

Editorial

Despite the 'tyranny of distance' that militated against ready contact between Australian scientists and their counterparts overseas in the early 1960s, Jim Shannon was in the vanguard of the applications of mass spectrometry to structure determination, and it was his seminal contributions and persuasive advocacy that triggered the remarkable growth of organic mass spectrometry 'Down Under'. In this commemorative issue of *Organic Mass Spectrometry* we have included some personal reminiscences of him by three of his contemporaries: Sev Sternhell, Fred McLafferty and Allan Maccoll.

James Stanley Shannon was born in Adelaide, South Australia, on 11 July 1926. After wartime service with the Royal Australian Air Force, Jim graduated in 1949 with first class honours in Chemistry from the University of Adelaide. Following postgraduate studies at Adelaide on the stereochemistry of terpenoid compounds, he was awarded one of the two prestigious British Council Fellowships for 1952. This enabled him to undertake doctoral research overseas at Imperial College London with Professors R. P. Linstead and E. A. Braude on mechanistic and kinetic studies of homogeneous hydrogen-transfer reactions.

After a short period in industry as a development chemist with British Petroleum, Jim joined CSIRO in 1955 at the Division of Coal Research in Sydney, where he pursued basic research studies in the chemistry of polynuclear compounds as models for elucidating coal structure (e.g., R. A. Durie and J. S. Shannon, *Aust. J. Chem.* **11**, 188 (1958); J. S. Shannon, *Fuel*, **37**, 352 (1958)). His research prowess and leadership qualities were quickly appreciated and he rapidly gained promotion to become a Senior Principal Research Scientist by 1963.

These were exhilarating times for the staff of the Division of Coal Research. Its Chief, H. R. Brown (*q.v.* Sev Sternhell's reminiscences), was relentless in his drive to appoint scientists of the highest calibre and to secure the highest status for the many research activities of his Division. Inevitably this led to rivalry and conflict among divisions, and thus there was much cause for concern within Coal Research in 1957-58 when there was a major review under the chairmanship of Sir William Pettingell. Jim Shannon was one of those selected to appear before the review committee and he asserted vigorously that the Division of Coal Research would languish unless it had access to advanced scientific instrumentation such as a mass spectrometer to facilitate its research. The Division came through the review unscathed and the crucial final recommendation of the Pettingell committee was that a mass spectrometer and a nuclear magnetic resonance spectrometer should be purchased forthwith!

Jim had referred in particular to a note on the mass spectra of terpenes (R. I. Reed and P. de Mayo, *Chem. and Ind.* 1481 (1956)) as a justification for mass spectrometry in coal research, and he spent 1959-60 with Reed's group in Glasgow. This was one of the few mass spectrometry laboratories in the world where fundamental studies on the applications to molecular structure determination had begun. He spent the time familiarizing himself with the problems that can befall the callow mass spectroscopist. He recalls how samples were introduced to the ion source of the Glasgow mass spectrometer (an AEI MS2) by way of a home-made direct insertion probe without a vacuum lock, whereupon the brass probe was sealed in place through the source housing with the aid of picene wax! Thus, when Jim travelled to Bremen to view the Atlas CH4 instrument under construction for Coal Research, he immediately recognized the potential benefits of the high-temperature ion source, even though it was specifically designed for inorganic samples, and therefore arranged for its installation on the mass spectrometer destined for Australia. The inclusion of this source proved to be of considerable advantage in subsequent applications of the Coal Research mass spectrometer to less volatile organic compounds.

When the Atlas CH4 instrument was installed in Sydney in 1961, it was the only mass spectrometer in Australia and New Zealand devoted to the elucidation of the structures of organic compounds. Consequently, Jim was inundated with requests and samples from every university and many other research establishments in Australasia. His reputation rapidly spread as a mass spectroscopist who resolved cryptic problems of molecular structure, and requests from organic chemists came from far and wide (*q.v.* Allan Maccoll's reminiscences).



Jim Shannon (1961) at CSIRO Division of Coal Research, Sydney. The newly installed Atlas CH4 mass spectrometer was the first in Australia devoted to organic mass spectrometry.

In those pioneering days of the 1960s, there were many instances, amply exemplified in his publication record, where Jim's major contributions to the collaborative studies were the applications of mass spectrometry to characterize organic compounds *de novo* and to decipher the structures of complete unknowns, or to correct structures that had been previously assigned erroneously. For instance, he solved almost entirely by mass spectrometry the complex and unique structure of the fungal metabolite spirodesmin C in which three sulphur atoms were linked together in an eight-membered ring (R. Hodges and J. S. Shannon, *Aust. J. Chem.* **19**, 1059 (1966)). He published the structural assignments to saturated triterpenoid compounds using mass spectrometry (e.g., J. L. Courtney and J. S. Shannon, *Tetrahedron Lett.* No. 1, 13 (1963)) and to cyclodepsipeptides (e.g., D. W. Russell, C. G. Macdonald and J. S. Shannon, *Tetrahedron Lett.* No. 38, 2739 (1964)). In 1964, he used mass spectrometry to elucidate the structure of zeatin, the first plant cell growth factor isolated in pure form (D. S. Letham, J. S. Shannon and I. R. McDonald, *Proc. Chem. Soc.* 230 (1964)). He announced the correct structure for zeatin that year at an invited lecture in New Zealand under the auspices of the New Zealand Institute of Chemistry, whereupon the success was publicized in an editorial in the *Auckland Times* (the first part of the editorial described how the great New Zealand runner Peter Snell had just created a new world record)!

Jim was a forceful and eloquent advocate of the need to gain a better understanding of the mechanisms of gas-phase ion fragmentations. Thus, his first publication in mass spectrometry (J. S. Shannon, *Aust. J. Chem.* **15**, 265 (1962)) described the use of metastable peaks and *in situ* isotopic labelling to elucidate the ion fragmentations of benzyl alcohol and its *ortho*-, *meta*- and *para*-hydroxy isomers. This paper was closely followed by a second in which the occurrence of random hydrogen rearrangements in aromatic molecular ions was discovered (C. G. Macdonald and J. S. Shannon, *Aust. J. Chem.* **15**, 771 (1962)). Likewise, deuterium labelling was

used to reveal stereospecific 1,3- and 1,4-eliminations from the molecular ions of cyclic alcohols and acetates (C. G. Macdonald, J. S. Shannon and G. Sugowdz, *Tetrahedron Lett.* No. 13, 807 (1963)).

At the same time, Jim realized that there was an urgent need for the general adoption of appropriate symbols to characterize ion structures and electron shifts in mass spectrometry. Thus, he proposed a new ion charge symbolism (J. S. Shannon, *Proceedings of the Royal Aust. Chem. Inst.*, 323 (1964)) that would make apparent the odd- or even-electron character of ions and promote rigorous electron book-keeping. He denoted the radical character of molecular ions and other odd-electron ions with the superscript⁺ symbol and he used fish-hooks and arrows to distinguish one-electron and two-electron transfers, respectively. The new symbolism quickly gained international recognition (*q.v.* Fred McLafferty's reminiscences) and is now universally accepted in organic mass spectrometry.

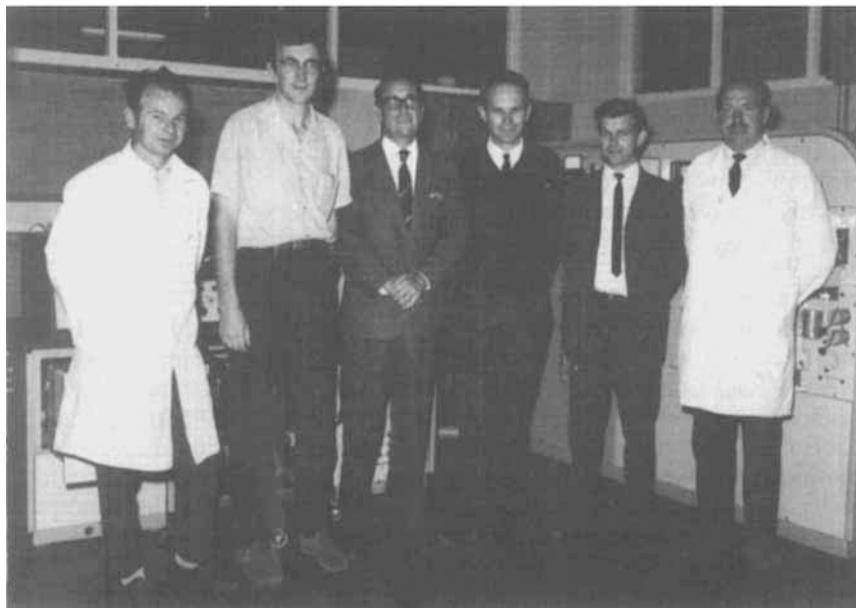
He broadened the scope of mass spectrometry by applying it to metal coordination compounds, including those having previously unresolved or incorrectly assigned structures (e.g., H. G. Ang, J. S. Shannon and B. O. West, *Chem. Commun.* 10 (1965); S. H. Chaston, S. E. Livingstone, T. N. Lockyer and J. S. Shannon, *Aust. J. Chem.* **18**, 1539 (1965)). During the course of these studies, he recognized that the modes of ion dissociation of the metal complexes were markedly dependent on the valency states normally assumed by the metal atom. He therefore proposed the concept of valency change to explain the peculiar fragmentation pathways of coordination compounds (J. S. Shannon and J. M. Swan, *Chem. Commun.* 33 (1965)) and applied it extensively, *inter alia*, in rationalizing the mass spectra of metal acetylacetonates (C. G. Macdonald and J. S. Shannon, *Aust. J. Chem.* **19**, 1545 (1966)). During the course of this latter study, he used negative-ion mass spectrometry to demonstrate the existence of the trimer of calcium acetylacetonate in its un-ionized vapour.

In recognition of his achievements in the development and application of mass spectrometry to structural problems in organic, biological and coordination chemistry, Jim was awarded the H. G. Smith Medal for 1967 by The Royal Australian Chemical Institute. Also in 1967 he was awarded the honorary degree of Doctor of Science by the University of Adelaide. But it was the same year that the forces opposing the policies of the Chief of the Division of Coal Research eventually became victorious, overwhelming H. R. Brown and dispersing the research teams. The Mass Spectrometry Unit transferred to Canberra in 1968, together with its newly purchased double-



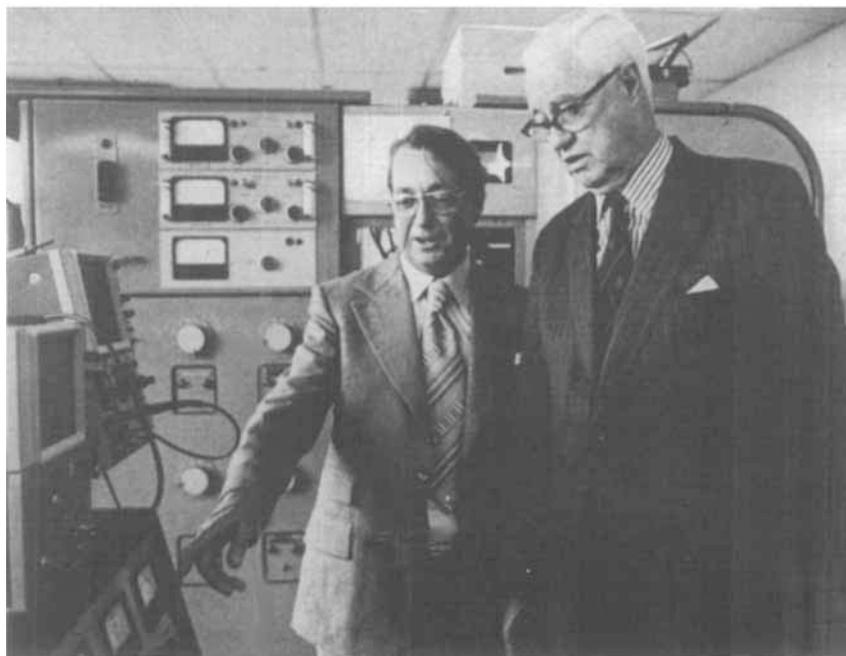
Meeting in London, 1968, of Jim Shannon and Allan Maccoll (centre pair) with Gunther Heyden (right) and staff regarding the inauguration of the journal *Organic Mass Spectrometry*. It was decided that Allan would be the Editor-in-Chief and Jim the Regional Editor for Australasia.

focusing mass spectrometer (AEI MS902), to provide research and support in the biological sciences. 1968 was also the year that *Organic Mass Spectrometry* was inaugurated. Jim was involved in its inception and was the obvious choice as its Australasian Editor, contributing a paper from his research group to its first issue.



Jim Shannon and his mass spectrometry group (1968) at CSIRO Division of Entomology, Canberra. *Left to right:* Ivan Pavelic, Tom Bellas, Jim Shannon, Colin Macdonald, Mike Lacey, Tony Daley.

In 1969, Jim was appointed to a personal chair as Professor of Chemistry at the University of New South Wales in Sydney, although he maintained his collaboration with the remaining group in Canberra on fundamental studies in ion fragmentations. Jim soon acquired sufficient funds for the School of Chemistry to purchase one of the first mass spectrometers outside



Jim Shannon showing Lord Todd the newly installed GEC-AEI MS902 mass spectrometer at the School of Chemistry, University of New South Wales, 1972. The instrument was the first in Australia devoted to research in chemical ionization mass spectrometry.

the U.S.A. and Europe that was devoted to research and applications of the newly emergent field of chemical ionization. He recognized the intriguing skeletal rearrangements of $[M + H]^+$ ions that could occur in the chemical ionization mass spectra of ethers (V. Diakiw, J. S. Shannon, M. J. Lacey and C. G. Macdonald, *Org. Mass Spectrom.* **14**, 58 (1979)) and he focused his research efforts on their elucidation (reviewed by E. E. Kingston, J. S. Shannon and M. J. Lacey, *Org. Mass Spectrom.* **18**, 183 (1983)).



Jim Shannon and his mass spectrometry group (1980) at the School of Chemistry, University of New South Wales. *Left to right:* Patrick Gordon, Reg Diakiw, Jim Shannon, Joe Brophy, Eric Kingston, Derek Nelson, Jerry Bartnicki.

Jim's leadership skills came to the fore at the University of New South Wales and he became increasingly involved in academic administration. He served as Head of the School of Chemistry in 1976–77 and subsequently 1980–84. He was on innumerable committees of the Professorial Board of the University and served as Chairman of the Research and Higher Awards Committee and Chairman of the Board of Studies in Science and Mathematics. He was elected to the University Council and represented the Faculty of Science.

Jim's influence on mass spectrometry at the University of New South Wales was equally great. In 1980 and 1981, Jim masterminded the transfer from La Trobe University, Melbourne, to the University of New South Wales, Sydney, of the grand-scale double-focusing mass spectrometer, as it was known in Melbourne (shortened to an acronym MMM in Sydney). The move itself was organized and supervised by Mr John Chippindall. The negotiations between Universities and within the University of New South Wales were handled by Jim, and the latter were significant because they resulted in the conversion of substantial areas of teaching laboratories into state-of-the-art mass spectrometry research laboratories.

The commitment made at that time by the University of New South Wales to mass spectrometry research was one that would last for a decade or more. The commitment to research in mass spectrometry was redoubled following an initiative by Jim and one of the undersigned; which, in hindsight, is reminiscent of Jim's success many years before with the Pettingell committee. Through coupling the arguments in favour of the acquisition of a 500 MHz NMR spectrometer with those for a Fourier transform ion cyclotron resonance (FT/ICR) spectrometer, it was possible to persuade the University to buy both of these instruments from Bruker. The FT/ICR was installed in 1985–86, and prominent among the varied applications have



Jim Shannon and John Chippindall (1981) outside the School of Chemistry, University of New South Wales as the magnet of MMM is installed.

been studies of Buckminsterfullerenes, in collaboration with CSIRO's present-day Division of Coal Research.

Jim Shannon retired in 1987 and was appointed Emeritus Professor. This issue of *Organic Mass Spectrometry* commemorates his major contributions to mass spectrometry, and reflects the esteem and high regard in which he is held by his contemporaries, colleagues and ex-students.

Peter J. Derrick

Michael J. Lacey

Colin G. Macdonald