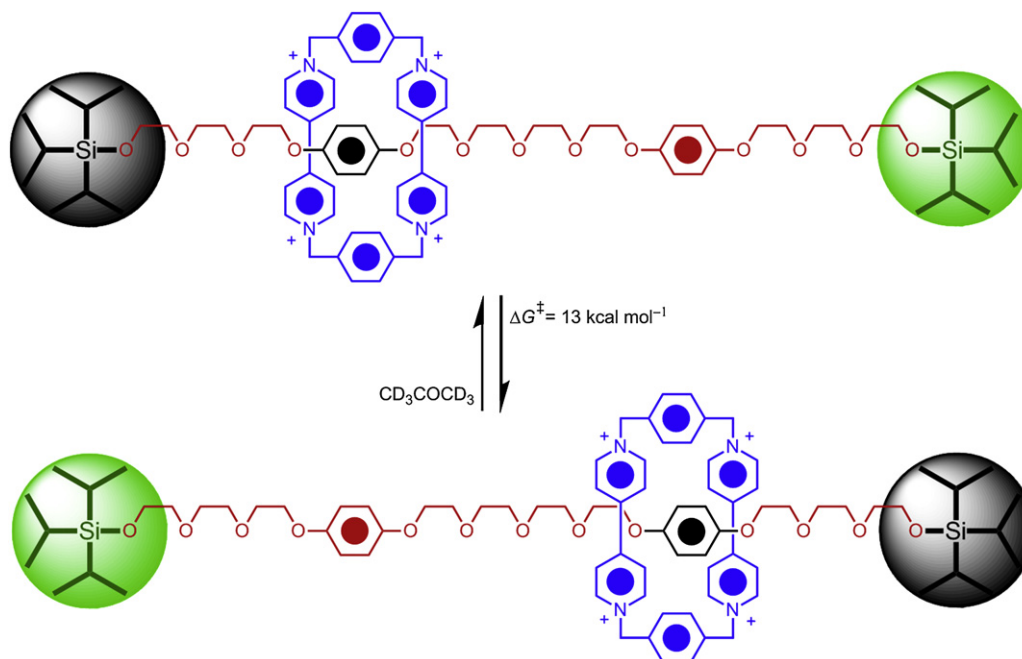


Figure 9. A degenerate donor-acceptor [2]rotaxane for which the phrase 'molecular shuttle' was coined in 1991. The CBPQT⁴⁺ ring darts back and forth between the two hydroquinone stations at a rate of around 500 times per second at room temperature. The triisopropylsilyl end groups act as stoppers, preventing the CBPQT⁴⁺ ring departing from the dumbbell component. This molecular shuttle can be regarded as the prototype for the construction of molecular switches and machines based on molecules with mechanical bonds.

Nonetheless, postdoctoral researcher Richard Bissell stayed the course and eventually went off to the University of Miami for a few months to make the first chemically and electrochemically switchable molecular shuttle in collaboration with Professor Angel Kaifer. The reason for Richard crossing the Pond lay in the fact that laboratory safety rules and regulations in the United Kingdom relating to the use of chemicals prevented Richard from using benzidine—a well-known carcinogen—in our Birmingham research laboratories. In Miami, Richard replaced one of the two hydroquinone rings in the dumbbell component of the degenerate molecular shuttle with a benzidine unit and the other one with a biphenol residue. The outcome (Fig. 10) was a [2]rotaxane in which the CBPQT^{4+} spends 84% of its time on the benzidine unit and 16% on the biphenol residue at equilibrium at room temperature in acetonitrile. This disparity in the populations of the two translational isomers was good enough to allow us to induce the movement of the CBPQT^{4+} ring in the major isomer away from the benzidine unit, either by oxidizing it to a radical cation electrochemically or by protonating the two nitrogen atoms of the benzidine unit to give a dication. Either way, the presence of the positive charge on the benzidine unit is sufficient to coax the CBPQT^{4+} ring to move to the biphenol residue. Both chemical (acid/base) and electrochemical (redox) processes are reversible. The results are summarized in an article⁵⁵ published in *Nature* in 1994.

What I find really attractive about the donor–acceptor catenanes and rotaxanes is that they allow us to work with molecular compounds,^{56–60} which—since they have been obtained using template-directed protocols^{31,32,47} that rely on noncovalent bonding—have many weak intramolecular interactions that can then be modified and mediated in so many different ways. One has the best of both worlds—namely, a world reminiscent of the supramolecular one localized inside a molecular world. Since the weak noncovalent bonding interactions that orchestrate the emergence of the mechanical bond in molecules live on between the components of the molecules afterward, the same molecules have been programmed during the discrete steps in their synthesis with more than sufficient information to revisit them in a mechanical context. Thus we can induce their components to move relative to each other either in a linear fashion—e.g., the ring component shuttling back and forth along the rod section of the dumbbell component in a bistable [2]rotaxane—or in a rotary manner—e.g., one asymmetrically constituted ring in a [2]catenane circumrotating through the cavity of the other symmetrically constituted ring. So far, the best example^{61,62} of a bistable [2]catenane was designed and synthesized by postdoctoral researcher Gunter Mattersteig. He replaced one of the two hydroquinone rings in the BPP34C10 component of the original [2]catenane (Fig. 5a) with a tetrathiafulvalene (TTF) unit and the other with a 1,5-dioxynaphthalene

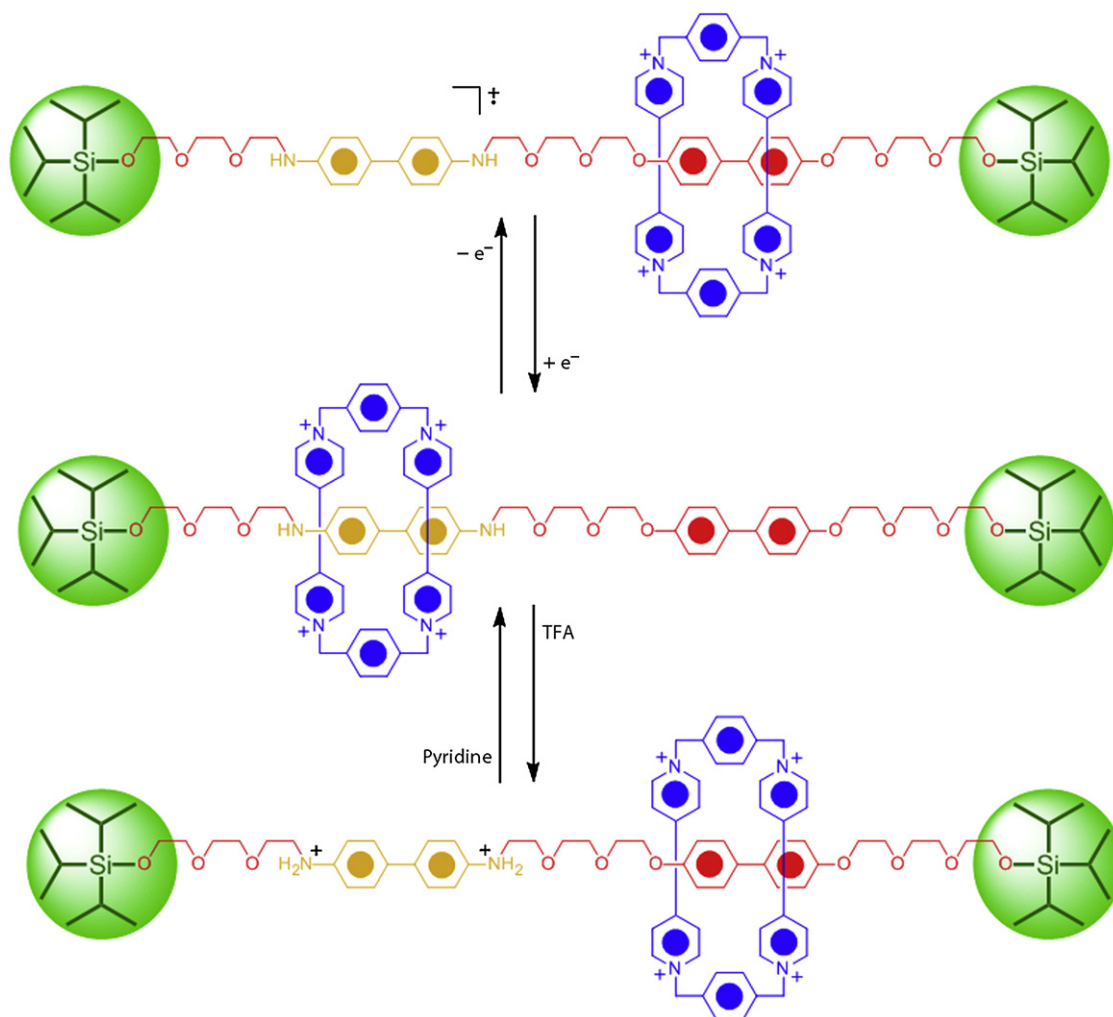


Figure 10. The first switchable donor–acceptor [2]rotaxane in action. At equilibrium in acetonitrile, both stations are occupied by the CBPQT^{4+} ring, the benzidine one (84%) more so than the biphenol one (16%). Electrochemical oxidation (top) of the major translational isomer results in movement of the ring so that it resides preferentially around the biphenol station. Protonation of the benzidine station also results in the movement of the CBPQT^{4+} ring.

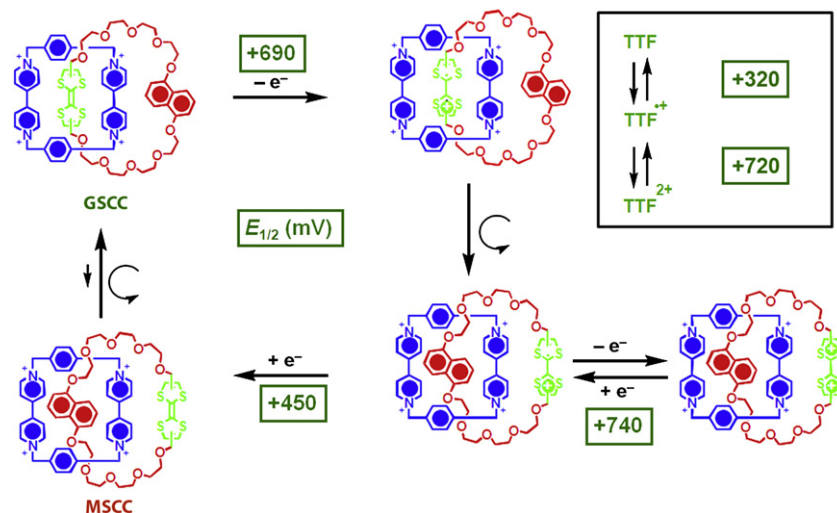


Figure 11. A switchable donor–acceptor [2]catenane wherein tetrathiafulvalene (TTF) is the redox-active unit being put through its paces electrochemically. The fact that the first oxidation potential of the TTF unit in the [2]catenane is +690 mV compared with +320 mV for free TTF tells us that the TTF unit in the crown ether is ensconced in the middle of the CBPQT⁴⁺ ring. The fact that the second oxidation occurs at +740 V as opposed to +720 mV in the free TTF tells us that circumvention of the crown ether ring through the CBPQT⁴⁺ ring occurs after the first oxidation. Reduction of the TTF radical cation in the crown ether at +450 mV affords a translational isomer of the starting [2]catenane. This isomer is of considerably higher energy and so has been referred to as the metastable co-conformation (MSCC), as opposed to the ground state co-conformation (GSCC). In crossbar devices used to display molecular memory, the MSCC is more highly conducting than the GSCC. In the parlance of the binary code, the MSCC is equated with a ‘1’ and the GSCC with a ‘0’.

(DNP) ring system. The solid-state structure (Fig. 5b) of this bistable [2]catenane shows us exactly what we expected to observe—namely, the TTF unit inside the CBPQT⁴⁺ ring and the DNP ring system sitting outside and alongside one of the bipyridinium units of the CBPQT⁴⁺ ring. Gunter based his design of this bistable [2]catenane on an observation^{63,64} made by postgraduate student Douglas Philp that TTF, of all the many π -donors complexed by CBPQT⁴⁺, is by far the most tightly bound inside the cavity of the tetracationic cyclophane, and certainly very much more so than 1,5-dimethoxynaphthalene by close on two orders of magnitude with reference to stability constants in acetonitrile solution. Free TTF is easily oxidized (see inset in Fig. 11) to its dication in two steps via its radical cation: the process is completely reversible. In collaboration with Professors Vincenzo Balzani and Alberto Credi at the University of Bologna, Gunter went on to show⁶² that his bistable [2]catenane (Fig. 11) can be switched electrochemically between the ground state co-conformation (GSCC) and a metastable co-conformation (MSCC) in a fully reversible manner. This bistable [2]catenane was used subsequently in collaboration with Professor Heath at UCLA to construct a solid-state electronically reconfigurable switch in a simple crossbar device.⁶⁵

Before the bistable [2]catenane could be introduced into a crossbar device, we had to learn how to effect its transfer, using the Langmuir–Blodgett (LB) technique from a Langmuir monolayer. Postdoctoral researcher Francisco Raymo in collaboration with Masumi Asakawa in the Nanoarchitectonics Research Center in the National Institute of Advanced Industrial Science and Technology at Tsukuba in Japan showed that if the four PF₆⁻ counterions associated with the tetracationic bistable [2]catenane were exchanged for dimyristoylphosphatidyl anions, then stable molecular monolayers of the catenane could be obtained⁶⁶ at the air–water interface using a Langmuir trough. It was calculated that the area occupied by each molecule is just a little over one square nanometer, meaning that, at around one cubic nanometer in size, the bistable [2]catenane probably constitutes the smallest molecular switch operating in the world today. In a crossbar device, wires of 70 and 100 nm cross each other—producing a cross-sectional area of 5000–6000 molecules sandwiched in the form of a one-molecule thick film. The packing of the bistable [2]catenane molecules in the solid state (Fig. 5d) had suggested that we explore the use of the LB technique

for the fabrication of molecular switch tunnel junctions (MSTJs) in crossbar devices. So yet again it was David Williams who provided that all-important picture, which prompted us to take that leap of faith and use the LB technique to make MSTJs. Our initial expertise in the production of Langmuir monolayers had been gained when postgraduate student Jon Preece spent a period in the mid-1990s in the laboratory of Helmut Ringsdorf at the University of Mainz. Inspired also by David Williams’ solid-state superstructure (Fig. 5c) of the degenerate [2]catenane, he demonstrated that it formed⁶⁷ stable molecular monolayers at the air–water interface in a Langmuir trough when the supporting counterions were dimyristoylphosphatidyl anions. If we are going to become successful at making devices that function then we are going to have to recognize that chemical synthesis does not begin and end with the making and breaking of covalent bonds, or for that matter, mechanical bonds. We have to work out how to marshal collections of molecules using the concepts of molecular recognition and self-assembly.

The incentive to turn to bistable [2]rotaxanes—containing, as the stations for the CBPQT⁴⁺ ring, TTF and DNP units—grew out of a desire to increase the ON/OFF ratio in the MSTJs of crossbar devices. At UCLA, visiting graduate student Jan Jeppesen from Jan Becher’s group at the University of Southern Denmark in Odense designed and synthesized by templation a range of bistable [2]rotaxanes.^{68–71} Not only are these compounds capable of being switched (Fig. 12) in solution but they have also been shown to undergo switching in condensed phases. Thus, this bistable [2]rotaxane can be induced to switch (1) chemically in Langmuir films and on LB monolayers,⁷² (2) electrochemically in a highly viscous polymer matrix,⁷³ (3) electrochemically as a self-assembled monolayer (half device) on gold,⁷⁴ and (4) electronically within MSTJs.^{65,75,76} Not only has reversible, electronically driven switching been observed^{65,75} in the MSTJs incorporating a monolayer of bistable [2]catenane molecules sandwiched between a bottom polysilicon electrode and a top titanium/aluminum electrode, but a crossbar random-access memory circuit and a simple logic circuit have also been engineered⁷⁶ by members of Heath’s group using amphiphilic, bistable [2]rotaxanes. Using one of these rotaxanes (Fig. 13a), a 160,000-bit molecular electronic memory circuit has been fabricated⁷⁷ at a density of 100,000,000,000 bits per square

centimeter. This density of memory is roughly analogous to the density of a DRAM circuit projected to be available by 2020. The entire 160-kbit crossbar assembly is smaller than the cross-section of a white blood cell. Taken together, all these experiments carried out in condensed phases and in solid-state devices provide compelling evidence that the bistable, switchable [2]catenanes and [2]rotaxanes operate mechanically in soft-matter environments—and can even withstand harsh device-processing steps.⁷⁸

The field of molecular electronics had a rough ride around 2003 and we were, to some extent, caught up in the maelstrom. I have given my account elsewhere⁷⁹ of how some misunderstandings might have developed in relation to our research about this time. I am not going to repeat my analysis or arguments here. I would merely point out that while the controversy raged, we sought to broaden and quantify the scientific basis on which our contributions to molecular electronics rest today. We also brought more senior collaborators into our research program. And so it happened that, in close collaboration with graduate student David Steuerman in Jim Heath's group at CALTECH, and Professor Xiang Zhang's group at UCLA, postdoctoral scholars Hsian-Rong Tseng, Amar Flood, and Andrea Peters identified—by time-dependent cyclic voltammetry in solution at low temperatures⁸⁰ as well as in the polymer matrix⁷³ and in the half device self-assembled on gold⁷⁴—a metastable state (MSCC) for the molecular switches where the CBPQT⁴⁺ ring resides on the DNP unit. In MSTJs, this state is postulated^{65,75–77} to correspond to the closed or ON position (more conducting) of the switch. When the CBPQT⁴⁺ ring encircles the TTF unit (GSCC), the switch is in the open or OFF position (less conducting). First-principles calculations of the current/voltage responses, carried out⁸¹ in Professor Bill Goddard's group at CALTECH on model bistable rotaxane systems of the metastable (MSCC) and ground (GSCC) states, support their association with the ON and OFF positions, respectively, of the switch—and so lend support to the proposed switching mechanism^{65,75–77} in MSTJs. The metastable state of a switch in an MSTJ decays back to the ground state during a period of 10–60 min. If, however, the bipyridinium units in the CBPQT⁴⁺ ring are reduced from dications to cation radicals, then molecular recognition is greatly diminished and

switching becomes almost instantaneous. It transpires that the MSCC to GSCC relaxation times of the bistable molecules in solution are much shorter than they are in the polymer matrix and half device by an order of magnitude. Furthermore, the relaxation times in the MSTJs are longer than they are in the polymer matrix and half device by yet another order of magnitude. Expressed in terms of activation barriers, to get from the MSCC to the GSCC, a value of around 16 kcal mol^{−1} in solution climbs to around 18 kcal mol^{−1} in the polymer matrix and half device, and finally rises to around 22 kcal mol^{−1} in MSTJs.

In collaboration with Professor Jeff Zink at UCLA we began a few years ago to construct valves on the nanoscale level using the bistable [2]rotaxanes that have supported our molecular electronics program. Construction of such a device requires, in addition to nanovalves, mesoporous nanoparticles (100–500 nm in diameter) that are robust and amenable to surface functionalization. Mesoporous silica fits the bill. We have, using mesoporous silica nanoparticles adorned with a bistable donor–acceptor [2]rotaxane, demonstrated⁸² devices, which operate reversibly. The nanovalves, which can be turned ON and OFF by redox chemistry (Fig. 13b), trap and release small molecules in the maze of nanoscopic passages that pervade the mesoporous silica nanoparticles. This type of integrated device holds considerable promise^{83,84} in many areas where the delivery of small molecules, including drugs, needs to be controlled. This is another story, however, for another day.

With such a highly developed understanding of the workings of switchable donor–acceptor-based mechanically interlocked molecules and the successful incorporation of them into molecular electronic devices, our appetite to create larger and more complex molecular architectures—in which more than one ring may be incorporated—was whetted. Someone who was particularly inspired by this challenge was postdoctoral scholar William Dichtel. On coming to UCLA on a half-time appointment with Professor Jim Heath at CALTECH, he immediately applied himself to the task of developing strategies for forming mechanical bonds in a highly efficient manner through the stoppering of pseudorotaxanes. This particular template-directed protocol took advantage of the high

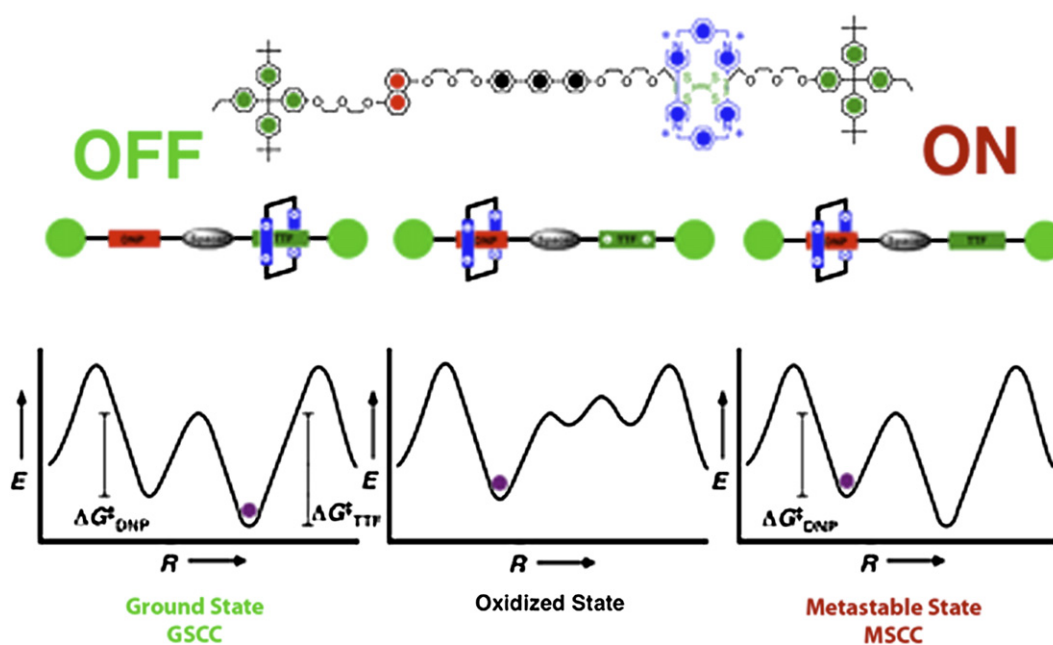


Figure 12. A switchable donor–acceptor [2]rotaxane showing the mechanism with reference to free energy profiles, and the mode of switching exhibited by the TTF-based system on oxidation. The GSCC starts off being around 3 kcal/mol more stable than the MSCC. Once again, in crossbar devices, amphiphilic bistable rotaxanes based on this constitution can be switched between GSCC (OFF state) and the MSCC (ON state).

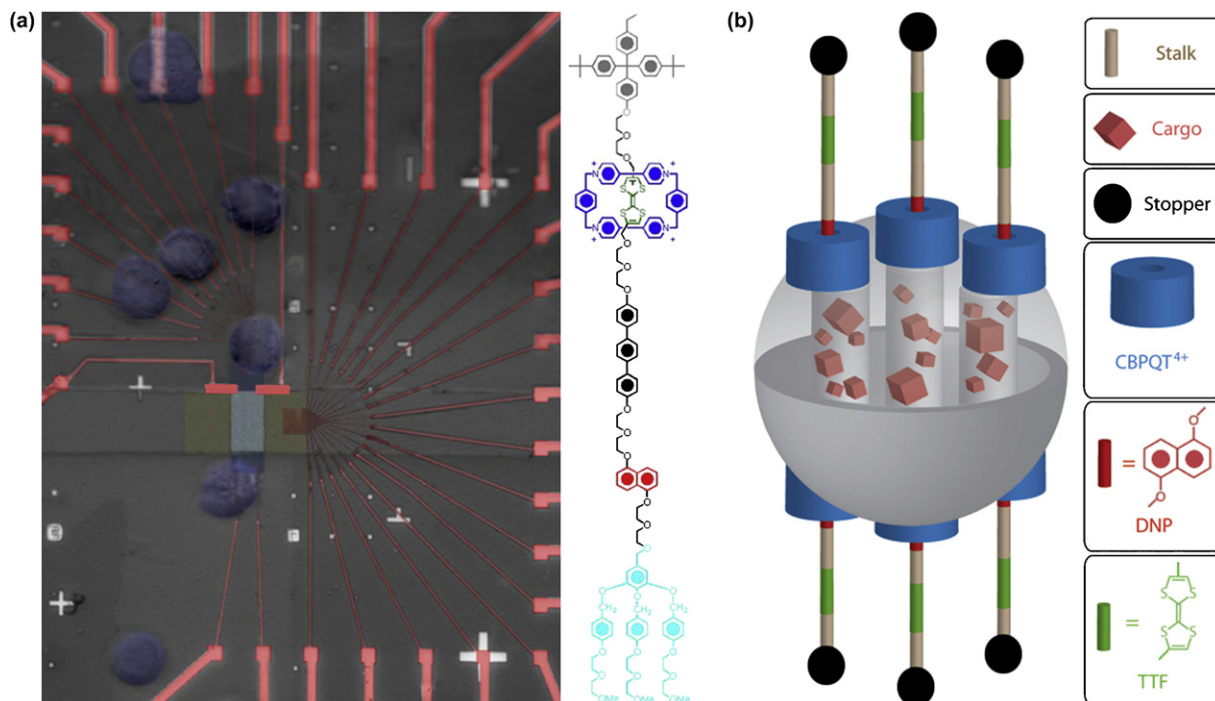


Figure 13. (a) A 160-kilobit crossbar molecular memory (orange in the middle of the picture) is smaller than the cross-sectional area of a white blood cell (purple). The structural formula of the amphiphilic bistable [2]rotaxane employed in the fabrication of this device is shown on the right. (b) A graphical representation of a mechanized nanoparticles (100–500 nm) composed of mesoporous glass covered with nanovalves based on a switchable donor–acceptor [2]rotaxane similar to that used in the work in molecular electronics. This device can be employed repeatedly to deliver small molecules (dyes, perfumes, scents, drugs, etc.) on command in prescribed environments.

stability constants of complexes formed between π -electron-rich DNP and TTF threads with the fully formed CBPQT⁴⁺ ring under ambient conditions—a twist on the tried and true template-directed strategy in which molecular recognition is turned on by sequential alkylation steps of the bipyridine units that ultimately form the CBPQT⁴⁺ ring. The key challenge to this threading-followed-by-stoppering strategy lies in the fact that the reaction conditions required to join the stoppers and the thread must be mild enough not to disrupt the binding of the ring and thread—or even worse—destroy the sensitive CBPQT⁴⁺ ring. Will found this special combination of conditions in the so-called ‘click’ reaction—that is, the Cu^I-catalyzed azide–alkyne cycloaddition,⁸⁵ recently popularized⁸⁶ by Professor Barry Sharpless. Will, with the help of many others, including postdoctoral scholar Ognjen Miljanić and graduate students Jason Spruell and Wenyu Zhang, addressed the challenge of utilizing this kinetically controlled reaction for novel rotaxane and catenane syntheses. Not only did the mild conditions and near-quantitative yields—afforded by this reaction—prove useful for rotaxane synthesis, but also the threading-followed-by-stoppering strategy could be employed⁸⁷ to stopper pseudorotaxanes containing not just one ring but also two, three, and even more rings. A further advantage of this synthetic approach is that simple starting materials can be made to converge upon much larger and functional mechanically interlocked molecules quite rapidly. One such example is that of the liquid-crystalline (LC) bistable [2]rotaxane (Fig. 14) synthesized⁸⁸ by postdoctoral scholar Ivan Aprahamian in collaboration with Professor Takashi Kato at the University of Tokyo. The synthesis of this bistable [2]rotaxane was centered around stoppering the general TTF/DNP diazido thread with two identical alkyne-containing mesogens. This compound lived up to our expectations and indeed formed LC domains—providing alignment and order to the bistable [2]rotaxanes in a higher order self-assembly process. We are now looking toward being able to switch these new LC phases electrochemically.

Looking back, it took us close on a decade to go from noting the ability of simple donor–acceptor π -systems to self-assemble under the influence of molecular recognition to employing this knowledge in the template-directed syntheses^{31,32,46} of the first donor–acceptor [2]catenane,⁴² [2]rotaxane, and molecular shuttle.⁵¹ In retrospect, the rate of progress would have been faster if we had known then what we know today about the driving forces behind the formation of the mechanical bond by templation. Tom Cottrell, my Professor of Physical Chemistry at Edinburgh, while I was an undergraduate student there, used to insist that a pretty good definition of the mathematical constant, π (≈ 3.14156), is the time it actually takes to complete a piece of research, compared with the amount of time it is anticipated that it might take at the onset. During the ensuing two decades, I often had to find refuge in Cottrell’s Law while learning about Moore’s Law—namely, that, in relation to computer hardware, the number of transistors that can be placed at little or no cost, within an integrated circuit is increasing exponentially, doubling approximately every two years, according to Gordon Moore, the co-founder of Intel. Why is there this difference between Cottrell’s Law and Moore’s Law? I suspect that it might be because the former relates to science whereas the latter is much more about the pace of technological change. I am in no doubt that the rate at which new agricultural machinery appeared on my father’s farm during 1950s and 1960s followed a pattern that is reminiscent of Moore’s Law.

Elsewhere,⁷⁹ and earlier in this article, I have related how dreams of nano Meccano drove our research program along according to Cottrell’s Law and how the beginnings of real nano Meccano were forged at UCLA in collaboration with Jim Heath. Jim is a remarkable scientist. As a graduate student in Rick Smalley’s laboratory in the mid-80s, he was probably the first person to set his eyes on the experimental evidence⁸⁹ for C₆₀ being a ‘remarkably stable cluster consisting of 60 carbon atoms.’ Prescient as that observation was to become in the fullness of time, I also find his achievements in taking bistable [2]catenanes and [2]rotaxanes

from our laboratory and getting them to perform as switches in MSTJs in a manner which has gone from one crossbar to 160,000 in a relatively short period of time, nothing short of breathtaking. In my mind, Jim is a gift to science in a manner that is not too dissimilar from the way in which David Williams was to my group and many others in the previous two decades. He has put the art and science of molecular electronics onto a completely new footing. Regrettably, in the scientific community, there are a few self-appointed gurus who, having done little themselves, find it difficult to give credit where credit is due. I am in no doubt that, with the passage of time, Jim's contributions to molecular electronics at the turn of the millennium will be seen to have been monumental. To date, his group is the only one that has taken molecules that switch in numerous condensed phases and not only introduced them into a single device setting but has also taken them to the level of integrated systems. I cannot help drawing a comparison between molecular electronics and a garden where grass grows (on lawns), flowers blossom (in the beds and borders), vegetables grow (in the kitchen garden), and trees produce fruit (in the orchard). In the context of this fable, Jim has already set foot in the orchard and he has long since moved on from picking the low hanging fruit.

My late wife Norma used to say to me that it is important for scientists to recognize when they have reached their 'sell-by date.' I hope I am able to identify when that time has come in my own case. My parents, in their profession as tenant farmers, certainly did after I vacated the farm. Eighteen months after my departure for Canada, a farm sale was held at Edgelaw and they migrated into Edinburgh to start up another small business in the service industry, one which my mother was able to run for many years after my father died in 1973. I was probably more crucial to the smooth running of the farm than I appreciated at the time. To my parents' everlasting credit, they never let on to me that I was indispensable! On reflection, this cutting of the umbilical cord was to repeat itself over and over again in my own professional life as I said my farewells to

hundreds of (post)graduate students and postdoctoral scholars. Time inexorably comes round for them to sever their day-to-day connections with the research group. When it does, I always try to follow my parents' example and let the youngsters leave the nest feeling good and with them and me on the best of terms.

The nature of the mechanical bond has now been revealed to all and sundry in chemistry, not just by our own efforts but also, in large measure, by those of Sauvage,^{90,91} Balzani,⁴⁶ Credi,⁹² Hunter,⁹³ Vögtle,⁹⁴ Leigh,⁹⁵ Sanders,⁹⁶ and many others. I have tried to capture the essence of the mechanical bond in the caption that accompanies the front cover of this issue of *Tetrahedron*: it reads—

The subtle interplay between covalent, noncovalent, and mechanical bond formation in the template-directed synthesis of a [2]catenane is reminiscent of the metal struts, nuts and bolts that are used in Meccano to build a car. Exquisite order emerges out of a toolbox of bits and pieces that are made to measure in a highly programmed manner.

The way in which covalent bond formation activates non-covalent bonding interactions by progressively switching on molecular recognition that leads to self-assembly by component building blocks as a prelude to the template-directed synthesis^{31,32,47} of mechanically interlocked molecules has introduced a level of integration into chemical synthesis that has not previously been attained jointly at the supramolecular and molecular levels. The grand challenge now is to carry this level of integration acquired during molecular synthesis beyond relatively small molecules into the realms of precisely functionalized extended molecular structures and superstructures that are capable of performing functions in a collective manner as the key sources of instruction, activation, and performance in multi-component integrated circuits and devices.

I must confess that I always feel uneasy when it comes to pontificating about the future. However, I believe I can proffer the

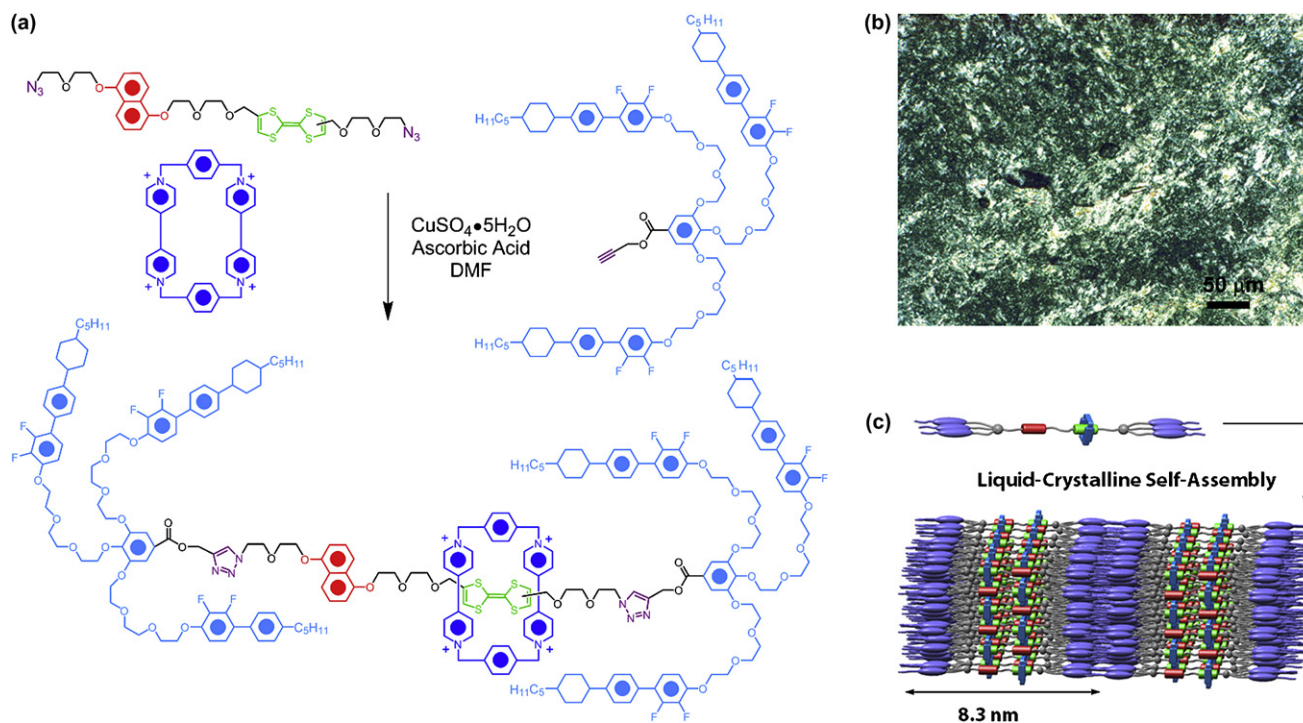


Figure 14. (a) An example of the use of 'click chemistry'—the copper(I)-catalyzed alkyne-azide (1,3-dipolar) cycloaddition—in the template-directed synthesis of a liquid-crystalline bistable [2]rotaxane under kinetic control. Given the fact that, with this particular synthesis, two cycloadditions have to occur along with the formation of a [2]pseudo-rotaxane, and the attachment of two gigantic stoppers, a 50% yield of the [2]rotaxane is more than adequate, a testament to the power of 'click chemistry'. (b) A polarized optical photomicrograph of the liquid-crystalline bistable [2]rotaxane. (c) A schematic illustration of the supramolecular organization of the liquid-crystalline [2]rotaxane into a smectic A phase.

claim, with good reason, that the stage is now set for us to move on from making molecular switches—which are already finding their way into working devices—to designing and constructing artificial molecular machinery.^{97–101} But making artificial molecular machines is not going to be enough by itself. We cannot leave the molecules—however, sophisticated they might be—to swim around aimlessly in solution. We have got to identify appropriate settings for them so that they can express their collective functions in the surroundings in which we live, work, and play. Artificial molecular machines are going to have to be located on surfaces and at interfaces, and perhaps also in three-dimensional space in a precise and periodic fashion. The conundrum facing the wider chemical community at present is to work out how to get from relatively small, yet highly programmable molecules to contraptions and gadgets that do something useful in the real world. In a nutshell, if unnatural product synthesis is to have a future above and beyond the mere intellectual satisfaction of making exotic compounds for their own sake, then we have got to discover how to build integrated systems where emergent behavior will most likely reveal a much higher level of complexity and purpose than we can even contemplate today.

Acknowledgements

During the course of telling this story I have gone out of my way to make mention by name of some of my key collaborators, postdoctoral fellows, and (post)graduate students. They played a major role in furthering my research program on donor–acceptor catenanes and rotaxanes, and were supported handsomely by many, many others whose names appear in the list of publications. Although I would argue that the human resource has been the most important by far in the furthering of my research program, we, as a research group, could not have functioned in the way we have without the generous financial support from institutions and funding agencies, both here in the US today, in previous times, and back in the UK. In more recent times, Birmingham University, UCLA and Northwestern University, along with the EPSRC, the BBSRC, the NSF, the NIH, the ONR, and DARPA have been particularly generous. Thank you all! At the end of the day, however, it is not the vast collection of papers we have published—some would say far too many—nor the hundreds of talks I have given all around the globe that have given me the most pleasure. For me, pleasure comes about when I look at past group members and witness them achieving on a grand scale. Nothing gives me greater satisfaction than following the careers—some of them stellar by any standards—of one-time postdoctoral scholars and (post)graduate students in private industry and government service, as well as in academia. Keep up the good work, lads and lassies!

My wife Norma was highly supportive of my research group and the young people who populated it during a 30-year period. Herself a BSc-chemist-turned-PhD-biochemist, she appreciated their needs just as much as mine. She played a key role at Sheffield, where she worked for years in a space no larger and considerably less appealing than a broom cupboard, and then in considerably more salubrious surroundings at Birmingham and finally at UCLA, solving their everyday problems without involving me most of the time. She also assumed the lion's share of the responsibility for raising our two daughters, Fiona and Alison, both of whom graduated with PhDs in Chemistry, Fiona from Imperial College London and Alison from Durham University, after obtaining a masters degree from Cambridge. Norma looked upon Fiona and Alison as her lasting legacy, referring to them in her latter years as 'my girls'. Since their mother's death I have come to appreciate just what a wonderful job she did in bringing them up for they are very much now 'my girls', lending their support to me and my research group on a frequent basis from afar. Thank you, girls!

When Norma and I arrived in Sheffield in January 1970 from Canada, we were befriended from day one by three superb postgraduate students—Dave (Brickwood), Dick (Taylor), and Steve (Potter)—doing their research in three different groups. It is as a result of one of those wonderful turns of fate that I have had the immense pleasure of working closely once again, for most of the past year, with Richard Taylor on this particular Tetrahedron Symposium-in-Print. From the outset of the exercise last summer, he has been supportive and accommodating throughout, firm and insistent that he get a manuscript out of me, extremely efficient and hard working in the soliciting and handling of 37 additional manuscripts submitted by collaborators, ex-associates, and friends, and courteous and diplomatic when my Celtic temperament reared its ugly head. Thanks to all of the 37 lead authors and their co-authors—and, on completion of a job extremely well done, congratulations, Richard!

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Unrefereed Publications

The list that follows relates to articles published in monographs and scientific journals, most of which were solicited and were not subjected to peer review.

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