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Varian Associates and the birth of commercial NMR spectroscopy

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ABSTRACT

The birth and early growth of commercial NMR instrumentation, and the parts played by the brothers Russell and Sigurd Varian and their colleagues, are described.

1. Introduction

We think of Silicon Valley as a modern phenomenon – the rapid growth of businesses in the Santa Clara Valley near San Francisco, starting in the 1970s, based first on semiconductor technology, and subsequently on the myriad uses to which such technology can be put. The changes in society that this small geographical region has brought about are proving as far-reaching as those of the Industrial Revolution, which began in the area around Manchester in the early 19th century. Just as the Industrial Revolution had its roots in earlier technological developments in rural Lancashire, so modern-day Silicon Valley grew from a nucleus of high technology companies clustered around the campus of Stanford University. One of the first of these – indeed the first to move into the Stanford Industrial Park in Palo Alto, California – was Varian Associates.

Russell Harrison Varian and Sigurd Fergus Varian were born to Irish parents (the surname Varian is particularly associated with County Cork) who were members of the utopian Theosophist Temple of the People community in Halcyon, California. The great American photographer Ansel Adams was a family friend, and all three were involved in the Sierra Club. Russell was named after the Irish poet, theosophist and mystic George Russell, better known under the pseudonym Æ, who was a friend of his father. Russell was dyslexic, and took six years to graduate in Physics from Stanford University; after his application to the Stanford PhD graduate school was rejected, he worked first in the oil industry, where he developed a magnetometer, and then in television technology. Sigurd dropped out of college and worked as a pilot, flying particularly in Mexico and Central America and learning first hand about the limitations of the navigation systems then available. In the mid 1930s the two brothers left their jobs and set up a laboratory together in Halcyon, where they worked on developing a microwave-based system for locating aeroplanes, in part motivated by Sigurd's concern at the dangers posed by aerial warfare and the rise of fascism. A request for help to a friend of Russell's, Stanford professor William Hansen, led to their relocation to the Physics department at Stanford, and eventually to the development of the klystron.

As a practical high power microwave source, the klystron played an important role in the development of radar. It was quite quickly supplanted in radar by the cavity magnetron, but remained important in a range of microwave applications, from telecommunications to particle accelerators. After war work at Sperry on Long Island, the Varian brothers returned to California, and in 1948 they set up Varian Associates to market the klystron and to develop other electronic technologies (see Fig. 1). Their links with Stanford meant that as early as 1946 they were aware of the first, almost contemporaneous, successful measurements of nuclear magnetic resonance in condensed phases [1], by the groups of Ed Purcell at Harvard [2] and Felix Bloch in Stanford [3]. Remarkably – at a time before either the chemical shift or the scalar coupling had been discovered – Russell Varian saw the potential for NMR as a tool for chemical analysis. As the nephew of a patent agent, he was aware of the value of protecting intellectual property. When Bloch and Hansen proved unwilling to pursue a patent themselves, Varian arranged to file a US patent for them in return for exclusive rights that would in due course be assigned to Varian Associates. The patent was filed in 1946 (just one day before the deadline imposed by Purcell, Torrey and Pond's publication of their first observation of nuclear magnetic resonance[1]), granted in 1951, and then reis-

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Fig. 1. Russell (left) and Sigurd Varian with a klystron. Reproduced with permission from Shoolery, "High-Resolution NMR: A Dream Come True", eMagRes, DOI https://doi.org/10.1002/9780470034590.emrhp0170. © 2007 John Wiley & Sons, Ltd.

sued in 1955 to cover electron as well as nuclear resonance. The patent protection both enabled Varian Associates' early NMR efforts to become self-sustaining, and generated a substantial financial return to Stanford University (including contributing to a new building for Physics).

Further evidence of Russell Varian's far-sightedness was provided by his filing of a patent for what became pulse Fourier transform NMR, in 1956, ten years before the first successful demonstration of the method (and long before the availability of the computing power needed to make it commercially viable). Again the nucleus of this idea came from the very early days of NMR. The possibility of pulsed, rather than continuous, excitation was set out in Bloch's 1946 paper, and in 1948 Varian invented a free precession magnetometer for detecting small perturbations in the Earth's magnetic field [4]. One of the applications for which this was intended was to find victims of avalanches: skiers would carry small magnets, which would enable rescuers equipped with portable magnetometers to find them. This idea did not take off – other applications such as land mine and submarine detection were more successful – but has an echo in the current widespread use by skiers of radiofrequency avalanche transceivers/beacons.

2. Experimental

In the very early years there remained quite close cooperation between the nascent Varian Associates and Stanford. When Jim Arnold and Wes Anderson in Bloch's group set out to build a 30 MHz permanent magnet spectrometer, in order to avoid the rapid field drift experienced with early electromagnets, equipment loaned by Varian was used when energising the magnet [5]. (The solenoid used for this required a one second long pulse with a current of 400 A, and a power of almost 2 MW, so was performed at 1 am in the morning). The magnet and spectrometer were constructed taking advantage of the ready availability of cheap war surplus radiofrequency and mechanical equipment; the choice of field was dictated in part by the 30 MHz standard intermediate frequency of wartime US radar systems. The results produced, and in particular the spectrum showing the three chemically shifted signals of ethanol published by Arnold, Dharmatti and Packard [6], provided powerful evidence of the potential chemical utility of the new technique. Bloch's name was not included in the author list of this classic paper, despite his direction of the project, apparently because he was more interested in the physics of NMR than the chemistry [7]. Erwin Hahn later commented "In the beginning of NMR Bloch hated chemical effects" [8].

Varian Associate's first high resolution spectrometer, the HR-30, was sold in 1952 to the Humble Oil Company, where Russell Varian had worked designing a magnetometer before the war. This 30 MHz water-cooled electromagnet spectrometer (see Fig. 2) weighed over two tons, and had sufficient field homogeneity to resolve the chemical shifts of the three groups of protons in ethanol. The magnet was based on a design by Harry Weaver, of Bloch's group. Weaver subsequently joined Varian, and was later responsible both for the first commercial superconducting magnet NMR spectrometer, and the first commercial EPR spectrometer. Within a year, the resolution achievable at 30 MHz had improved sufficiently to resolve structure due to scalar coupling [9], but by modern standards sensitivity remained very low and instability and irreproducibility were major problems. These problems remained in the next instrument, at 40 MHz, but the HR-40 did provide a major advance in resolution, by incorporating sample spinning. This advance came again from Felix Bloch, who in 1954 had realised (reportedly while stirring a cup of tea[7]) that the effects of static field inhomogeneity could be greatly reduced by rapid stirring or spinning of the sample (Herman Carr had had the same insight three years earlier, but had not published the idea). Like all the early instruments, the HR-40 required some heavy engineering, including a 1.5 kV, 1 A current-regulated power supply for the water-cooled electromagnet. The original magnet had no facilities for adjusting the magnetic field homogeneity: all the user could do was to use two screw drives to move the probe around the magnet gap to search for the position that gave the sharpest lines.

Early attempts to improve magnetic field homogeneity used mechanical deformation of the magnet yoke, and the insertion of

Fig. 2. HR-30 spectrometer in the applications laboratory at Varian Associates. Reproduced with permission from Shoolery, "High-Resolution NMR: A Dream Come True", eMagRes, DOI https://doi.org/10.1002/9780470034590.emrhp0170. © 2007 John Wiley & Sons, Ltd.

thin pieces of metal (shims) between magnet pole faces and probe, so the improvement on spinning the sample, averaging out the effects of variations in magnetic field perpendicular to the sample spinning axis, was considerable. This time Bloch did patent his idea, which remained in use for many years. There is perhaps a pleasing irony in this subtle and insightful physicist patenting this simplest, and in retrospect the most obvious, of all the major advances in the practice of NMR spectroscopy. Subsequently, over the period 1957–1959 Wes Anderson and Ed Jaynes at Varian and Marcel Golay at Perkin Elmer developed the idea of shim coils, allowing magnetic field homogeneity to be optimised by changing the electric currents running through coils shaped to give a basis set of different magnetic field distributions, rather than by mechanical means. This, along with the use of magnetic susceptibility-matched materials in probes, allowed the contribution of static field inhomogeneity to instrumental linewidths to be greatly reduced.

3. Results

By 1953 it was clear that the phenomenon of nuclear magnetic resonance had a great deal to offer the chemist, but the unfamiliar nature of the physical principles, and the practical challenges of running experiments, presented real obstacles to its exploitation. Varian therefore set up an Applications Laboratory, a model that has been widely adopted, in which potential academic and industrial users could see instruments demonstrated. The App Lab also published a regular "Technical Information from the Radio Frequency Laboratories of Varian Associates" (later the "Technical Information Bulletin"), which was circulated to potential users in industry and universities, and a monthly advertisement, 'This is NMR at Work', in Analytical Chemistry. The work of the App Lab, and in particular of Jim Shoolery, played a major part in the rapid acceptance of NMR spectroscopy by the chemical community. Among the many innovations of the App Lab were the Varian NMR spectra catalogue, and "Shoolery's Rules" for predicting chemical shifts in families of cognate chemical structures. Customer feedback, often through the App Lab, played a significant part in setting the agenda for instrumental developments. Subsequent application laboratories were set up on the East Coast and, in 1962, in Zürich.

The problem of field stability that bedevilled early instruments was partially solved in 1956, allowing an advance in field strength to 60 MHz proton resonance, through the introduction of the "superstabilizer". This used a coil wound around the magnet pole faces to sense any change in field, generating a voltage that was amplified and fed back to reverse the change. This negative feedback loop improved stability by several orders of magnitude, and the resulting instrument, the 1958 HR-60, was a commercial success for Varian. Only a year later the full potential of iron-cored electromagnets was realised, reaching the limit imposed by magnetic saturation, with the introduction in 1959 of the 100 MHz HR-100. This was a formidable machine, with a vacuum tube power supply that occupied a separate room. It used a rather temperamental field stabilizer that relied on a mirror galvanometer, but it brought a major gain in spectral resolution. Its 1 Hz instrumental linewidth represented a resolution of 1 part in 10^8, giving rise to Shoolery's famous analogy that "astronomers with a telescope of equal power would be able to see two cats on the moon sitting 1 ft. apart" [10].

High resolution NMR was by no means the only magnetic resonance market explored by Varian at this time. Other products included fluxmeters, magnetometers, magnets, a wideline NMR spectrometer, and, based on this last, the first commercial EPR spectrometer, in 1956. In the early days, developments in NMR were subsidised by sales of klystrons, only later becoming self-sustaining. The abbreviation "NMR" itself owed its adoption to the difficulties Varian sometime encountered exporting equipment because of the "nuclear" in "nuclear magnetic resonance" [11].

All of Varian's NMR instruments up to this point required significant skill – and patience – to operate. Their next step was to create a machine for the mass market, that could be run by a much wider range of scientists and that could compete in accessibility and in breadth of acceptance with instruments such as infrared spectrometers. The A-60 instrument (A for Analytical) was specifically designed for chemical structure elucidation, and was engineered to be compact and self-contained, with a much smaller electromagnet than its predecessors, with 6″ pole faces (see Fig. 3). The key component, which had been patented by Russell Varian in 1956, was a second level of negative feedback for field stabilisation that used the NMR signal itself to sense any change in field. Up to this point it had normally been necessary to establish the chemical shift scale of a spectrum by quickly running a second spectrum, of a reference material, hoping that the field did not change in the interim. The introduction of field-frequency locking to the signal of a second, water, sample, coupled with the introduction of tetramethylsilane, by Tiers in 1958, as an internal chemical shift reference, meant that this was no longer necessary and allowed pre-calibrated chart paper to be used. (Later, following a suggestion by Hans Primas, TMS itself was used to provide the lock signal). The A-60 played a major part in establishing NMR as a routine chemical tool, supported by Shoolery, Bhacca and Johnson's illustrative "Varian High Resolution NMR Spectra Catalog" of over three hundred 60 MHz spectra. Some A-60 instruments remained in use as late as the 1980s, despite all the intervening advances.

The 1960s were in part a period of consolidation, for example replacing the electromagnet of the A-60 with a permanent magnet in the T-60, and adding an internal field-frequency lock based on a sideband of the TMS signal (as opposed to the external water lock signal of the A-60) to the HR-100 to give the HA-100. A range of double resonance experiments were implemented by Wes Anderson and Ray Freeman, using sideband generators to provide a second radiofrequency field of controllable frequency. One major advance was the first commercial superconducting spectrometer, the HR-220, with a Nb-Ti wire magnet developed by Harry Weaver, in 1962–4. This was followed in 1967 by a multifilament wire 300 MHz instrument, the SC-300, but in both cases sales were limited by the high cost both of the instruments themselves and of the

Fig. 3. The A-60 spectrometer. Fig. 1. Reproduced with permission from Shoolery, "High-Resolution NMR: A Dream Come True", eMagRes, DOI https://doi.org/10.1002/9780470034590.emrhp0170.

cryogens needed to maintain superconductivity (the early magnets needed a helium refill every few days, rather than months). A second advance, the introduction of the Computer of Average Transients or CAT, allowed signal-to-noise ratio to be improved by averaging the results of multiple scans through a spectrum. The CAT was available for use with the HA-100, HA-60 and DA-60 spectrometers. The biggest step forward, though, was provided by Ernst and Anderson's first demonstration in 1966 of the big sensitivity advantage enjoyed by acquiring spectra in pulse Fourier transform (FT) mode, rather than the continuous wave (CW) mode that had been the rule until that point. In retrospect this was – appositely – a transformative advance: it opened up the periodic table to NMR chemists, and played a major part in the award of the Nobel Prize in Chemistry to Richard Ernst in 1991. At the time however, things were much less clear: with no mini- or microcomputers it could take several days and a journey across Palo Alto to get a free induction decay Fourier transformed using an off-site computer. Indeed Ernst and Anderson's paper was rejected twice by the Journal of Chemical Physics before being accepted by the Review of Scientific Instruments [12].

Varian Associates' US patent and rapid development of the NMR market gave them a flying head start, but already in 1956 JEOL were offering a 32 MHz electromagnet instrument and Trüb-Täuber a 25 MHz permanent magnet. By the mid-1960s they had been joined briefly by AEI (Associated Electrical Industries, at the time the largest electrical group in the UK) and by Perkin-Elmer in the UK, and by Hitachi in Japan, and Trüb-Täuber in Switzerland had morphed first into Spectrospin and then into Bruker. Varian were slow to capitalise on the potential competitive edge offered by their development of FT methods, and were beaten to the market by more agile competitors, though they did offer an FT accessory for the HR-100 instrument as early as 1968. The HA-100 was replaced by the versatile XL-100 spectrometer, a dual CW/FT instrument that covered a wide range of nuclei and used large-sample probes to boost the sensitivity of lower-gamma isotopes. In an echo of the development of the A-60, Varian then decided to produce a lower-cost electromagnet instrument dedicated to measurements on ^{13}C, the CFT-20, operating at 20 MHz for ^{13}C (and subsequently, as the FT-80, at 80 MHz for ^{1}H). With an internal field-frequency lock based on the use of a deuterated solvent and a built-in Varian Data Machines 620 minicomputer, this was in some respects an instrument designed down to a price rather than up to a specification, but the use of the minicomputer to control many of the functions of the instrument was to prove crucial to the blossoming of multiple pulse NMR techniques in the mid-1970s (and to the doctoral studies of the author with Ray Freeman in Oxford, following Freeman's move there from the Varian research laboratory).

Up to this point, each different type of NMR experiment required dedicated hardware. Now for the first time it was possible to change from a simple pulse-acquire FT NMR experiment to inversion recovery simply by changing the parameters supplied to the software. Importantly, for those prepared to invest the time and effort in mastering the internal programming of the instrument, it was possible to programme arbitrary sequences of pulses. Instead of requiring weeks of preparation in the workshop, a new experiment could be designed and implemented in an afternoon, simply by changing the software. ("Simply" is slightly disingenuous here: the CFT-20 was supplied without source code or compiler, so changes to the software were accomplished by changing the 16-bit machine code using a small utility programme. One false move and the computer would crash, necessitating a complete reload of the software, initially from cassette tape. In the early days this required a "bootstrap" programme of several dozen 16-bit numbers to be entered manually, in binary, correctly and in the correct sequence, using mechanical switches.)

The power of the computer-controlled approach rapidly became clear with methods such as DANTE and early heteronuclear 2D NMR techniques, and Varian were quick to provide a 1 megabyte hard disc accessory. This both made it much simpler to switch between different versions of the software, and made it possible to process 2D datasets larger than 8 kilobytes in size. Considerable ingenuity was required, principally on the part of Geoffrey Bodenhausen and David Turner, to programme a double Fourier transformation within the same memory space as the Varian-supplied single FT spectrometer control programme. The 620 computer had a 1 MHz clock speed and used magnetic core memory; the computer was not shielded, so the deuterium lock display showed interference that changed in character if the software timing was changed. The transistorised electronics were relatively accessible, allowing the timing circuitry to be improved and, for the development of INEPT [13], a 90° phase shifter to be added to the ^{1}H decoupler. The Varian 620 computer was also used by Richard Ernst in a modified DA-60 instrument for the very first 2D NMR [14] experiments, and in Fourier NMR imaging [15] experiments.

By this stage Varian's ethos had changed from the early days of the Varian brothers' pursuit of creative development of new technologies; Russell had died in 1959 and Sigurd in 1961. The demands of the stock market led the company to concentrate on generating a return on investment rather than pushing the boundaries of instrument development. Concentration on the middle of the market and diminished enthusiasm for research left Varian with just one superconducting instrument, the very expensive SC-300, at a time when competitors were carving out a new market for high field instruments, and for the first time they were faced with real competition within the NMR market. By 1978, when Varian belatedly introduced the 200 MHz XL200, instruments from other manufacturers were already available at 250, 270, 300, 360, and 400 MHz. Bruker's alliance with Oxford Instruments allowed them to dominate at the top end of the market, initially using multiples of the 90 MHz radiofrequency of their HX-90 instrument for ease and speed of console development. However, the innovative control system of Varian's XL200, which used separate computers for processing and for experiment control and data acquisition, set a pattern that continues to this day (as does the frustration of persuading multiple semi-autonomous computers to communicate reliably at high speed in order to carry out ever more complex experiments.) Initially the XL200 used Sperry-Univac V77 computers, but later these were replaced by Motorola VM02 single board computers. (The author's early V77-based XL300 instrument suffered from a bad batch of integrated circuits used by Varian in one of its periodic economy drives; until the new VM02-based computer system arrived the system would crash once a day on average, so an automated system was built, based on an early PC, to restart the computer and resume the interrupted experiment should the system detect that pulsing had ceased).

Varian had also learned from Ray Freeman's experience developing new pulse sequences and techniques on the CFT-20, and made the far-sighted decision not only to give users the freedom to code their own pulse sequences (in a subset of PASCAL), but also to make a user programming kit available that would allow high-level language reprogramming of the main computer software. While this was only used by a small minority of customers, the work of that minority helped Varian to maintain a competitive edge in technique developments despite lagging behind the market in hardware. The ease with which new experiments could be implemented meant that for a considerable time Varian remained the market leader in the development of novel experiments despite having a much reduced overall market share. Thus in the 1996 festschrift "Magnetic Resonance in Perspective: Highlights of a Quarter Century" [16], celebrating 25 years of the *Journal of Magnetic Resonance*, while the articles selected to be included were divided roughly evenly between those using Varian instruments and those using Bruker, over 60% of articles on technique development used Varian spectrometers.

Varian's communications with current and potential customers took a variety of forms over the years. The early "Technical Information Bulletins" and "This is NMR at Work" advertisements in Analytical Chemistry were followed by such vehicles as "Magnetic Moments - the voice of Varian NMR" (1985–1998) and the email newsletter VNMR News (1994–2013). The latter was unusual in giving existing customers a public mechanism for reporting software problems, and providing a regularly-updated list of problems awaiting resolution. The public nature of the list gave Varian a strong incentive to fix problems in a timely manner, and gave users reassurance that bugs were known and confidence that they would be corrected. It also often led to the user community contributing to the solutions of problems. In 2014 the email newsletter was replaced by a more ambitious web portal, "Spinsights" [17], but the public bug list was discontinued.

4. Conclusion

The melancholy tale of decline and fall in the later years of Varian NMR, culminating first in its sale to Agilent in 2010, and then in 2014 to its closure, has already been told [18] and will not be rehearsed here. While many Varian/Agilent instruments survive, already time is taking its toll on their numbers, and users are slowly migrating to competitors' systems and adjusting to the individual strengths, weaknesses and idiosyncracies of their software. In one of the few positive outcomes from Agilent's disastrous purchase of Varian, Inc., in which a business bought for $1.5bn was written off in less than five years, Varian's VnmrJ software has been released as open source code and acquired a new life as Open VnmrJ [19]. The code remains under active development by the NMR community, nobly supported by former Varian staff, and is now available for a range of modern operating systems. While most if not all NMR spectroscopists will mourn the loss of the great tradition of innovation that Varian represented, its passing leaves scope for newcomers to the market to bring their own different contributions. In a return to the past, permanent magnet instruments are once more being sold, taking advantage of modern developments in rare earth magnet technology and in electronics to provide (relatively) low-cost benchtop Fourier transform instruments. While Varian's patents and designs are condemned to limbo, accounting rules preventing their sale once Agilent had written off their financial value, the hard-won expertise of Varian's engineers, programmers and applications chemists has filtered out into the wider market and continues to contribute to developments in NMR.

In TS Eliot's words – misquoting Emerson – "The lengthened shadow of a man\Is history". In the history of NMR, few shadows are as long as that cast by Russell Varian: a visionary engineer, creative and far-sighted, who anticipated many of the most important developments in NMR. Politically progressive and a lifelong supporter of conservation, he died suddenly in 1959 while hiking in Alaska to scout out locations for new national parks. Many other great men played their part in those developments. Felix Bloch, who shared the 1952 Nobel Prize for Physics with Ed Purcell, contributed both the central insights into the semiclassical behaviour of nuclear magnetism that still inform descriptions of magnetic resonance phenomena today, and the classically simple and appealing idea of sample spinning. Frederick Terman, author of the standard 1940s (and 50s) textbook on radio engineering, the *Radio Engineers' Handbook* [20], taught Russell Varian at Stanford and was successively Dean of Engineering and Provost. As an early director of Varian Associates he was responsible for their move in 1953 to the Stanford Industrial Park, which he created; Varian were joined by Hewlett-Packard, Eastman Kodak General Electric and Lockheed to form the seed from which Silicon Valley grew.

In the history of science, however, great men and women tell only half the story. NMR also prospered when it did and how it did because of a range of outside influences: the growth of nondestructive analysis techniques such as infrared spectroscopy prepared the ground for the ready acceptance of NMR by chemists; the rapid development of radio communication and radar technologies during the Second World War, and the flood of war surplus equipment that followed it, made it much easier to design and build early NMR instruments; and the return of academic scientists from war work with five or six years' worth of stored up ideas [21] reinvigorated many areas of research. Chance also played a major role. Had Russell Varian's uncle not been a patent agent - or had he not shared a room at Stanford with William Hansen – or had Sigurd Varian not learnt at first hand about the problems of aerial navigation while working as a pilot in Mexico and Central America – the history of NMR would have been very different.

Acknowledgements

The author pays tribute to the pioneering contributions made to the development of NMR spectroscopy by the employees of Varian Associates and its successors.

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