

## **Concluding Remarks**

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## Concluding remarks

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I think that we have all been very impressed by the ingenuity of the experiments described at this Meeting, and the wealth of information now available from n.m.r. experiments in the solid state. It was not always so. In the early days of nuclear magnetic resonance, when we were essentially forced to work in the solid state, because the dipolar interaction was the only one large enough to be measured, the experiments were a little more difficult and rather less rewarding. It happened that I was starting my research at that time (in Sir Rex Richards's group) and all the comparatively straightforward chemical applications of Pake's original work had been done; what was left was the investigation of materials that were either very poisonous, or dangerously explosive, or both. At the conclusion of each experiment we took the sample outside of the laboratory and, after a brief ceremony, buried it in the ground. It was a great relief when Anderson and Arnold showed the way to high-resolution n.m.r. of liquids, and most of us seized the opportunity and abandoned solid state work forever.

Fortunately a few courageous souls carried on. Professor Andrew was one of them, and he went on to introduce and perfect the magic-angle spinning technique which has proved to be of such practical importance in many of the experiments described in this meeting, and may well have been the spur to the invention of the multi-pulse techniques introduced by Professor Mansfield and Professor Waugh. Now Professor Haeberlen tells us that the boom in multi-pulse experiments is over. His actual words were 'there are stringent limitations on the choice of compounds suitable for multi-pulse work.' I would like to suggest to him that he will know for sure that the boom is over when he finds his graduate students burying samples.

The next stage was heteronuclear decoupling used in conjunction with the cross-polarization experiments pioneered by Hartmann and Hahn. An essential feature of this technique is the use of one spin species of low natural abundance, such as <sup>13</sup>C, to ensure that homonuclear dipoledipole interactions are very weak. This 'dilute spin' approach has been used for many years by electron spin resonance spectroscopists, and was used at a very early date to observe the <sup>13</sup>C n.m.r. spectrum in a single crystal of calcite, the classic experiment of Professor Lauterbur. Perhaps after thanking the Science Research Council for equipment grants, one should now also acknowledge the foresight of Providence in getting the natural abundance of <sup>13</sup>C just about right.

The separation of different n.m.r. interactions, for example the dipolar interaction and the chemical shift, is the next important step. It seems that it is now possible to switch on or off the individual terms of the Hamiltonian essentially at will, and separation in the time domain becomes separation in the frequency domain by virtue of two-dimensional Fourier transformation. Thus, paradoxically, it becomes feasible to remove chemical shift anisotropy effects by magic angle spinning and yet nevertheless map out the anisotropic line shape at the same time.

Finally there are the multiple-quantum experiments, so clearly described in Professor Pines's [ 209 ]

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review. By its very nature, a multiple-quantum transition bypasses certain spins in the molecule. It is as if we could observe the proton resonance of a sample that had been specifically deuterated - just as if we had taken the molecule apart and set out to examine the interactions operating in the various fragments. The resulting simplification of the spectrum can be of crucial importance. Professor Pines has provided us with a tantalizing glimpse of a new experiment that concentrates the available intensity into a particular order of multiple-quantum coherence, for example the four-quantum transitions.

It has been a splendid meeting. I bring it now to a close, hurrying back to Oxford to try to disinter some of those old n.m.r. samples....