Brief History of the Potassium-Argon Dating Laboratory in the ANU

1960 to 2000

The laboratory was initiated by Professor J C Jaeger, head of the Department of Geophysics, Research School of Physical Sciences (RSPhysS), in 1960, the first K/Ar laboratory in Australia. There are now four laboratories in Australia, mainly devoted to age measurement on rocks by the $^{40}Ar/^{39}Ar$ variation of the conventional K/Ar method. After a visit by Dr Garniss Curtis of the Department of Geology and Geophysics, University of California, in ~1958 for a month or so, Jaeger was impressed by the new approach to potassium-argon dating that Curtis and colleagues were initiating. Jaeger, who already had good ties with people in geology at the University of California, Berkeley, decided to initiate K/Ar dating in ANU. When the existing Department of Radiochemistry in RSPhysS was shutdown in ~1959, Jaeger took on Dr John Richards and his Metropolitan Vickers gas–source mass spectrometer, on which Richards had been measuring lead isotopes by the lead tetramethyl method. Dr W Compston converted this machine to a thermal ionization machine on his appointment by Jaeger in 1960 to undertake Rb/Sr dating. At much the same time Jaeger invited Dr Jack Evernden, a colleague of Garniss Curtis, to visit ANU for ~6 months to work on the installation of an argon extraction line and a Reynolds mass spectrometer that Jaeger had ordered from Dr J Reynolds in the Department of Physics, University of California, Berkeley. This was successfully accomplished during 1960-61 by Richards and Evernden, and resulted in the first major paper on the application of the K/Ar dating technique, mainly to Australian rocks (Evernden & Richards, 1962. Potassium-argon ages in eastern Australia. Journal of the Geological Society of Australia, **9**, 1-49). This landmark paper heralded many years of a high level of production of data by the K/Ar method to a host of geological problems in Australia and the wider world.

Ian McDougall was a PhD student in the Department of Geophysics at this time and was persuaded by Jaeger to go to Berkeley for a year to Curtis and Evernden's laboratory to learn the K/Ar technique directly from the leaders in the field at the time. McDougall was supported by a CSIRO overseas post-doctoral fellowship in this endeavour. An outcome of this visit was the successful measurement of K/Ar ages on plagioclase, pyroxene, K-feldspar and whole rock from the Red Hill intrusion in Tasmania, showing that emplacement had occurred in the Jurassic (McDougall, 1961. Determination of the age of a basic igneous intrusion by the potassium-argon method. Nature, **190**, 1184-1186). McDougall had completed a petrological study of the Red Hill intrusion in 1960 for his PhD. Although a geologist with relatively little knowledge of K/Ar methods or mass spectrometers, McDougall rapidly acquired the necessary know how from the remarkable trio at Berkeley, Curtis and Reynolds as well as Evernden on his return from Australia. Jaeger appointed McDougall to a Research Fellowship in his department, presumably in part to have geological knowledge within the small K/Ar group headed by John Richards, a chemist by training. On his way back to Australia to take up the appointment, McDougall spent a month collecting samples from the volcanoes of the Hawaiian island chain for possible dating. Although, at this time it was thought to be quite impractical to measure ages on rocks of youthful volcanoes, as in Hawaii, Jaeger typically was an enthusiastic supporter of the project. That the physical K/Ar age measurement on Hawaiian turned out to be so successful, showing that volcanism had in fact progressed along the chain from at least Kauai in the NNW via Oahu, Molokai and Maui to the present activity on the big island of Hawaii at the ESE of the chain, just as many previous workers had argued, but that rates could now be calculated at 10-15 cm per year. Results from this work, initially published by McDougall in 1964 (McDougall, 1964. Potassium-argon ages from lavas of the Hawaiian Islands. Bulletin of the Geological Society of America, **75**, 107-128), also lead to involvement in developing the geomagnetic polarity time scale, as a PhD student of Ted Irving's in the Department of Geophysics, ANU, also collected from many of the same lava sequences as McDougall in the Hawaiian Islands for palaeomagnetic measurements, mainly to test models of secular variation. In this work, Don Tarling measured the actual direction of magnetization, which also resulted in the determination of polarity, which is normal or reverse, compared with the present geomagnetic field (McDougall & Tarling, 1963. Dating of polarity zones in the Hawaiian Islands. Nature, **200**, 54-56). Ultimately this work with various colleagues and with the team at the US Geological Survey, lead to confirmation that the geomagnetic field had changed polarity in the past and to the geomagnetic polarity time scale. This was then applied to the magnetic stripes seen across mid-ocean ridges, in turn leading to the acceptance of the new paradigm of seafloor spreading and the plate tectonic hypothesis. Meanwhile, John Richards continued his active role in the laboratory, mainly dating Australian rocks, particularly granitic rocks, by the K/Ar method.

The arrival of the Nuclide thermal ionization mass spectrometer for the isotope laboratory and the development of lead isotope measurements by thermal ionization methods, resulted in John Richards returning to his lead isotope studies and ultimately his withdrawal from the K/Ar laboratory, leaving McDougall to run that facility. McDougall set about commissioning a second Reynolds mass spectrometer in the same facilities in the old hospital buildings across from the Menzies Library. When the new Geochemistry building, on the present site of Research School of Earth Sciences (RSES), became available in the late 1960s, the two Reynolds machines, were carefully transferred to the basement of the new building, the current site of the VG1200 gas-source mass spectrometer used for Ar/Ar dating that was originally purchased in about 1978. Subsequent to Reynolds suggesting that his glass enveloped machine could be operated statically, that is with the valve to the pumps closed, the amount of argon gas that could be successfully isotopically analysed was reduced dramatically, so that the amount of rock required for extraction of argon for analysis also decreased markedly. All current laboratories, mainly Ar/Ar laboratories these days, utilize similar measurement techniques, and sealing systems have resulted in such improvement that metal machines have largely replaced the glass machines developed by Reynolds. On the move to the new building, space was allocated in a room immediately above the basement for extraction of argon for K/Ar dating purposes. Potassium was measured by flame photometry from the earliest days and this continued until about 2004, when the potassium dissolution laboratory and the flame photometry room were taken over for other purposes. In the late 1960s or early 1970s an MS10 was purchased for the isotopic analysis of argon. This was attached directly to one of the argon extraction lines, and became the main work horse for analysis of the argon from rocks for conventional K/Ar dating, until it was removed from the laboratory in ~2007. McDougall retired from ANU in 2000, and much of the infrastructure developed by him was subsequently removed, except for the VG1200 machine, which still produces good data under its present management.

During the early 1970s, McDougall, supported by technical help provided by Zarko Roksandic, developed the Ar/Ar method of dating rocks, utilizing the MS10 followed by the VG1200 for the isotopic analysis of the extracted argon, after irradiation in the HIFAR nuclear reactor at Lucas Heights, south of Sydney in New South Wales. Although irradiations had to be done outside the core of the reactor where gradients of the fast neutron flux, required for the conversion of some of the ^{39}K to ^{39}Ar, were large, it became possible to monitor the gradient very well. In fact the gradient was reduced to a few percent along the irradiation can by the relatively simple expedient of inverting the can exactly half way through the designated irradiation time. A somewhat similar situation applies to the replacement reactor on the same site, OPAL, but this remains to be tested. In the 1980s it became possible to measure single crystals by the Ar/Ar method, because of great improvements in the residual background in the metal mass spectrometers as well as using electron multipliers to detect the small argon ion beams.

In the early 1990s a new machine was installed, a VG3600, which had greater sensitivity than the VG1200, a new extraction line was built adjacent to the machine, together with installation of a continuous wave laser and the whole system automated, principally through the efforts of Tony Doulgeris, whose technical assistance was paramount. This system, which included a double vacuum resistance furnace as well as the laser, was successfully utilized for over a decade, before the whole system was removed in about 2007, and the laser subsequently given to Earth Sciences in the University of Queensland. From about 1985 a Research Fellowship was allocated to McDougall for argon dating purposes, and a succession of appointments were made, including Sue Baldwin, Terry Spell and Jim Dunlap, all of whom did excellent work, mainly associated with thermal histories of core complexes in New Zealand and Papua New Guinea. There were also a number of visitors who used the laboratory facilities successfully on very useful projects. Altogether, over this period of 1960 to 2000 over 200 papers were published by members of this group on a huge variety of projects that were of local, regional or of world wide significance. Of course, during this period there were many PhD students who undertook significant studies and contributed to the growing reputation of the laboratory. While all did remarkably good work using the facilities provided, mention will be made of a few of the more outstanding achievements. Thus in the early 1970s Peter Wellman in studying the age of the many sites of Cenozoic volcanism in eastern Australia, recognized that the central volcanoes showed a younging to the south at a rate and in a direction commensurate with the movement of Australia to the NNE under the plate tectonics hypothesis. Subsequently Bob Duncan measured K/Ar ages on volcanic island chains in the Pacific Ocean, mainly the Society, Austral and Marquesas chains, showing that the rate and direction of migration of the volcanism in these island chains were concordant and were consistent with plate tectonic models for the Pacific plate. Thus, it was the K/Ar data that provided information on the plate motions, now to some extent superseded by modern GPS measurements in the islands themselves. Nevertheless, this work is still giving information on the migration of volcanism at much earlier times. The studies undertaken by T Mark Harrison in the late 1970s on the resetting of the K/Ar system owing to the emplacement of a granitic body at Separation Point in the south island of New Zealand is also particularly notable. In addition to demonstrating that the K/Ar system responded to heating owing to the later emplacement of the granite, he was able to show that some of the hornblendes absorbed much excess argon at their margins by careful bulk analysis of argon by Ar/Ar step heating (Harrison & McDougall, 1980. Investigations of an intrusive contact, northwest Nelson, New Zealand I & II. Geochimica et Cosmochimica Acta **44**, 1985-2003). These are just a few of the more notable contributions made by students in the group. A number of other students did much of their work using the K/Ar or Ar/Ar facilities, although they were not formally supervised within the group.

McDougall and Harrison published a monograph on the $^{40}Ar/^{39}Ar$ technique of dating in 1988 and an updated second edition was published in 1999 (McDougall & Harrison, 1999. Geochronology and Thermochronology by the $^{40}Ar/^{39}Ar$ Method. 2nd edition, Oxford University Press, New York, 269pp.)

In 1978 McDougall became involved in the measurement of ages in the stratigraphic sequences of the Omo-Turkana Basin in East Africa, to determine the history of deposition and the age of the many hominin fossils recovered from the subaerially exposed sedimentary sequences up to 800 m thick mapped in the region. The latest of the many papers describing this work, with over 35 levels now reliably dated with ages in the range of 4.25 to 0.1 Ma, using mainly alkali feldspar from tuffaceous beds within the stratigraphic sequences, was published recently (McDougall et al., 2012. New single crystal $^{40}Ar/^{39}Ar$ ages improve time scale for deposition of the Omo Group, Omo-Turkana Basin, East Africa. Journal of the Geological Society, London, **169**, 213-226).

In the late 1980s, through the encouragement of Professor Ted Ringwood, and support of the Director at the time, Professor Kurt Lambeck, McDougall became responsible for the setting up of a Noble Gas Geochemistry laboratory. This entailed the purchase of a VG5400 gas source mass spectrometer, the appointment of Masahiko Honda to an academic position to facilitate the research program which initially involved design and building of an extraction line attached to the mass spectrometer. Once this was accomplished a program of analysis was begun. Separation of the noble gases from one another was possible through the use of a cryogenic apparatus built in the RSES workshop, so that the analysis of the isotopic composition of He, Ne, Ar, Kr and Xe was routinely undertaken. Initial results on gases extracted from deep sea basalts showed that a component of solar composition was identified leading to the important inference that gases of solar origin were incorporated in the Earth when it formed. This was the basis for much of the later noble gas work, which continues to this day, although many other topics have also been investigated successfully. This noble gas facility became a separate entity when McDougall retired at the end of year 2000.

The isotopic dating of rocks by the K/Ar system continued as the department founded and built by Professor Jaeger morphed into a Department of Geophysics and Geochemistry within RSPhysS, and when it separated from RSPhysS and became the Research School of Earth Sciences (RSES) in 1973, with Professor Anton Hales appointed as the first director. Altogether a proud history that continues today under Dr Marnie Forster and Professor Gordon Lister.