

Autobiography chapter: Science

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My first attraction to science was to astronomy in the form of the articles on the subject in the Children's Encyclopaedia that we possessed – vintage about 1908. This interest started when I was about eight and flourished for about ten years, encouraged by Christmas presents of books on it from Uncle Wilfred, and by loans of a telescope – and subsequently the gift of two telescopes in 1936 – by Mr Craig our Methodist minister. During this time I also made a telescope and worked out for myself how to make an equatorial mounting for it – though it was too crude to be useful. I read all the books I could get hold of, observed the moon, Jupiter's satellites, a few double stars, and most of all sun-spots by the projection method. In 1938 I gave a lecture to the school Science Society on "Cosmology and Cosmogony"! And the same year I spread myself on an essay question in the general paper of my scholarship on "Large astronomical telescopes". This led to considerable discussion with the physics tutor at Magdalen at my interview and may have helped to get me my demyship. The ultimate thrill was to see Saturn through the telescope at the University Observatory in 1939-40. After that my own facilities were too inadequate to give much satisfaction. In about 1938-9 I had plans to grind a 6" mirror and build a reflector, but never solved the practical problems of where to do it, so astronomy faded to a background interest. I never considered attempting to make a career of it because I did not regard myself as sufficiently competent at maths to do the necessary physics, and in any case chemistry had become much more important to me.

Though I had no idea of it at the time, in retrospect I can see the first germ of my scientific career must have occurred also at about the age of eight. Mum had gone to church on a Sunday evening and I got Dad to read to me something (I know not from what book or journal) about why things hold together – the word "cohesion" occurred considerably in it. This was not explained either by the article or by Dad, but was invoked as the explanatory principle just by naming it. I found this unsatisfactory and unsatisfying and never forgot it; it must have remained to provide the hook to attach me to ideas of valency and crystal structure much later on. The next event was much more specific. It was my first "science" lesson in September 1931 when Mr Boak did a lecture-bench demonstration of the preparation of nitrogen from the air. This was followed the next week by the preparation of oxygen, and the week after of carbon dioxide. I was completely enthralled, and started doing experiments in the kitchen

with jam jars and lime water and candles in bottles. In November I used some birthday money to buy for 2/6 a small Kay's Chemistry Set containing about a dozen chemicals in little red tins, two test-tubes, litmus paper, and the like. Then at Christmas, without knowing anything about all this, Uncle Wilfred and Auntie Anne gave me a much bigger chemistry set in a lovely blue box. This had its chemicals in glass tubes standing in a kind of test-tube rack, and I think must have included a bunsen burner. The next advance came in the Easter holidays in 1932. Mum and Dad and I went to stay for a weekend with an old friend of Dad's, Mr Winterton, at Hatch End. We returned in the Monday evening and as Mum regarded the 6:00 train non-stop to Wilmslow as too fast to be safe because it averaged just over 60 mph, we came on the 6:10 and changed at Crewe. As a result we happened to meet and travel with a Dr Harding, a biochemist, who had at one time lived at Alderley. He asked me what I was interested in and I said "chemistry"; he then asked "do you do it at school" and I said "no, I do it at home". He had had a similar interest from his early youth, and he gave me the address of Griffin and Tatlock Ltd and told me to write to them for a catalogue of apparatus – they unlike another firm had been willing to supply him as a boy and so he had always traded with them since. So I duly wrote off and received a handsome hard-backed catalogue which I used to read with great enthusiasm, and I eventually had a couple of orders delivered from them in beautiful flasks and beakers and things (though I had bought some of these previously at Wiles' shop in Manchester). I also found there was a chemist's shop in Wilmslow where I could buy test-tubes and glass and rubber tubing, and I bought a variety of chemicals at chemist's shops, including such things as sulphuric and nitric acids and potassium chlorate, which I am sure they would not sell to a child now. I only once had an accident, when I very foolishly sealed the cork into a test-tube with sealing wax (because it was loose) and then heated the tube containing potassium nitrate to prepare oxygen. The sealing wax melted, ran down into the molten nitrate, and exploded. Fortunately no damage was done to me or anything else except the test-tube. The most complicated experiment I ever did was to make nitrogen trioxide. Dick and I did this together on the day when the Duke of Kent got married in November 1934 and we had a holiday to celebrate! It involved making a freezing mixture with ice from the fish-mongers, and setting up two simultaneous generators of nitric oxide and nitrogen dioxide and loading the two gases into a tube in the freezing mixture. We were rewarded with a few drops of a green liquid. But my main interest in those days was in the compounds of copper. I was particularly fascinated by copper sulphate – its colour, its crystals and its relatives – anhydrous copper sulphate, copper ammonium sulphate, and cuprammonium sulphate. The formula $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ did not satisfy me – I wanted to know which atom was attached to which, and was disappointed when I asked about this at school that the master had no idea. I felt that by gradually synthesising the substance by a series of reactions $\text{Cu} \xrightarrow{\text{O}_2} \text{CuO} \xrightarrow{\text{SO}_2} \text{CuSO}_3 \xrightarrow{\text{O}_2} \text{CuSO}_4 \xrightarrow{\text{H}_2\text{O}} \text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and by thinking hard enough about the shape of the crystals one ought to be able to deduce something about it. Much later I was to learn that Lipson and Beevers were working out the crystal structure at around this period!

The final phase of chemistry as a hobby was in 1935-6 when I discovered the joys of qualitative analysis. But after we moved to Derby in 1936 I think I only ever once did any chemistry at home. By then what one could do at school was so far in advance of what was possible at home that the latter was no longer of interest. The only exception was the attempted growth of crystals which I continued right through undergraduate days.

At Oxford I revelled in the practical work, especially the organic preparations and crystallography, but was disappointed to find that the meticulous accuracy necessary in quantitative analysis seemed to be beyond me. It was ironic that this skill which I never really acquired was one which I was subsequently to supervise at Ferodo and to teach in the Diploma in Geochemistry course at Oxford! Crystallography was the real new delight however. At school I had found the odd references to crystal systems baffling but now they all fell into place and linked up via X-ray crystallography with my feelings about copper sulphate. I really enjoyed using the (now almost obsolete) optical goniometer, and was fascinated when Tiny Powell told us that they had just solved the structure of PBr_5 and it was not like that of PCl_5 . The latter was a salt $[\text{PCl}_4]^+[\text{PCl}_6]^-$, which had been discovered at Oxford a year or two earlier, but PBr_5 was the salt $[\text{PBr}_4]^+\text{Br}^-$, and this was not unexpected because the bromine atoms were too big to put six of them around a phosphorus atom. The idea of understanding the actual sizes of atoms was then still relatively new, and it had not reached down to school teaching, though I had met it in 1938 in connection with steric hindrance in organic chemistry and been sufficiently excited to refer to this at my second scholarship interview with Sutton – and I always felt it had helped slightly in getting my scholarship. It was to become important in my work later (1958-70).

An interesting episode occurred during the supplementary crystallography course. There were never more than five or six of us doing it, and at one stage one of them had been preparing quinol by reducing quinine with SO_2 in the organic course. However instead of quinol he obtained some nice crystals of a substance known to form sometimes under these conditions and having the formula $(\text{C}_6\text{H}_4(\text{OH})_2)_3\text{SO}_2$. How the quinol was attached to the SO_2 was unimaginable at the time. He brought the crystals to the crystallography practical course and we all had a joint attempt to work out the structure. It proved to be too complex for us in the time available, but after the war Tiny Powell put a DPhil student on to it, and they discovered clathrate compounds – thereby earning Tiny his FRS and opening up a new and important field.

Of course the real beginning of my scientific work was when I started Part II in mid-August 1942. Because of the war it was only possible to have one's call-up deferred to do Part II provided that one joined one of the "teams" in the university working on war problems. Fortunately there was one which required some X-ray crystallographic work – powder diffraction on gas-mask charcoals, and I was to be supervised in it jointly by Sutton (a member of the team and my tutor) and Powell the senior crystallographer (the junior one being Dorothy Hodgkin!). These charcoals commonly had copper added

to them in some form, and my job was to find what form it took, how it was affected by the preparation and activation of the charcoal and what happened to it when the charcoal was treated with (poisonous) gas. The traditional material was coconut charcoal, but coconuts were obviously in short supply in 1942 and other materials were under investigation, notably coal. Coconut shells could be soaked in copper solutions, but coal could not, so it was powdered and mixed with promoters before coking. Sutton was away on holiday when I started, so he left me instructions to get the method started on a charcoal he had made, called CPCI, with coal and powdered copper metal (about 2%). So I took my first powder photograph, looked for the lines of copper, and they weren't there. When he returned my result was met with incredulity, because he had put the copper there, and I had an early training in standing up for my experimental results against other people's expectations! I then had to prepare comparison photographs of all sorts of copper compounds to identify and determine amounts and (qualitative) particle size of them in the charcoals. The solution of the first mystery was of course that the copper powder had reacted with the sulphur in the coal to give copper sulphides! It was a training in powder photography that was to stand me in good stead for many years. In the course of my eight or nine months work I took almost 110 photographs. The work did not lead to any great discovery but I think it contributed to the work of the team by stopping other people following false trails. As a side-line I was also put on to making a new sort of charcoal by carbonising nettles with concentrated phosphoric acid! At any rate my thesis was subsequently adjudged adequate for the award of a BSc without further alteration or addition.

From January to April 1943 I was busy applying for jobs to do industrial chemical research of some kind. I had interviews (in Oxford) for several companies, and was rather put out by the fact that the interviewers mostly tried to put me off research, and implied that I would probably only do that for a few years and then move on to other things. I was quite sure that that would not be what I would want to do. Only one firm called me for a second interview and that was ICI Dyestuffs at Manchester. They actually had a vacancy for someone to do crystal structures of their products, and that sounded like the job I wanted. Then while at home for Easter I had a letter from Mr Spiller the acting head of the Department of Mineralogy and Crystallography enclosing a letter from a Mr Hancock, Research manager of Ferodo Ltd, seeking a new graduate with a knowledge of crystallography. I applied, went to Chapel-en-le-Frith for an interview, was offered the job, and with some hesitation accepted it. It did not sound as interesting as the ICI job, but they had never written to say yes or no, so it was a question of a bird in the hand. In fact the choice almost certainly did lead to a much more varied and interesting career, and one in which I had far more scientific freedom than people whom I subsequently got to know who were working on crystal structures at ICI.

I started work at the beginning of August 1943 as "assistant physicist". The first shock was that the X-ray equipment, which was on order, had not been delivered. Dr Parker,

my boss, and I went to Metro-Vickers to discuss when it would come, only to be told that it was not scheduled for production because Ferodo had not put forward any evidence of war-priority. This was rectified and it was eventually delivered in November 1944, but the amount of use I was able to get out of it for the first 12-18 months was very limited. It was a continuously pumped demountable tube with vacuum seals, constantly made and remade with plasticine, and it was very unreliable, apparently because the vacuum wasn't good enough. When the Metro-Vick people came to deal with complaints about this they would always demonstrate that it could hold a good vacuum overnight after the pumps were switched off, and they obviously thought the troubles were my fault, until at last I found, and demonstrated, that the difference between their results and mine was that they simply pumped it down, whereas I of course then used it to produce X-rays. With the filament switched on the porcelain insulator got hot and leaked; if one then shut down the pumps the vacuum in the morning was bad, whereas if one waited till the insulator was cold before shutting down the pumps the vacuum in the morning was good. The result of this was that the vacuum was always poor while running. So I retrieved my reputation, we got a new insulator and all was well, though I also had a somewhat similar battle with Metro-Vick to prove that the design of their powder camera was intrinsically inadequate and different examples gave different results. Again I eventually got a reasonably good replacement, though poor by modern standards.

One result of all this was that my X-ray work got off to a very slow start, and I had to be employed on other projects. This prevented me from becoming merely a narrow specialist and I worked on the properties of resin-mineral composites and devised a very ambitious programme of work, design and practice, and designed, and in some cases made, pieces of apparatus to use in this programme. In these connections I improved what I knew of mineralogy and learnt some statistics. This background all combined to make me suitable, in Ferodo's eyes, for a switch of jobs nearly 20 years later and thence to my reaction of leaving! But at the time I found it all very stimulating, and in any case I was encouraged to move progressively back to my X-ray crystallographic interest.

The freedom I had was remarkable. Once the X-rays were available I was asked what I was going to do with them – not told what to do! So I started cautiously by saying I would take standard powder photographs of the company's raw materials, and this led to a continuing bread-and-butter usage of the X-ray method for identification and analytical work. This was the only X-ray work that survived my departure eventually.

The next field I explored was the structure of phenol-formaldehyde resins. These were of course amorphous, not crystalline, but they gave fascinatingly different diffuse diffraction patterns depending on the particular phenol derivatives used, and I longed to interpret them by Fourier inversion. Indeed my first full-blown published paper was a purely mathematical one on calculating Fourier transforms by a Fourier synthesis method. With the advent of computers 20 years late the "fast Fourier transform" became very popular in many branches of science and was really only a more sophisticated version of my approach in 1947, but that was lost in the literature and my name never

featured in its acknowledged parentage. Unfortunately this work on resins never led to anything useful, though there was one feature which I am still sure must have had some significance – a low angle ring in the patterns of resins made with para-substituted phenols which I have always regretted not having had sufficient opportunity to interpret.

An off-shoot of this work was the structure of crystalline intermediates in the formation of the resin – variously substituted dihydroxydiphenyl methanes. I did preliminary studies of quite a number of these, and felt that I was really getting down to the sort of crystallography I had always wanted to do. One of them proved to be crystallographically the simplest and I worked out and published its structure – 2,2'.dihydroxy-4,4'.dichlorodiphenyl methane, but by the time this was finished all the time that I could get for crystallography was diverted to asbestos – as manager of chemical research including resin development, infra-red and chromatography and chemical analysis time for fundamental research was restricted. In the late 50s there seemed hope of doing more of it at one remove – I had got Dave Swinburn trained to be able to do most of it himself, but the carefree days were ending and commercial pressures increasing and he was never able to finish the one structure he started.

It was probably in 1945 or 1946 that my attention was first directed towards research on asbestos itself. Our X-ray lab was supposed to be available to do work for T&N as a whole and not just for Ferodo. One Saturday morning Mr Hancock came to me with twelve little specimen tubes containing various kinds of asbestos. They had been given to him by Mr Forbester of Turner's Asbestos Cement with the suggestion that we should see what we could find out about them by X-ray diffraction. They were C+G3, HVL3, VRA3, Bells S4K, NAM, Italian tremolite, Finnish anthophyllite, Cape crocidolite, Bolivian crocidolite, amosite, monstasite and palygorskite.

The first five were chrysotile, and not in a very suitable form for investigation as they had been “opened”, that is to say mechanically treated to separate the fibres, which were therefore bent and mangled. However I took diffraction photographs of them as well as I could on the only vaguely suitable camera that I had at the time. This was a Metro-Vick “Universal Camera” which consisted of a steel “optical bench” on which a variety of fittings could be placed at arbitrary distances apart. It was quite absurdly large-scale, and included a fibre-holder intended for organic fibre specimens or rubber. I managed to clamp the bigger asbestos fibres into it but soon made (with my own hands) a tiny miniature version. The photographs had to be on flat films which made them troublesome to measure, but I soon confirmed that the tremolite and anthophyllite patterns could be interpreted in terms of the unit cells published around 1930, though I immediately noticed that the anthophyllite gave diffuse scattering along the layer lines, as also did the amosite and “montasite” which were identical to one another. According to the available literature they were supposed to be orthorhombic like anthophyllite but they were not, and I convinced myself that they were monoclinic – my first discovery about asbestos. However it did not seem a sufficiently large finding to write a paper about on its own, so I subsequently kicked myself for not at least publishing a brief

note when the finding was published in a fairly lengthy paper about anthophyllite in 1948 by a mineralogist called Rabbitt. The diffuse scattering phenomenon was to haunt me for about 30 years though. I guessed that it was due to something like random twinning, and about 1950 built a little wire model to aid thought about it, but I had no real idea how to tackle the theory. About 1960 I attempted to sort it out using the help of a Dutch student who came to work with me for a few months for work experience, and we tried to use optical diffraction methods at UMIST to sort it out. The guess about random twinning was eventually proved by electron microscopy with my research student Carmen Irusteta in 1974, and that year and the next I also did more extensive optical diffraction during a sabbatical at Cardiff, and in 1975 succeeded in computing what effects were to be expected. But the electron microscopy also showed that there were other contributing effects and I only got qualitative and not quantitative agreement between theory and experiment, so never published it – though I did talk about it in papers that I gave to the X-ray Analysis Group in 1949 and again in 1975!

The palygorskite was very poorly orientable and its structure was then unknown. I did a little work on it but had not got anywhere before its structure was eventually published elsewhere.

It was the blue crocidolites that really got me started. The Bolivian one intrigued me because it was apparently beautifully silky, yet an imperfectly aligned specimen gave diffraction arcs containing sharp spots suggesting the presence of relatively coarse crystals as well as fine ones. This proved to be the case under the microscope, and I was able to pick out a (twinned) crystal that was just big enough to do a complete crystal structural study – my first. Since the main silicate structures had been solved in the period 1925-35 before the days of Fourier synthesis and electron density maps, it was something of a departure to do it this way. Of course I based the work on the known basic structure of tremolite (known since 1931) but I got some very interesting results by not assuming that everything was regular. Especially I found that the Fe^{3+} concentrated in the M2 site and explained this on an electrostatic potential basis. I was extremely lucky in starting with the Bolivian crocidolite because it is almost the easiest amphibole in which to find the effect – now known to be generally true for the tri-valent ions Fe^{3+} and Al^{3+} . because it contains nearly all its iron as Fe^{3+} and nearly all its trivalent ions are Fe^{3+} this concentration is easily shown up by a much higher electron density at M2 than at any of the other sites. In other species without this density marker the effect is only detected by much more accurate measurements than I was able to make in 1948. My 1949 paper on the subject really established my reputation scientifically. Indeed when I was considering applying for the lectureship in geochemistry in 1964 I looked up *Geochimica et Cosmochimica Acta* to see what was involved in geochemistry (!) and found a paper that referred to my work on crocidolite. This evidence that I had been doing geochemistry, unknown to myself, gave me courage to proceed!

The remaining six specimens were all chrysotiles and they all gave identical zero and first layer lines on the diffraction photographs, but differed appreciably on their second



layer lines. I soon found that the Canadian Bell's fibre agreed with Bragg's observations in 1933 of a monoclinic structure but the others had both the reflections and some others. I remember puzzling about this as I did my daily $\frac{3}{4}$ mile walk from the bus stop at Furness Vale to my house at New Mills. It suddenly dawned on me that everything could be explained if the specimens were mixtures of the known monoclinic chrysotile with another orthorhombic one, because these could have identical zero and first layer lines but differ on all even order layer lines of which only the second was usually visible. Much later I found some very early published chrysotile diffraction patterns dating from about 1927, and some of these showed the same effect but no-one at the time had been able to interpret them at all. Again it was pure luck that Bragg in 1933 and those who began the reinterpretation of the structure (Warren and Aruja about 1942 and 1944) all studied only the Canadian material where this orthorhombic component is at low concentration and hardly noticeable, and so it was left to me to discover ortho-chrysotile.

The next step was to refine the structure of clino-chrysotile and find that of ortho-chrysotile. By this time, about 1952, I had acquired more suitable equipment, and oscillation camera, and also a Weissenberg camera which I adapted for all sorts of purposes not envisaged by its designer. Even then I really had to push things to the limit. With a crystal-reflected monochromatic X-ray beam, and moving the film holder to integrate the reflections in one direction it took exposures of the order of 150 hours to get suitable photographs which I could then photometer along the layer lines with the micro-densitometer (that I had designed and the electricians of which I had built). I worked out the structures, believing them to be cylindrical but assuming that the result would be the same as if it were an ordinary crystal that was rotated. Then I had to do the theory to justify this which took a year or so! And the theory was backed up by optical diffractions of circular, spiral, and arc-like lattices which I did in the Physics Department at UMIST. I shall always remember the first time I put the circular lattice on the optical diffractometer and Professor Lipson asked to look through the microscope first, and he said "the rings have fine structure in them!" and I said "yes, I know, that's what the theory showed should happen". My reputation was made with him at that point!

My connection with UMIST was very useful in giving me some access to an academic environment. It had started in 1943 when we had no X-rays at Ferodo and we wanted to try and X-ray investigation of a short-term production problem on railway brake blocks. Dr Parker had some years earlier given some lectures for them at Manchester College of Technology as it then was, so he rang DR W H Taylor who was then head of the Physics Department there and arranged for me to go over there to take some photographs. There were four crystallographers there and I got to know them. About 1945 W H Taylor went to Cambridge as reader and Henry Lipton took over his job. At an X-ray conference he announced that he was going to run colloquia from time to time for crystallographers in the Manchester region, and so I became a regular at these and was eventually asked to give one of the colloquia. This would be in 1948 when I had

just got quite interesting preliminary results in both the resin and asbestos fields, and I asked Mr Hancock (our research manager) which I should concentrate on so as to have enough to talk about and he said he thought I ought to make a good how of being an expert on asbestos – so I concentrated on that and gave the colloquium on it. It was just such an off-the-cuff decision (which I remember took place in the open air outside the Test House door) which decided the whole of my future career – without it I would never have become a lecturer in geochemistry or a reader in mineralogy.

The idea of curvature in the layers of the chrysotile structure had originated with Warren in 1942 and Aruja in 1944, but I really took it to its logical conclusion in deriving the details of the structure. I was almost beaten to it by Jagodzinski and Kunze in Germany. Our theoretical work was very parallel and their first publication came out before mine. Fortunately they made a number of fundamental errors in applying the theory to the practical results, largely because of a Teutonic sort of conviction that things must be in accordance with their idea of how the fibres must have grown, and tried to force the results to fit a Procrustian bed.

By 1957 my results were being fairly widely accepted and I was very bucked when Sir Lawrence Bragg (founder of X-ray crystallography and author of the original incorrect chain structure of chrysotile) invited me to lunch in his flat in the Royal Institution on 6th March to show him my results and models. Since 1943 he had accepted that layer structure but he told me he had always felt that the fibres would be like  in cross section rather than  as I had found – at least by strong circumstantial evidence. However he accepted my ideas to the extent of putting them in the new edition of his book. It was not until 1967 that I was totally vindicated by Yada's high resolution electron microscopy. This was sent by Yada to Jack Zussman to ask him if it was suitable for publication (!) just at the beginning of the 1st Asbestos Conference which we had organised in Oxford. Jack managed to get his permission by telephone for us to present his results at the conference, and I had the welcome task of doing this and so demonstrating the physical reality of my ideas.

After 1957 I did a few more things on chrysotile but they were fairly peripheral – the main features were solved. It had been topped off also by the discovery, by powder diffraction, of the mineral lizardite which I shared with Jack Zussman. He had come to the Geology Department at Manchester University in about 1952 or 1953 and also started work on serpentines and amphiboles. We were introduced to each other by W H Taylor at a meeting of the Mineralogical Society, and we started collaborating on the nature of the serpentine matrix of chrysotile deposits. At that time it was vaguely supposed to be composed of antigorite, but we showed that it was largely disoriented chrysotile, ortho and clino, but I soon found that some of the apparent “ortho chrysotile” was different and we had to recognise a new mineral which we named lizardite. Subsequently I attempted further work on the serpentine rock by pure X-ray powder photography and did not get anywhere. Jack, with the techniques of a Geology Department, also used optical petrology and also did not act much further until 1965-7 when he set a

research student, Fred Wicks on to it using X-ray microbeams as well. They made a major advance but did not get on well together so when Jack left Oxford in 1967 Fred stayed with me until 1969 and we reaped quite a lot of fruit. We eventually wrote several papers reviewing various aspects of the chrysotile and serpentine structure, texture and formation and for two of these twice received the Hawley Award of the Mineralogical Association of Canada in successive years in 1977 and 1978.

Meanwhile I had returned to amphiboles. In 1958 Ferodo's new research labs were opened by the Duke of Edinburgh and we put on a big show for this. In preparation for it I built quite an extensive dissectable structure model of amphibole to show its difference from chrysotile. Once the festivities were over I realised that this model provided me with an opportunity to think about and perhaps understand some of the mysteries of the relationships of the many different amphibole compositions to one another, and why they were so various and yet limited in particular ways. This was pure crystal chemical mineralogy and right outside my real remit of studying asbestos, for most of these compositions were non-asbestiform. The project was very successful and led to a major publication. While I was writing it someone discovered that the lithium amphibole holmquistite was orthorhombic and not monoclinic as supposed (though actually still later the monoclinic form was found as well) and this fitted beautifully with my theory based on ionic radius and charge balance. Subsequently at Oxford in 1967 or so I followed this up by doing the structure of holmquistite in projection and subsequently about 1972-3 the full 3-dimensional structure with my student Carmen Irusteta, and in 1969-70 my first completely computational work on charge balance. The same theory was behind our (Carmen and my) high resolution electron microscopic study of fine scale twinning in various amphiboles – and the first observation of a triple-chain structure within a double-chain amphibole was a chance by-product of this work. Though we undoubtedly had priority in this discovery of a triple chain that fact has unfortunately tended to be submerged by the flood of results on the subject which quickly came in from the States, only partly offset by my subsequent work with Barbara Cressey around 1980 when we also extended it to cross sections of quadruple, quintuple, hexuple etc chains.

Much easier than this in 1966 I applied my 1959 work on amphiboles to devise a logical scheme to classify them. Although never formally adopted by others I used it in teaching for ten years, and it led to my eventually serving on the Amphibole Nomenclature Committee of the IMA. Then after that, about 1968-70 I further extended my 1959 work on amphiboles to make it quantitative by calculating electrostatic energies of different distributions of ions in the amphibole structure. This was the first project for which I wrote computer programs, and it was moderately successful. Further in 1970 I inherited the supervision of a research student (Tony Law) working on Mossbauer spectra of iron-containing amphiboles to identify the site distribution of Fe^{2+} and Fe^{3+} , supplemented also by infra-red spectroscopy. He and I also collaborated on a purely theoretical study of stacking patterns in amphiboles.

Before going on to describe new fields that I went into at Oxford it is appropriate to recall some other branches of scientific work that I was involved with at Ferodo. The first was infra-red spectroscopy. We bought a newly developed infra-red spectrometer from Grubb-Parsons Ltd in 1948-9 and it was assigned to me, with the help of Dave Swinburn, to work out methods for quantitative analysis of phenol mixtures. This was for the purpose of ensuring standardisation of the raw materials for resin production. We used two grades of cresol containing respectively 40% and 50% of the meta-isomer, but the remaining 60% or 50% was not guaranteed in any way, and the crucial meta content was always liable to fluctuation. We got a method going which was used for about ten years for quality control but the repeatability of the instrument was barely adequate, and in 1958 we bought the newly developed gas chromatograph and developed a much more satisfactory method on that.

In 1952 I was asked to start work on the chemical nature of cashew nut-shell liquid (CNSL), then becoming one of our major raw materials, and I recruited a new assistant, Barbara Dennis, to do this. She did all the practical work under my supervision. It was very tedious work using long, slowly dripping liquid chromatography columns, and the results tended to be a bit vague though we were able to institute major blending procedures to even out variations in reactivity of the material on the basis of one of its components. But the work became much more detailed when we were able from 1958 onward to apply the gas chromatograph to it. Eventually Barbara wrote the work up for an external London PhD, so she was actually my first research student – though she did not submit till 1968 so was my second such to get her doctorate.

From 1949 onwards a considerable part of the effort of my lab was devoted to the analysis of competitors' products, the main work (at least up till 1957) being done by Dave Swinburn with the help of others. We applied all our available techniques to this task – X-ray crystallography, infra-red spectroscopy, microscopy, and wet chemical analysis, and also gas chromatography when that became available. These analyses tested our ingenuity to the utmost, and I was able to make considerable play of having supervised this work when I had my interview with Prof Wager for the lectureship in geochemistry in 1965. The problems were sufficiently similar to those in geochemical analysis to be relevant, and sufficiently more complicated than those to be impressive!

Throughout the period 1949-63 however, the main work of the laboratory was the development of new resins. I mostly supervised this at one remove through my senior assistant – Allan Rea from 1949-57 and Dave Swinburn after that, – but when the time came to transfer a new resin to larger scale manufacture I always took charge myself. Always, that is to say, after the first time when I allowed a junior to do it and, in spite of his having much more experience than I would have had, I got the blame for letting him wreck £100 worth of chemicals. It involved working out how to scale up from a 5 litre glass flask to a 200-300 gallon steam-heated steel vessel.

It was a hard, tiring, and worrying day the first time we made such a transfer and usually meant working about a 12 hour day. But in a strange way I really enjoyed the challenge and the working with the foreman, charge-hand and operatives in the factory, and I felt considerable satisfaction in seeing quite a lot of materials through to regular large-scale production.

When I moved to Oxford I had to start thinking about quite different problems. My first research student was already half into his project which involved partition of trace elements between rock-melt and crystals. I also had to devise a course of lectures on some aspects of geochemistry, and as I read up the literature on this particular problem I found it to be full of the most incredible illogicalities in connecting such partitions to the ionic radii, electronegativities, and ionisation potentials of the elements concerned. This led to a major debunking paper which was very successful, It also led me to think a lot more about two more topics in themselves – ionic radii and the structure of silicate melts. My lectures on the subject caught the fancy of a post-graduate geochemistry student, Rosemary Muntus, and under my supervision she did her dissertation on ionic radii. In this she suggested an ingenious way of combining the two classical approaches to determining the radii of ions. That was in September 1969, and over Christmas I had 'flu and while recovering in about a week I applied the idea systematically and devised a new and better set of “ionic radii for use in geochemistry”. It was the quickest piece of work I ever did, and I published under our joint names, and it has been cited more often than any other paper I even wrote!

My exploration of the structure of silicate melts was much less successful. I came across the work done by Bernal a few years earlier on random close-packing of equal spheres, which he applied to the structure of metals and other monotonic liquids. In view of the relationship of many silicate crystal structures to regular close-packings of oxygen ions I latched on to this as an approach to silicate melts by putting the metal ions in the available cavities between the oxygens. Bernal had listed the types and numbers of these from an experimental examination of sphere packings and (in collaboration with Paul Henderson) I did lengthy computer simulations to deduce the availability of the cavities as cation sites on the basis of the observed partition of Ni^{2+} between crystals and melts, but this work was abortive. I made several attempts to use the coordinates of the sphere centres of Bernal's 1000 sphere model (which I got from him on punched tape) to identify and enumerate the various kinds of cavity so that I could then assess their suitability as sites for cations in terms of bonding. The first two or three attempts to write a program failed because it became so complicated that I could not keep track of it. Not until I learnt ALGOL68 in 1974 did I succeed because of the simplifications introduced by this language. But when I ran the program it became evident that Bernal's results were highly inaccurate and ignored some kinds of cavity. Moreover it turned out that the tetrahedral cavities were far fewer than he indicated and their mutual dispositions made the structure impossible for any reasonably polymerised silicate. Though I was able to publish this study it put an end to the development of the idea.

In 1970 at the meetings to celebrate the 80th birthday of Sir Lawrence Bragg a paper was given about a very sophisticated way of studying the structure of glasses by X-ray diffraction. This obviously provided an approach to melt structure, and I built the necessary apparatus and obtained a research student (Christine Sheppard) to work on it. We had a lot of difficulties with the apparatus which were eventually overcome, but much more serious ones in preparing glass specimens of a sufficient range of compositions. She just scraped her DPhil, but this work did not advance the subject much. A little more information was subsequently obtained by the EXAFS method on some of her glasses at the Daresbury synchrotron source, and this was the only part of the work that was properly published.

One small project turned up around 1980 which was only slightly related to anything that had gone before, though its possibility was suggested by my work in 1968-70 on electrostatic energies in amphiboles. I had never been happy with the lack of explanation of many common types of twinned crystals, especially those of interpenetrating type, where the twinning process obviously only occurs once in each crystal. This unsatisfactory situation was particularly borne in on me while writing my crystallography text-book in about 1979. I therefore tried to study it by building extensive small scale models of the fluorite structure to see what sort of error must occur in the nucleus of the crystal to cause it to twin. This appeared to involve a hexagonally symmetrical nucleus and I wanted to compute its energy relative to an untwinned nucleus, as the twin boundaries developed, I have never found out how to do this, but I did compute the bonding energies across various crystallographic planes in both halite and fluorite structures and so was able to give quantitative interpretations of their cleavage properties and I was able to publish this.

My last scientific field was to be quite different – the crystallography of higher dimensions. This all started in 1967 when Roger learnt 2-dimensional symmetry at school, and I was shocked that he learnt to call a 2-fold rotation point what I thought was a centre of inversion. It was some time before I realised that the kinds of symmetry change with the number of dimensions. I then got interested in representing four-dimensional figures and hit on the idea of using colour gradations to represent a fourth dimension. I regarded this as a mental diversion at home rather than serious scientific work, and I built and coloured my models of the 4-dimensional hypercube in the garage at home. I was not until I found that there were 15 fundamentally different projected models, and I needed some way of arranging them to illustrate their relationships, that I invented the hyperstereographic projection and the whole subject became serious. This was about 1971. Then having found how to design a projection of a hypercube in any desired direction it occurred to me that I could make two slightly different projections, view them stereoscopically, and so see the fourth dimension, I succeeded in this about 1973 or 1974 but could not persuade any journal to take my paper on it seriously. However I gave a lecture on the subject in the Physics Department at Cardiff following my sabbatical there in 1975 and Charles Taylor (the professor) was very impressed. He tried to get

various journals to accept it without success and eventually persuaded *Acta Cryst* of its virtues.

During the 70s there was a lot of theoretical work going on about 4-dimensional crystallography, and what I heard about this culminated in 1981 at the Ottawa congress of crystallography with my seeing an exhibit on computer graphics. It then struck me that I could produce hyperstereograms of all the 227 crystal classes in 4 dimensions by this means, along the lines that I had used to produce a physical model of the one for the holosymmetric hypercubic class in 1971. I sketched out a few of these on the spot, and by the end of 1981 had devised the essentials of the necessary computer program, though it was well into 1982 by the time I got it working properly. The complete project took about two years including the writing of a book on the subject – necessary because the amount to be illustrated far exceeded what could go into the series of papers which I also wrote. The book was completed just after I retired at the end of 1983 and published in 1985.

In 1984 at the Hamburg congress there was a poster presentation by a Frenchman which claimed that all the “strange” symmetry points in 4 dimensions were double rotations. This had never been suggested by previous workers and was at variance with what I said in the book, or at least went beyond it. It appeared to me to lead to impossible results, and I almost persuaded the author of this when he came to see me in 1985. It was not until the summer of 1985 that Roger helped me to sort out the maths, and he demonstrated that a double 5-fold rotation (and also 8-, 10-, and 12-fold) was compatible with a crystal lattice provided that the two planes were irrational ones, and he was able to find their orientations. I therefore revisited my computing of the hyperstereograms to illustrate the situation and Roger and I published a joint paper. One of the referees of this suggested a relationship to Penrose patterns, and so Roger and I then looked into this, found the connection and did quite extensive further work on generalised Penrose patterns and again published a joint paper. It was singularly appropriate that an interest inspired by Roger’s school maths when he was about twelve should have led to our publishing two joint papers some 20 years later. Finally, they led to our being invited to contribute a chapter to a book on fivefold symmetry, the writing of which finally clarified a number of the concepts involved that still puzzled me. At the time of writing this is still in press, but has sufficiently impressed the editor that he is proposing to publish it also (along with eight of the other chapters) in a new journal of symmetry.

[Note added later (1991)] The book was published in 1990 but the journal folded before publication for lack of orders. However I was further invited in 1990 to join a committee on nomenclature in 4-dimensional crystallography. I declined to join formally, but corresponded extensively with the English representative on it, and he persuaded me to write a paper on enantiomorphic crystal classes. Roger and I also wrote a paper in 1991 on the orientational parameters of symmetry operations in n dimensions.