1964-5 Norman Sheppard

(First Chairman of the NMRDG)

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Because I was elected the first Chairman of the Group and only for one year there were few meetings during my time in office therefore these recollections will, of necessity, relate to more general experiences.

My first acquaintance with nuclear magnetic resonance (NMR) was when, in late 1948, I visited the Chemistry Department of Harvard University on my way home after a year spent as a Visiting Assistant Professor at Pennsylvania State College (now State University). A friend of mine at Harvard, Gordon Stone, told me of this new technique which was thought to have promise for chemical structural identification. I was puzzled by this because it seemed to me that most of chemistry is related to the properties of the outer electronic orbitals, and that the role of the nuclei was essentially to act as charged point masses around which the electrons are arranged and move. But I had not anticipated the extremely high resolution of the new technique, soon to reach 0.5 Hz or nearly 10^{-10} cm^{-1}. This compares with 0.1 cm^{-1} for the best routine resolution in infrared spectroscopy, the then most effective spectroscopic method for molecular structural determination and my main interest at the time.

Several years later I visited the Texaco Research Laboratory in Beacon, New York State, following a Gordon Research Conference on infrared spectroscopy. I had a mutual interest with Dr. Robert Eischens of Texaco in the infrared spectra of adsorbed molecules. At Beacon I was shown a Varian Associates' brochure which illustrated the ^1H NMR spectrum of ethanol. From this I learned of both the effects of differing chemical (electronic) environments on the resonance positions of a particular type of nucleus (the chemical shift), and the way in which spin-spin coupling fine structure in the resonances provides information about the presence of neighbouring magnetic nuclei. Soon after, I accepted the position of Assistant Director of Research in Spectroscopy in the University Chemical Laboratory. Cambridge. My teaching role in the laboratory was primarily to present molecular spectroscopy to the postgraduate chemistry students from the point of view of structure determination. I suggested to Professor A. R. Todd (later Lord Todd), Head of the Laboratory, that NMR would be a valuable addition to the existing UV-visible and infrared spectroscopic facilities. I recall that he obtained a (then 'huge') grant of £18,000 from the Wellcome Foundation in order to purchase our first ^1H 40 MHz Varian spectrometer. It was installed by Chris Dersch in 1957. I particularly remember this period because Professor Todd had told ICI of the impending delivery of the spectrometer. They had asked if they could bring a 'few' samples for study by this new technique, and I had agreed to this. However, I had not changed the proposed date of their visit even though the delivery of the spectrometer was delayed by several months. They arrived about a fortnight after the installation by which time Ralph Elsey, a senior technician, and I had only run a few samples of our own and were still mastering the technique of "dishing" the magnetic field in order to obtain good resolution. The ICI scientists had brought about 50 samples with them and expected the spectra to be interpreted as they appeared! In fact it took us several months to run all their samples interspersed into the laboratory's own NMR program that soon became very active.

During the next few years NMR techniques developed rapidly through equipment initiatives taken by Varian Associates. The earlier very tiresome field drift was much reduced by a Super Stabilizer electronic device; resolution became optimized by cranking a handle to change the relative orientations of the magnet pole pieces rather than by 'dishing' the field, then sample spinning was introduced to average-out horizontal field inhomogeneities; and in due course (from Perkin-Elmer) 'Golay coils' enabled inhomogeneities in all three dimensions to be optimized by purely electronic means. Variable temperature operation and double resonance techniques all added to the power of NMR.

Jim Shoolery, in particular, played a vastly important role in acquainting the academic chemists with the use and value of the new techniques developed by the Varian engineers and, of course, in persuading the chemists to purchase the new spectrometers or accessories! I recall with amusement a sales pitch that he was making at a meeting in Oxford when he pointed out that, although the cost of the latest 60 MHz spectrometer seemed to be very high, it was capable of providing the unprecedented resolving power of 1 in 10^9. For comparison he pointed out that an optical telescope with such a resolving power would be able to resolve the images of two cats sitting side-by-side on the surface of the Moon. A voice with a strong Scottish brogue interrupted from the back of the lecture-hall, 'I don't believe there *are* two cats on the surface of the Moon!'

. The great success of NMR in structural diagnosis led to a consideration of the desirability of recording spectra in a standard format on printed charts. Up to that time, each spectrum had to be calibrated with sidebands because of field drift, etc. The advantages of such a situation were urged on Perkin-Elmer Limited (UK offshoot of the Perk in-Elmer Corporation of the USA) by a panel of spectroscopic consultants that included Rex Richards, H.W. (Tommy) Thompson, and myself. It was concluded that a permanent magnet-based spectrometer would be expected to have minimum field drift and also be economical in operational costs. Perkin-EImer Limited therefore developed such a 40 MHz spectrometer which was installed in Cambridge for my evaluation. Although this was very successful, its development into quantity production was slow. This was probably because NMR was a new and unfamiliar technique to the parent corporation in the USA. As a result, Varian

Associates were the first to realize this aim by marketing the well-known A-60 routine spectrometer. However, Perkin-Elmer spectrometers were marketed in due course and later developed up to the feasible permanent magnet limit of 90 MHz for ^{1}H nuclei. For research purposes the Varian HA-100 was the workhorse for many years. Subsequently, as is well known, much higher magnetic fields have been achieved using superconducting magnets, many of them produced by Oxford instruments Limited following encouragement from Rex Richards to move into this field. The major advances of pulse techniques coupled with Fourier transform analysis, the improved spectra! separations and sensitivities from the use of high magnetic fields, and (he 2D procedures for spectral analysis developed by Ray Freeman, have together caused NMR to become extremely effective in studying the large molecules of biochemistry, such as enzymes and proteins, in general in solution, again with encouragement from Rex Richards in the UK and from others elsewhere.

When I moved from Cambridge to the University of East Anglia in Norwich in 1964 to help found the School of Chemical Sciences, I carried my NMR interests with me and two other of our newly appointed teaching staff, Robin Harris and Ken Packer, also had strong interests in this area. I vividly remember our new HA-100 spectrometer being installed by crane in our one-storey temporary building after removal of part of the roof!

The interest in NMR grew throughout the UK as more spectrometers became available and several scientific meetings were held by practitioners. It became clear that a proper organisation should be put in place and in 1964, due to the efforts of Eric Mooney and Ernie Cummins, it was decided to form the NMR Discussion Group. Because of their interest in infrared spectroscopy, the format was to be that of the IR Discussion Group. I was elected to be its first Chairman with the late David Whiffen serving as Vice-chairman. In 1965 Rex Richards succeeded me as Chairman.

NMR provides an excellent example of the unity of science and of how the solution of long-standing needs or problems can come from seemingly unrelated directions. But, as in the case of NMR, the applications tend to become apparent only several decades after the original discovery (one and a half decades for NMR in Chemistry and four decades in medicine). Unfortunately, it is difficult for politicians and the public to take into account such timescales (for politicians their own timescale is about five years, i.e. the interval between elections: for financiers the timescale is about one year. Thus there is a real danger that curiosity-oriented research will be allowed to wither for the lack of financial support.