

JOHN HILL THERMOCHEMISTRY TREE

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‘Thermodynamics’ and its experimental manifestation ‘thermochemistry’ have never been popular science study areas in schools and higher education institutions (**P 142**) despite their obvious correlation with contemporary concerns on energy production versus consumption and in recent times, with concerns relating to the world-wide ‘energy crisis’ and the looming consequences of ‘global warming’. So it was somewhat surprising that I chose to undertake a thermochemistry research project for my PhD at Battersea College of Technology (BCT) London in late 1963. My supervisor in the Department of Chemistry was Roger Irving. Roger had recently returned from sabbatical leave at the Thermochemical Centre/University of Lund, Sweden and he brought back with him a prototype calorimeter along with a set of plans for setting up a solution calorimetry system at BCT and he was eager for a graduate student to assist him and to test the system. He convinced me that data analysis in thermochemistry was ‘relatively simple’ and only involved the first law (Hess’s Law) of thermodynamics, which states that ‘enthalpies’ are ‘state functions’ and are ‘additive’. However, he did point out that ‘precision’ was paramount in calorimetric measurements and I quickly had to understand the difference between ‘precision’ and ‘accuracy’. Since the proposed project was pioneering in scope and challenging in content, I decided to accept it, particularly since the determination of metal - ligand bond energies was the ultimate objective and this had never been achieved previously by solution calorimetry.

The Irving solution calorimetric system (Mk1) established circa 1963 - 4 is best described as ‘primitive’ with much of the periphery equipment, such as the calorimeter thermostat the calorimetric calibration system and the wheatstone bridge temperature measuring system were ‘in-house’ constructions by Roger initially and later (Mk2) by Ron Schulz – a fellow PhD student in the Irving group. Only the ‘precision potentiometer’, ‘galvanometer’, ‘calorimeter thermostat temperature controller’ and the essential stopwatch were obtained commercially. The crucial glass ampoule inserts into the calorimeter had to be ‘hand-blown’ into a mould and the weighing thereof was achieved using a pan microbalance which had to be calibrated with ‘standard weights’. It became obvious that my glassblowing skills were limited and my patience with balance calibration was seriously tested. Time data were obtained using a precision stop-watch. Temperature/time data output were recorded and graphed by hand since commercial chart recorders were only just becoming readily available at that time along with a paucity of electronic calculators which did not have data printout systems. However, despite these compromises and limitations, the overall system worked well and produced thermochemical data of admirable precision (**P 1**).

‘Metal-ligand bond energies’ had previously only been derived from combustion enthalpy measurements of coordination compounds using a bomb calorimeter. This was and still is a very tedious and time-consuming experimental exercise and is associated with multiple errors. By comparison, the solution calorimetric method is less tedious and more reliable than the combustion counterpart and later applications have shown that it is applicable to a wider range of compounds, particularly inorganic compounds. My PhD project produced 5 publications (**P 1 - 4** and **7**), the first four of which relate to the solution calorimetric determination of the formation enthalpies of Al(III), Cr(III), Fe(III) and Mn(III)–2,4-

pentanedionate complexes with subsequent derivation of the ‘homolytic’ and ‘heterolytic’ metal-oxygen bond energies and correlation of these with ‘crystal field theory’ predictions.

My contemporaries at this time were Ted Charsley, Peter Laye, Ron Schulz, Geoff Walter and Hugh McKerrell, the latter three of which all worked on thermochemistry research projects with Roger Irving. Ted Charsley was a postgraduate student working with John Redfern, who was developing thermal analysis techniques and had a prototype simultaneous thermogravimetric analysis (TGA) – differential thermal analysis (DTA) system, which was based on a Stanton HT-D thermobalance set up in the BCT Chemistry Department. The Redfern/Charsley collaboration in the early 1960’s led to major pioneering developments in this emerging field and in conjunction with the Irving thermochemistry group laid the foundation of a unique thermal studies centre at BCT. In the late 1960’s, Redfern left BCT to take up a partnership/directorship of ‘Stanton Instruments Ltd.’, based in Wimbledon, who were to become the leading UK manufacturer of thermal analysis instruments.

In 1967, I was awarded a NATO research fellowship which allowed me to accept a post-doctoral one year appointment in the Thermochemical Centre/University of Lund, Sweden with Professor Ingemar Wadsö as mentor. This world-renowned centre was an established centre of excellence in bomb calorimetry (Srunner) and solution/titration calorimetry and microcalorimetry (Wadsö) and had an advisory/collaborative agreement with ‘LKB Produkter AB’, Stockholm, who were a recognised leading manufacturer of scientific equipment. In particular at this time, LKB was developing a solution calorimetric system which could be adapted for titration calorimetry. This prototype system overcame all the shortcomings and tediousness of the BCT system used previously, particularly since temperature/time data output was automatically recorded on a chart recorder. Wadsö had already established tris(hydroxymethyl)aminomethane (‘TRIS’) as a calibrant for solution calorimetry via determination of the enthalpy of the TRIS/HCl neutralisation reaction at 298K. I was fortunate to be able to extend this study by determining this parameter at 0, 15 and 30°C (**P 8**), thereby endorsing ‘TRIS’ as a suitable solution calorimetry calibrant over a wide operating temperature range. Also, I was introduced to ‘vaporisation calorimetry’ and was involved in the determination of the vaporisation enthalpy of N, N, N-triacetylammonia (**P 5**) and subsequently calculated a suite of thermochemical properties of this compound.

My contemporary post-doctoral fellows at Lund were Peter Howard, Paul Monk, Peter Sellers, Pepe Layenef and Haru Watanabe, all of whom had Stig Stunner or Ingemar Wadsö as mentors.

At the beginning of 1968, I was awarded an NSF research fellowship which allowed me to accept a post-doctoral appointment in the thermochemistry laboratory/University of Louisville, USA with Professor Loren G. Hepler as mentor. Loren had built what he called ‘a large scale solution calorimetric system’ with the calorimeter being of five litres capacity and the overall system was developed ‘in-house’, which he had brought to Louisville from his previous position at Pittsburgh. I and a fellow PDF Ian Worsley used this somewhat primitive system to determine the ‘distribution coefficient’ of bromine in water and carbon tetrachloride (**P 6**). This was the first time that calorimetry had been used to determine this parameter. Loren was also a prolific writer of reviews of the thermodynamic properties of the elements and I was involved with the literature search of the thermodynamic properties of vanadium, niobium and tantalum (**P 9**). It was this exercise which gave me considerable confidence in ‘review writing’ in the future, despite involving exhaustive literature scanning without the aid of computers or the now common ‘search programmes’.

My contemporary post-doctoral fellows at Louisville were Ian Worsley, Jim Pearce and ‘Rajagopalan’. The higher degree students in the Hepler group at that time were Tom Zordan and John Larsson both of whom had transferred to Louisville with Hepler from Pittsburgh.

In mid-1969, I was awarded a further NSF research fellowship which allowed me to accept a one year post-doctoral appointment at the thermochemical centre/Brigham Young University, Provo, Utah with Professors Reed Izatt and Jim Christensen as mentors. This world-renowned thermochemical centre was an established centre of excellence in 'titration calorimetry' ('thermometric titrimetry') and at this time was embarking on an intensive study of the 'ion-binding' thermodynamics of metal ions with synthetic macrocyclic compounds. The latter had been discovered by Pedersen in the mid -1950's. I was involved with writing a review of this new field (**P 10**) and in synthesising many new macrocyclic compounds to be used in subsequent titration calorimetric studies. Later in 1994, I was invited to contribute to a book chapter titled 'Continuous Titration Calorimetry' in 'Solution Calorimetry'/ 'Experimental Thermodynamics', published by IUPAC (**P 158**) as a consequence of my experience in titration calorimetry gained at BYU.

Izatt and Christensen had an advisory/collaborative agreement with 'Tronac Inc.' based in Orem. At that time, this company were developing a thermometric titrimetry system based on the multiple 'in-house' constructions operating very successfully in the Izatt/Christensen thermochemistry centre for many years. The Tronac titration calorimetry system was later installed in the thermochemistry centre in the Chemistry Department of La Trobe University, Melbourne and had an operating life-span of some 20 years.

My contemporary post-doctoral fellows at Provo were Austin Yingst and Ian Worsley and higher degree students in the Izatt/Christensen group at that time were Barry Haymore, Lyn Kimball, Dennis Nelson and Dale Slade.

In September, 1970 I moved to Melbourne, Australia and accepted the position of Research Fellow in the Chemistry Department of La Trobe University. Over the next 20 years, I supervised many Honours and Higher Degree students undertaking projects in thermochemistry, which included studies in solution and titration calorimetry, simultaneous thermal analysis (STA) and differential scanning calorimetry (DSC). Initially, the solution calorimetry system was an 'in-house' construction, which was superseded by a Tronac solution/titration calorimetric system. The STA system employed was a Rigaku-Denki (Japan) commercial instrument.

Honours projects supervised included thermal analysis of coordination compounds – Ni(II) and Cu(II) thiourea chloride complexes (Martin Attard) (**P 47, 78**) : titration calorimetric studies – phenols, Fe(III) and Ag(I) ferricyanide complexes (Susan Korce) (**P 97, 99, 137**), Cd(II) ferricyanide and Ag(I) thiosulphate systems (Sharon Lim) (**121, 122**) : solution calorimetric studies of metal dithiocarbamate complexes (David McIvor) (**P 15**) and sublimation enthalpy determination of metal 2, 4 – pentanedionate complexes (John Sachinidis) (**P 39**).

MSc projects supervised included an exhaustive thermal analysis study of tin dithiocarbamate complexes (Graham Bratspies) (**P 21 – 24, 28, 34, 41, 42, 92**). In conjunction with academic colleague John Smith, who was a specialist in mass spectrometry, Graham developed a 'novel' thermal analysis technique whereby the 'sample' was flash-heated in the mass spectrometer probe and the decomposition products analysed 'in-situ'. This technique was later formally approved by the International Confederation of Thermal Analysis and Calorimetry (ICTAC) and named 'Programmed Probe Analysis' (PPA). John Murray was an academic member of the chemistry staff at Ballarat College of Advanced Education (later to become University of Ballarat and is now Federation University) and he determined the sublimation enthalpies of a series of metal 2, 4 – pentanedione complexes by DSC, initially proposing benzoic acid as a new calibration standard for this technique (**P 40, 57, 62, 82, 83, 152 – 5**).

PhD projects supervised included thermal analysis of Cu (II) dithiocarbamate complexes and Ni (II) and Pd (II) xanthate complexes (Greg Sceney) (**P 11, 12, 18, 20**) : thermal analysis of Ni (II) alkyl xanthate complexes, sublimation enthalpies of Ni (II), Cu (II) and Co (III) dithiocarbamate complexes by

DSC, and standard formation enthalpy of Ni (II) and Co (III) diethyldithiocarbamate by solution calorimetry and subsequent derivation of the nickel-sulphur bond energy (Kingsley Cavell) (**P 11, 14, 36, 37, 40, 43, 146**); thermal analysis of metal dithiocarbamate complexes and organometallic complexes (Bookhari Annar) (**P 13, 15, 17**); thermometric titrimetric studies of the complexation of alkaline earth metal ions with linear and macrocyclic polyamino carboxylic acids (Gavin Ewin) (**P 46, 55, 73**); DTA study of cis-platin (Stephen Roe) (**P 77**); DTA studies of phenols and binary phenol systems (Nellie Buckman) (**P 106, 107, 110, 113 – 5, 143**); thermometric titrimetric studies of the interaction of metal ions with metallochromic indicators (Rozali bin Othman) (**P 87, 88, 110**) ; thermal analysis studies of Victorian brown coal (Shengping Ma) (**P 72, 98, 102 – 5, 111, 126, 127, 134**).

During my tenure in Chemistry at La Trobe University, two periods of sabbatical leave were taken, the first of which was with the Loren G. Hepler thermochemistry group at the University of Lethbridge in 1978. Loren had received extensive funding from the Alberta Oil Sands Authority to undertake thermochemical studies on ‘oil sands’, which supported my accommodation costs in Lethbridge. I collaborated with Earl Woolley – a recent PhD graduate from the Izatt/Christensen thermochemical centre at BYU, to undertake thermodynamic studies of aqueous iodic acid (**P 27**). A contemporary postdoctoral fellow at Lethbridge was Inger Olofsson, sister of Gerd Olofsson, who was co-director with Stig Sunner of the Lund Thermochemical Centre. The second period of sabbatical leave was taken in 1986 and involved 3 months with the Arthur Finch thermochemistry group in the Department of Chemistry at Royal Holloway College/University of London, Egham, UK. I was involved in the derivation of the thermochemical properties of substituted ureas and pyridinium bromide using an LKB solution calorimetric system (**P 38, 116, 117**). A further 3 months was spent in the Research and Development laboratory at Stanton-Redcroft Ltd., Wimbledon. At this time, Ted Charsley was the head of this laboratory. I was extensively involved in a TMA study of a variety of compounds and in an investigation of potential TMA temperature calibration standards (**P 72, 107, 129, 133**). This was my first introduction to the principles and applications of a ‘less common’ thermal analysis technique.

In 1989, Stanton-Redcroft sponsored my visit to Chulalongkorn University, Bangkok and I gave a short course of 8 lectures on ‘Thermal analysis – techniques and applications’ in the Chemistry Department there to promote thermal analysis techniques and to reveal their wide range of possible applications in many fields of particular significance to ‘developing countries’ (**P 100**). It was fortuitous that at that time, Professor Siri Varothai was Head of Chemistry at ‘Chula’, since we were both PhD students at Battersea College of Technology in the mid -1960’s. I subsequently collaborated with Sopa Chirawongaram, Director of the Scientific and Technological Research Equipment Centre (STREC) at ‘Chula’ to promote thermal analysis education and research opportunities in Thailand (**P 140**).

In mid-1990, I was seconded from La Trobe Chemistry to the Department of Chemistry, National University of Singapore as a Senior Teaching Fellow for 4 years. Whilst I was not committed to undertake research in this role, I collaborated with several members of the academic staff and with their research students undertaking projects in thermochemistry (**P 124, 125, 136, 138, 139, 144 and 145**). My most significant achievement whilst at NUS was a successful DSc submission to the University of London based mainly on my thermochemistry research profile (**P 132**) which was conferred in London in 1992.

From mid-1994 until I retired at the end of 2005, I was redeployed to La Trobe University at its Wodonga campus at which I joined the Department of Environmental Management and Ecology and delivered courses in chemistry and environmental science. Whilst this deployment did not offer opportunities or facilities to allow research in thermochemistry, I was still able to publish in this field mainly in the form of invited reviews of thermal analysis and calorimetric techniques and their current applications (**P 160, 164, 168, 170, 173 – 7, 180 – 83, 185, 194 – 97**).

In 2001, I was appointed Visiting Professor to the Centre for Thermal Studies (CTS), Department of Chemistry at the University of Huddersfield, UK. Peter Laye was also a Visiting Professor in this Centre and the Director was Ted Charsley. These appointments facilitated a professional reunion of our time together in the mid - 1960's as postgraduate students in the Chemistry Department at Battersea College of Technology. The CTS was well established and recognised as a centre of excellence in the thermal sciences, both in the UK and overseas and was well-equipped with a wide range of state-of-the-art thermal analysis equipment and thermal activity monitors (TAM). In addition to undertaking academic research, the centre attracted a range of externally funded contracts. I was invited to spend limited periods at the centre in 2001, 2002 and 2005 and to be involved with thermal analysis projects in progress. The CTS team over this period included Ted Charsley (Head/Director), Gareth Parkes, Jim Rooney, Sarah Goodall and Hayley Markham along with Peter Laye (Visiting Professor). In 2001, I gave a keynote lecture on some pioneering (historical) applications of classical calorimetry in the CTS (**P 164**) which effectively summarised my career achievements in thermochemistry to date. During the first two visits to the CTS I gained experience with an emerging technique 'Sample Controlled Thermogravimetric Analysis' (SCTA) and applying this to study the combustion characteristics of a range of coals (**P 168**). The 2005 visit involved a DSC study of the equilibrium temperatures and enthalpies of solid-solid transitions of rubidium nitrate (**P 180, 185**).

Throughout my career, I have been a member of the International Confederation for Thermal Analysis and Calorimetry (ICTAC) and I have attended and presented at several ICTAC Congresses: 1974, ICTA 4, Budapest: 1980, ICTA 6, Bayreuth: 1985, ICTA 8, Bratislava (**P 72**): 1988, ICTA 9, Jerusalem: 1992, ICTAC 10, Hatfield. I am a continuing member of the ICTAC Awards Committee and the ICTAC Education Committee. In 1991, I produced and edited 'For Better Thermal Analysis and Calorimetry (III)' (**P 130**) which was widely regarded as an indispensable handbook for those professionals practicing thermal analysis and calorimetry. In 2012 and in conjunction with Professor Ranjit K. Verma, Chair of the ICTAC Education Committee, I published definitive papers on education in thermal analysis and calorimetry (**P 195 – 7**). These were presented in abstract form at an 'Education in Thermal Analysis and Calorimetry' workshop as part of ICTAC 15, Osaka in 2013. In 2002, I attended the European Society for Thermal Analysis and Calorimetry (ESTAC) conference in Barcelona at which (**P 168**) was presented as a poster. I have attended several Indian Thermal Analysis Society (ITAS) conferences: 1987 ITAS 6, New Delhi (**P 89**): 1989, ITAS 7, Srinagar (**P 101**): 1991, ITAS 8, Bhubaneswar (**P 124, 129**): 1993, ITAS 9, Goa (**P 150**): 2006, ITAS 15, Jaipur (**P 180**). I have also attended a few US 'CALCON' conferences: Midland, 1968, Bartlesville, 1988 and Honolulu, 2003. In 1963, I attended my first international conference (along with Ron Schulz and Geoff Walter) an IUPAC Chemical Thermodynamics conference in Exeter, UK and in 1979, I was invited by the conference chair, Professor C. N. R. Rao to give a plenary review lecture entitled 'Thermal Analysis applications in solid-state chemistry' at the Winter School in Solid-State Chemistry, Indian Institute of Chemistry, Bangalore. In 1982, I was invited to the 'Parton Memorial Chemical Thermodynamics conference' in Auckland in honour of Professor Parton, a leading thermodynamics researcher in the Department of Chemistry at the University of Otago in Dunedin in the latter half of the 20th century.

In conclusion and in my attempts to make contributions to the advancement of thermochemistry over more than half a century, I have been exceedingly fortunate to have had outstanding mentors, dedicated collaborators and, most importantly, diligent and enthusiastic postgraduate students. Without their commitment, passion and dedication to research, my thermochemistry tree would not have developed and been sustained. My successes are simply a reflection of their inspiration, enthusiasm and professionalism.

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- 201** Challenges for chemical education: Implementing the 'Chemistry for all' vision.
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- 202** Designing core concepts for the tertiary 'Basic Chemistry' course.
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- 204** La Trobe University Chemistry: The first fifty years.*
* In association with La Trobe 50th Anniversary celebrations.
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MY THERMOCHEMISTRY JOURNEY INSPIRED AND ENRICHED BY MY MENTORS, COLLABORATORS AND POSTGRADUATE STUDENTS



Battersea College of Technology, established in SW London in 1891 and became the foundation institution of the University of Surrey, established in Guildford in 1967.



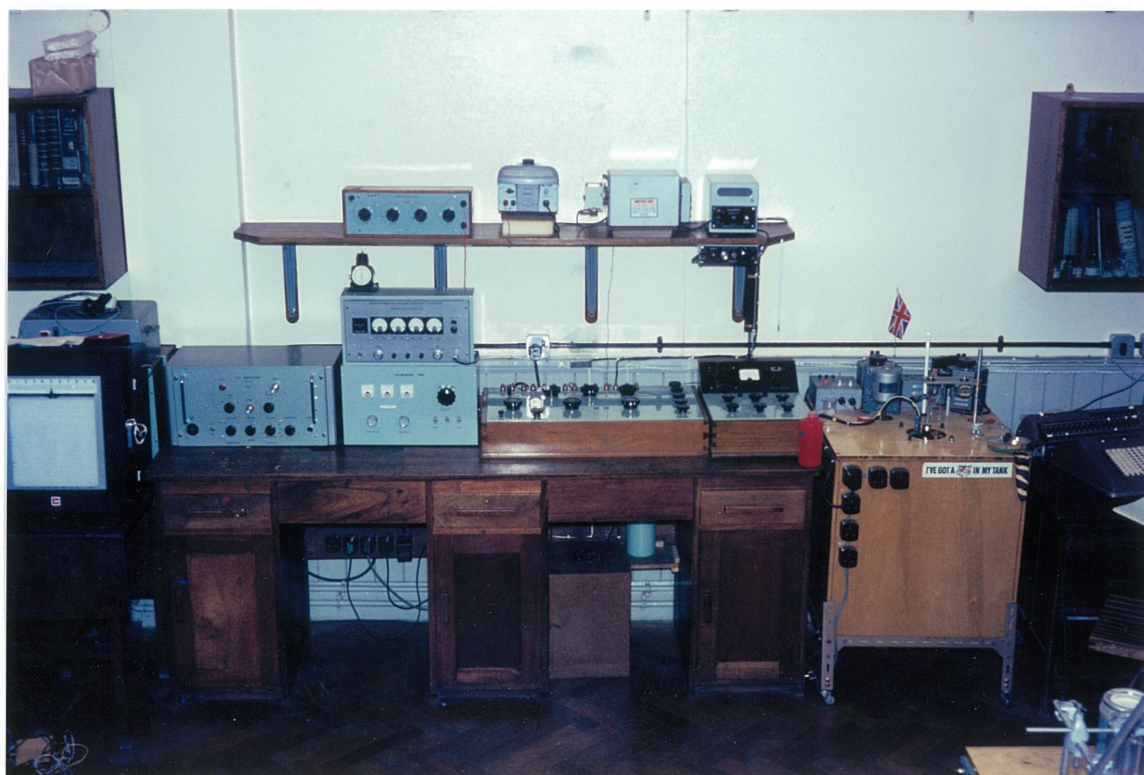
Greetings montage sent to Roger Irving for his 90th birthday by the 1960's BCT thermochemistry group.



BCT Chemistry class of 1962: Reunion, Iver, October, 2012. Back row, from left: Fred Parrett, Les Larkworthy, Joe Bullock, Joe Hegarty, Herb Askew, Malcolm Hogarth, Gordon Johnston, John Sherlock, Mike Abraham, Norman Boyland, Keith Goddard, Tony Butcher, Roger Mulberge. Front row, from left: Diane Askew, Margaret Cross (Waldron), Jean Larkworthy, Helen Hegarty, Ann Sherlock.



BCT Chemistry: Reunion, Sydney, January, 2013. From left, John Hill, Tony Butcher, Margaret Cross, and Keith Goddard



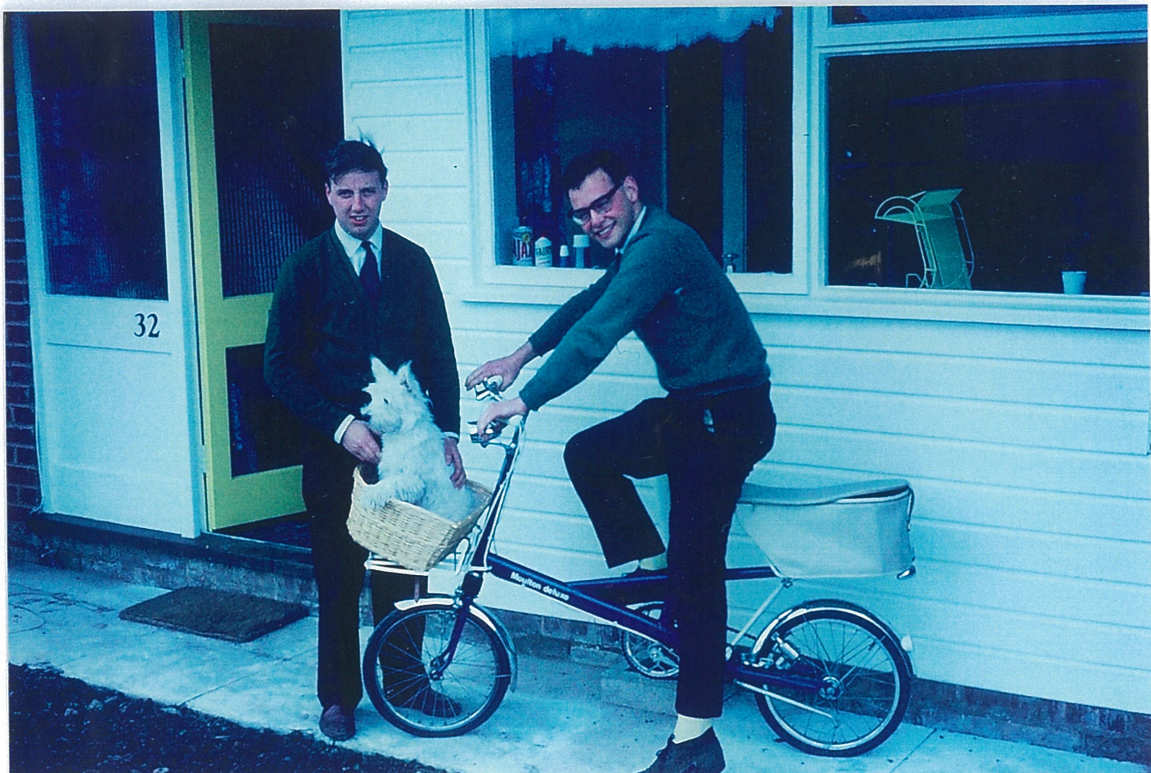
The Irving/Hill/Schulz solution calorimetric system (Mk 1) circa 1964.



The Irving/Hill/Schulz solution calorimetry system (Mk 2) circa 1967/8.



Geoff Walter and Ron Schulz at Exeter Cathedral 1965.



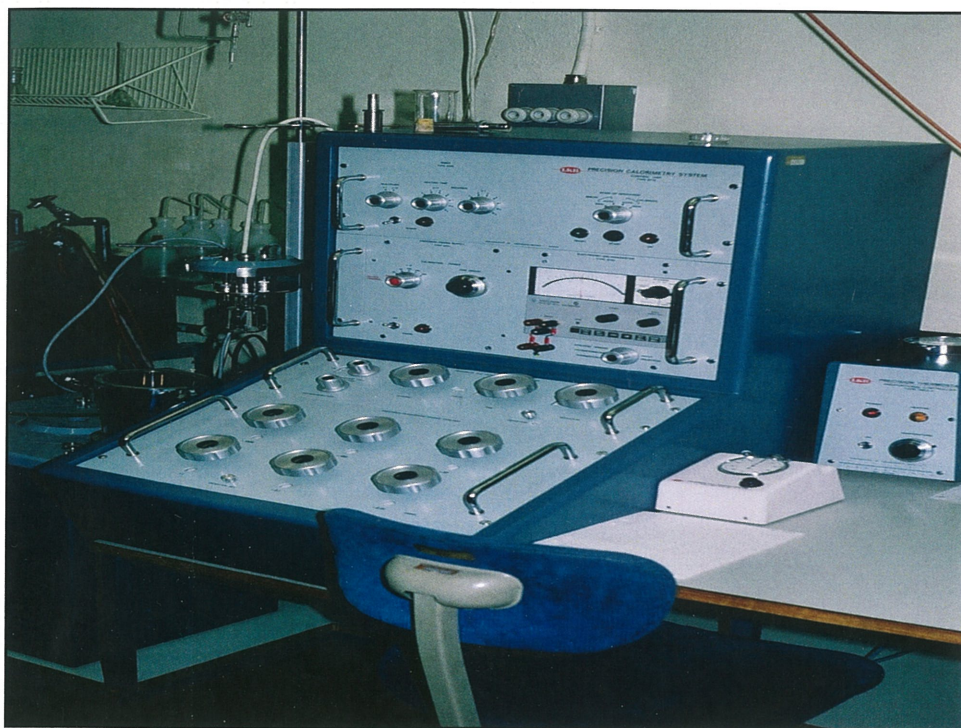
Hugh McKerrell and Geoff Walter: 1965.



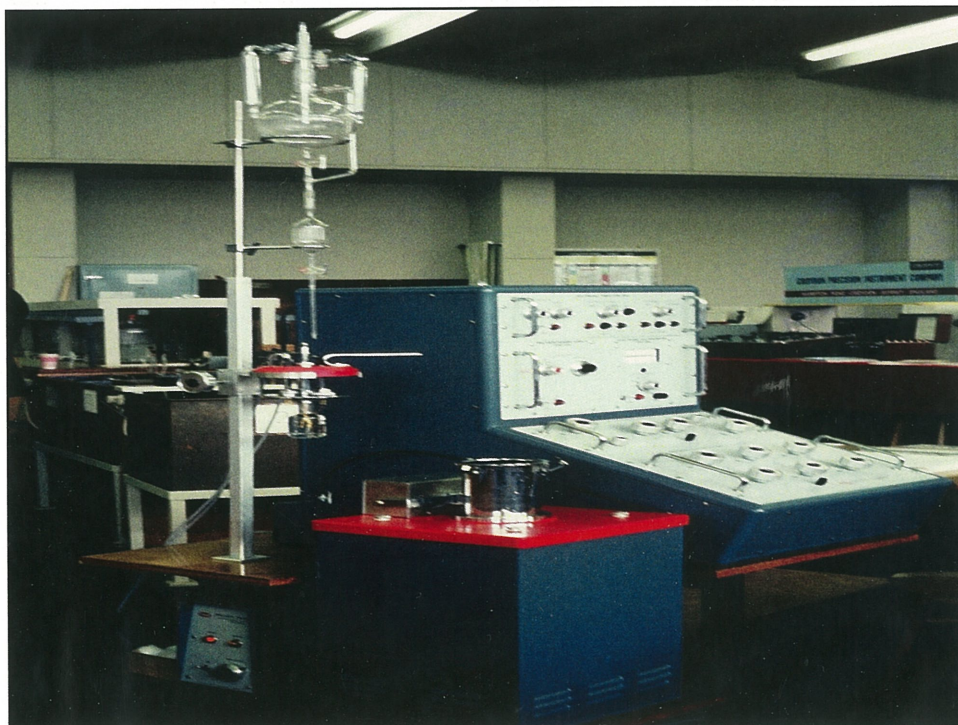
Jeff Walter and John Hill: 1965.



Ian Worsley at his home in Winnipeg: 2015.



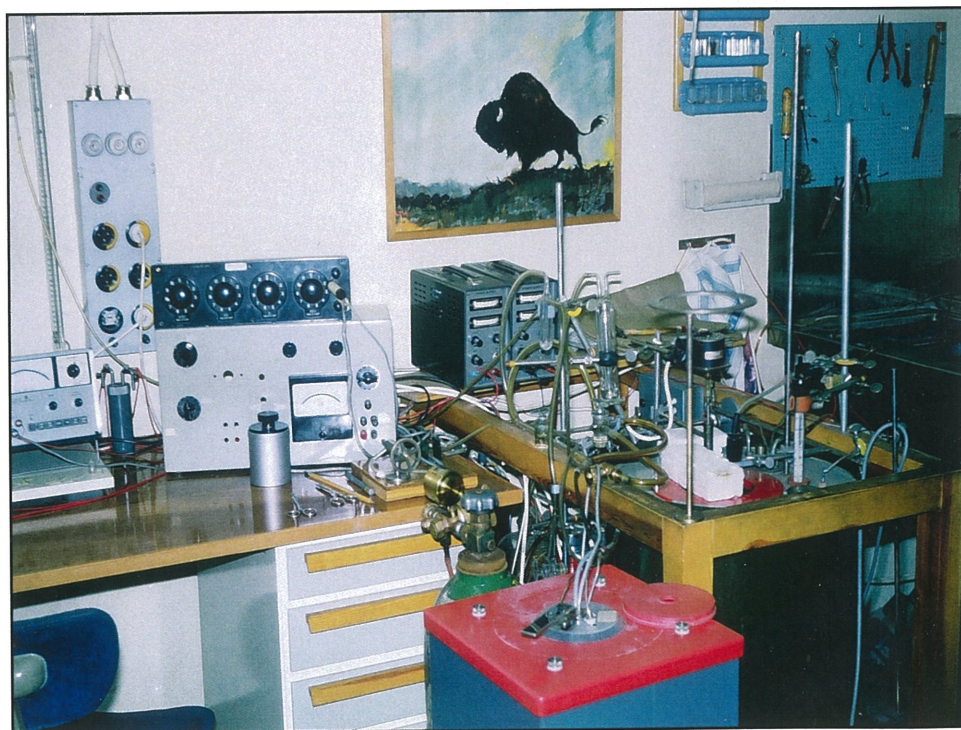
Prototype LKB solution calorimetric system, Lund Thermochemical Centre, 1967.



Prototype LKB titration calorimetric system, Lund Thermochemical Centre, 1967.



Prototype LKB vapourisation calorimeter, Lund Thermochemical Centre, 1967.

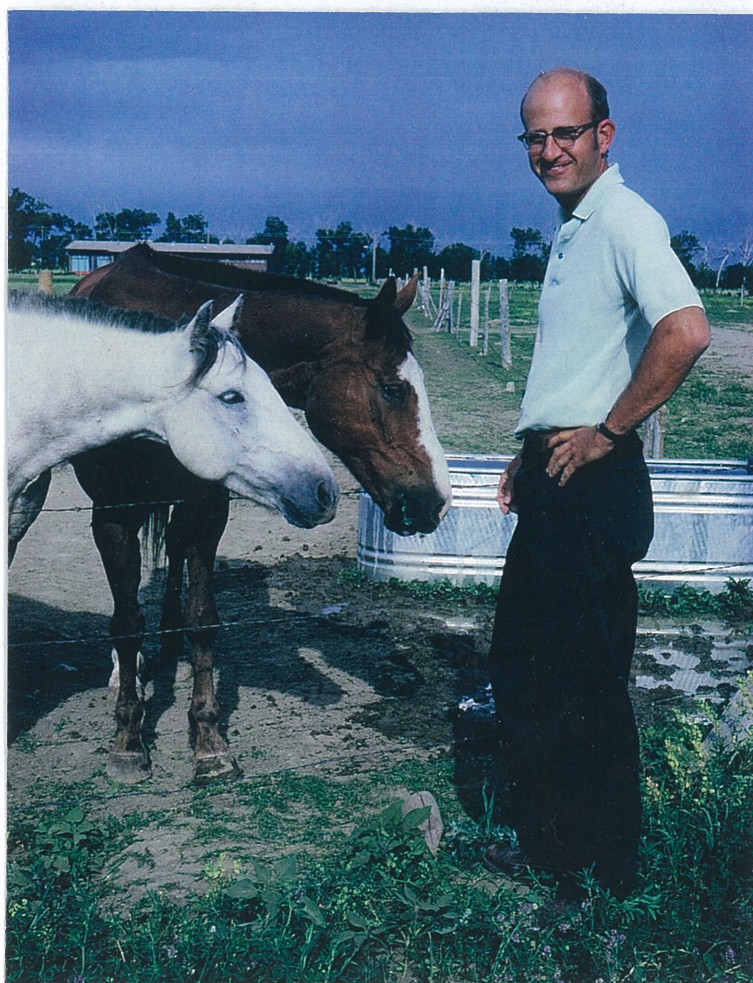


Prototype LKB vapourisation calorimetric system showing associated electronics.



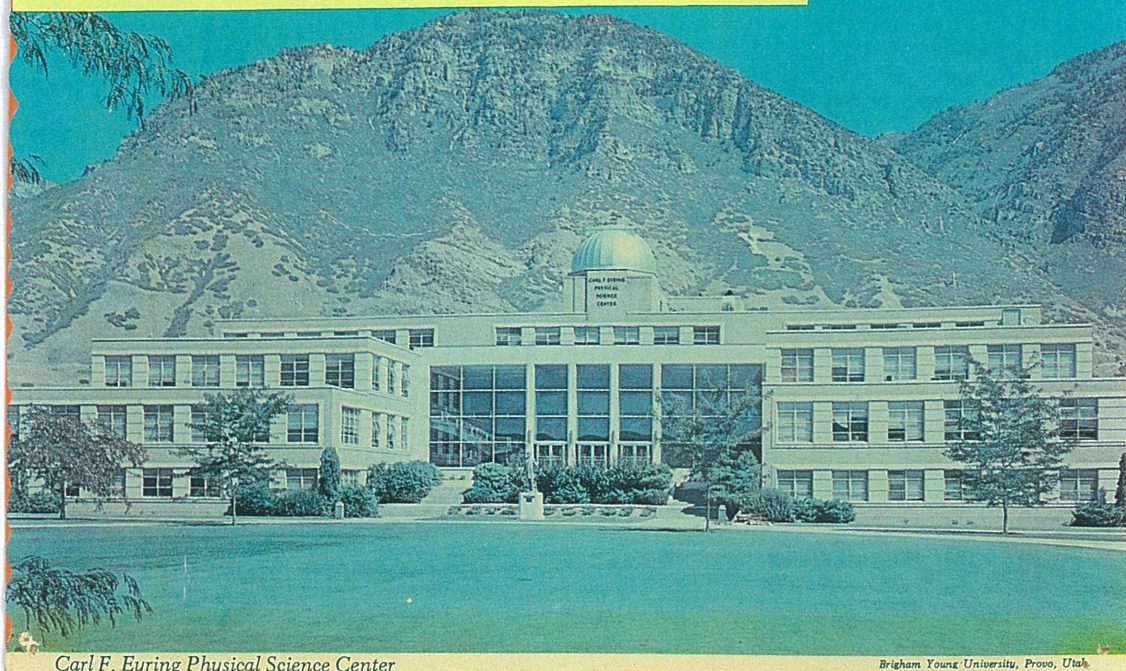
Lund Thermochemical Centre Post-Doctoral Fellows 1967:

L → R: Peter Howard, Paul Monk, Peter Sellers, Pepe Layenef, Haru Watanabe, John Hill.



Loren G. Hepler: 1978

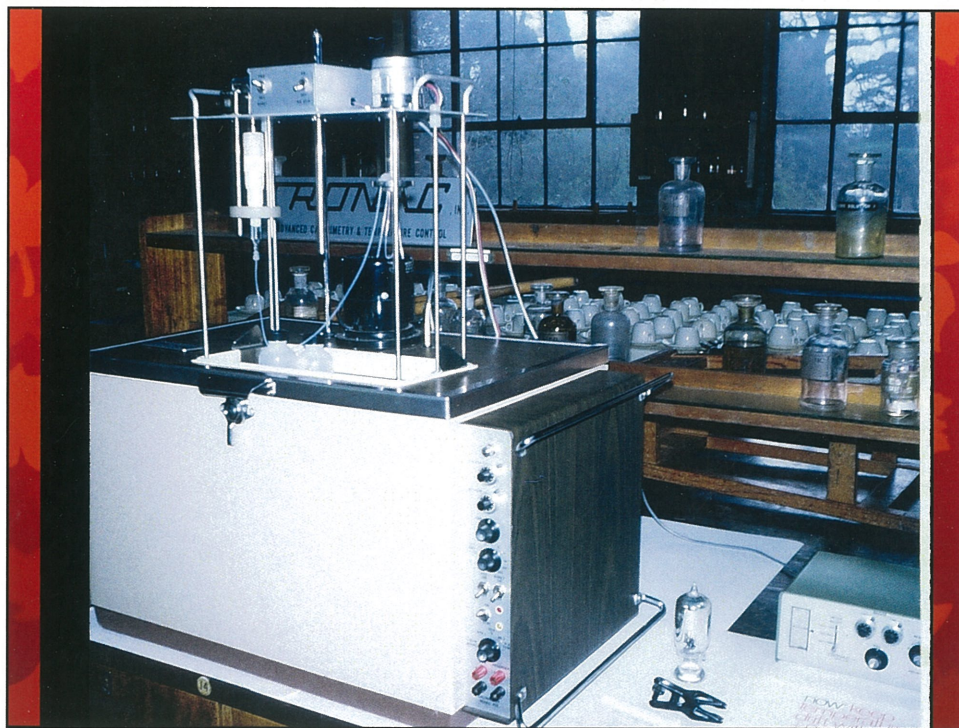
BRIGHAM YOUNG UNIVERSITY (Provo, Utah, USA)



Carl F. Eyring Physical Science Center

Brigham Young University, Provo, Utah

Eyring Science Centre (Chemistry Department), Brigham Young University, Provo, Utah: 1969.



Prototype Tronac titration calorimeter: Izatt/Christensen Thermochemical Centre, BYU: 1969.



Reed Izatt visiting our home in Pantton Hill in 1989:
R → L: Reed Izatt, Sue Hill, Anne Izatt, Emma Hill (Aged 8).



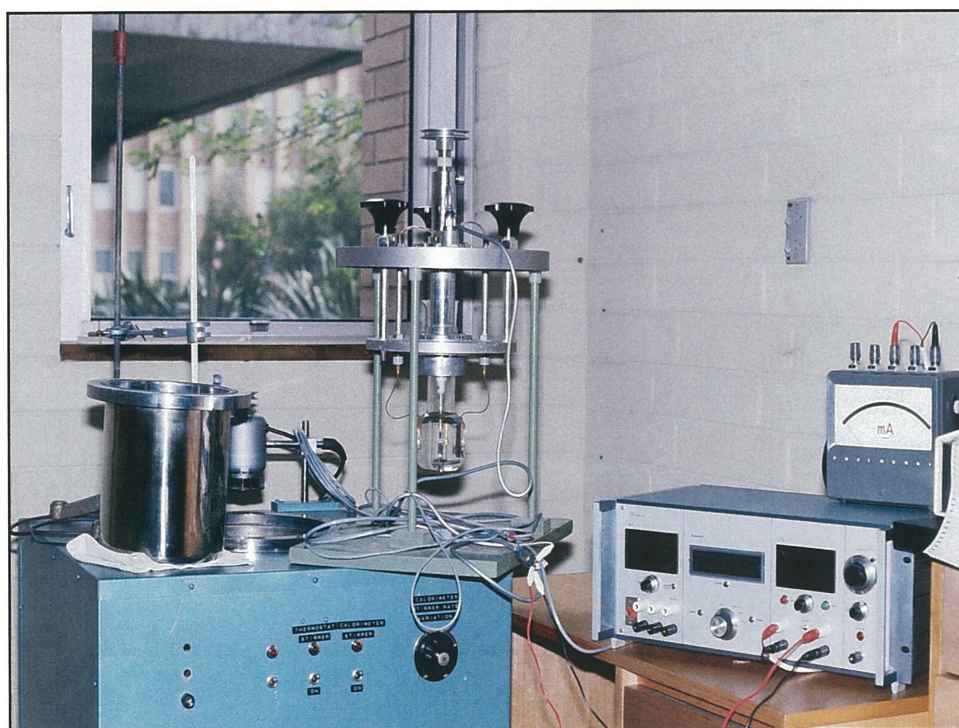
Izatt/Christensen Postdoctoral Fellows 'office', BYU: 1969.



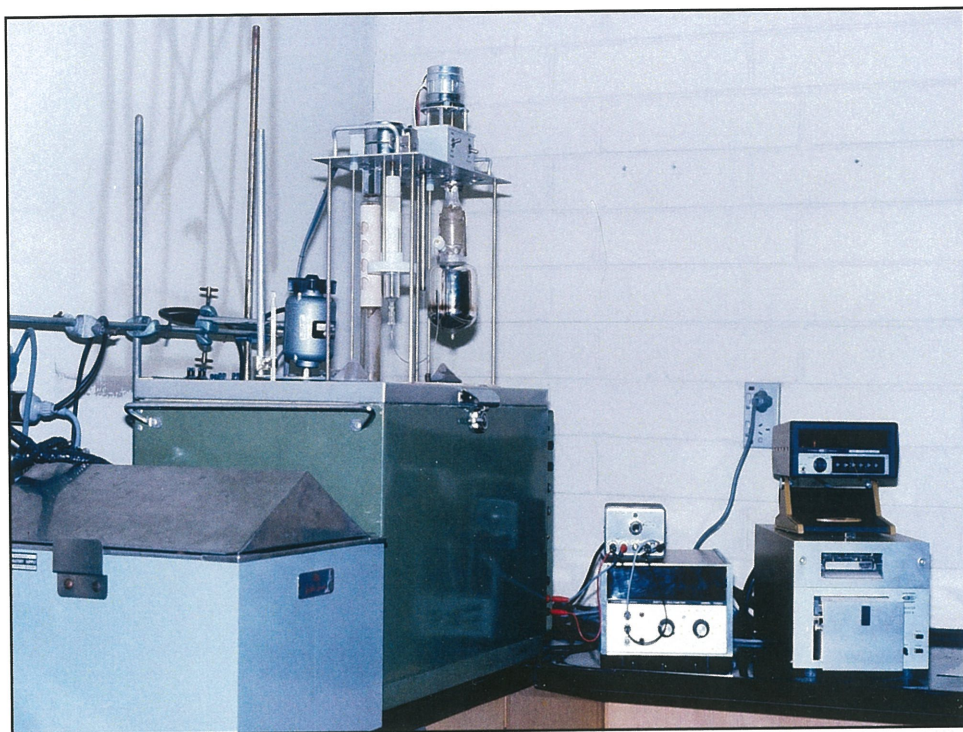
My BYU contemporaries: R → L: Austin Yingst, Ian Worsley, Sally Yingst.



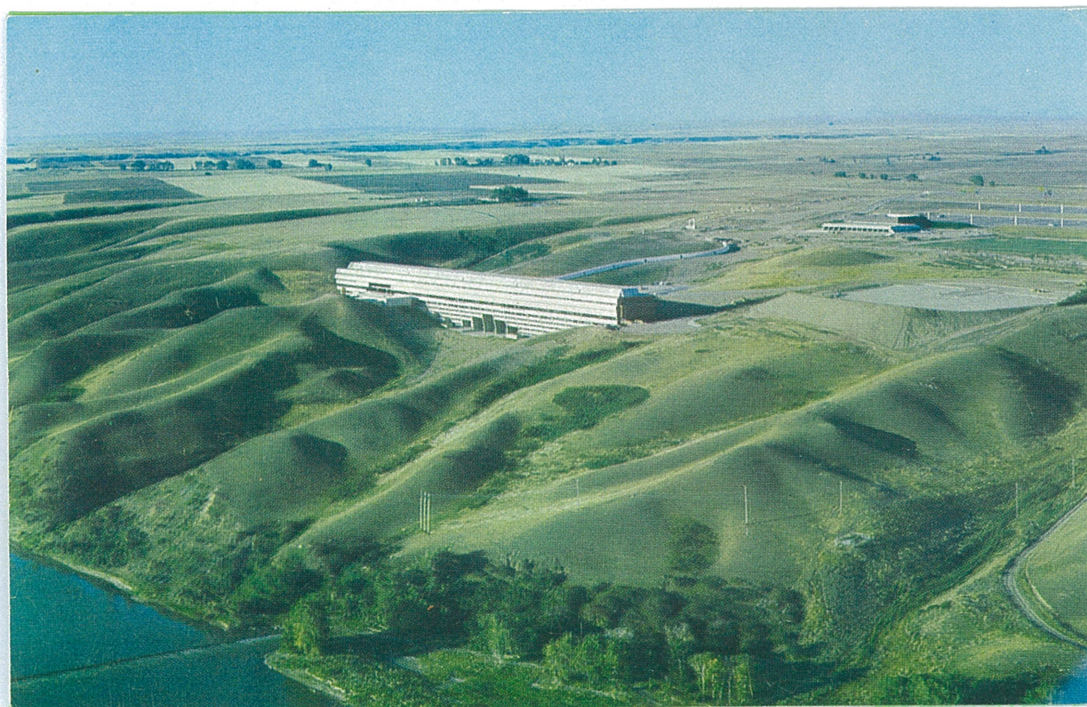
La Trobe University, Melbourne, Australia. I was appointed a Research Fellow
in the Department of Chemistry in 1970.



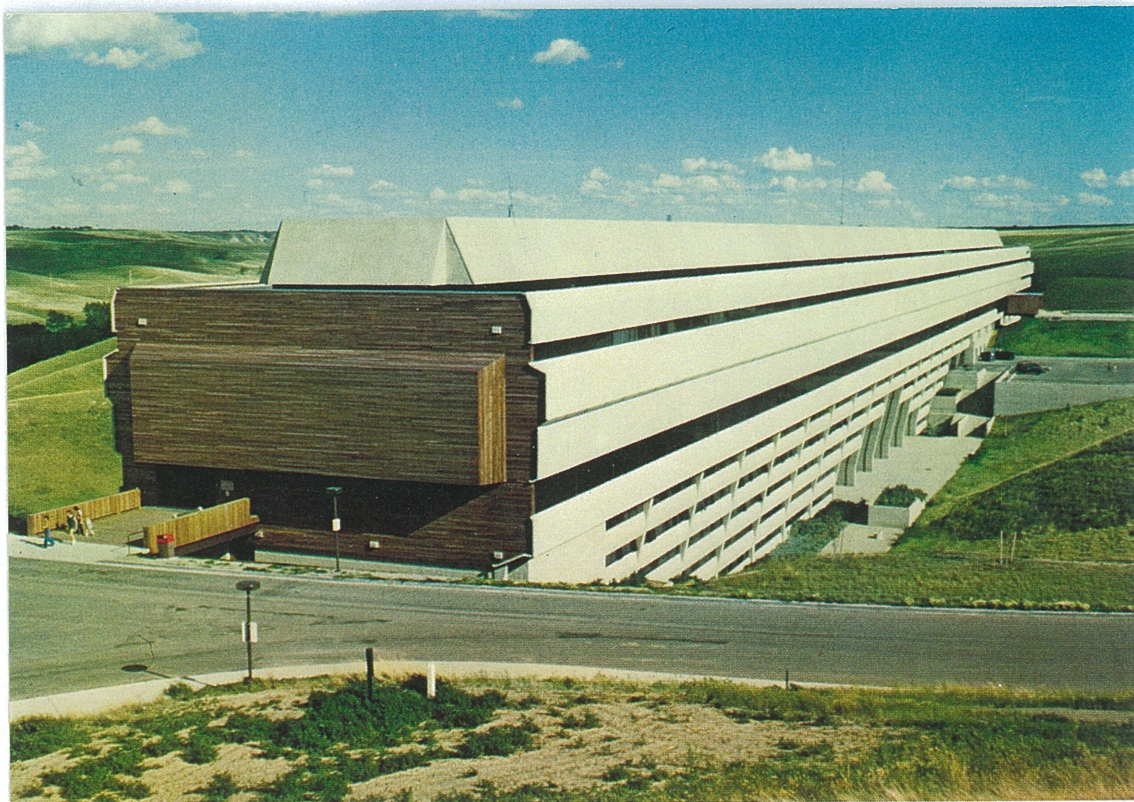
'In-house' constructed solution calorimeter, Hill Thermochemical Centre, LTU: 1970's.



Tronac solution/titration calorimetric system, Hill Thermochemical Centre, LTU: 1980's.



University of Lethbridge showing the sole building on campus in 1978.



University of Lethbridge: The only building on campus was 250m in length on 3 levels and accommodated the entire academic and administrative community.



University of Lethbridge: The Hepler thermochemistry group: 1978.



Founders Building, Royal Holloway College (University of London), Egham, UK.



Royal Holloway College Chemistry Department building: 1986.



Arthur Finch (2nd from Left/Front Row) thermochemistry group: 1986.



DSc (University of London): Conferment Ceremony, Barbican Centre, London: February, 1994.



Ted Charsley, Director, Centre for Thermochemical Studies, University of Huddersfield, 2005.



Huddersfield CTS group: L → R: Ted Charsley, Peter Laye, Jim Rooney: 2005.



Huddersfield CTS group: L → R: Hayley Markham, Peter Laye, Jim Rooney: 2005.



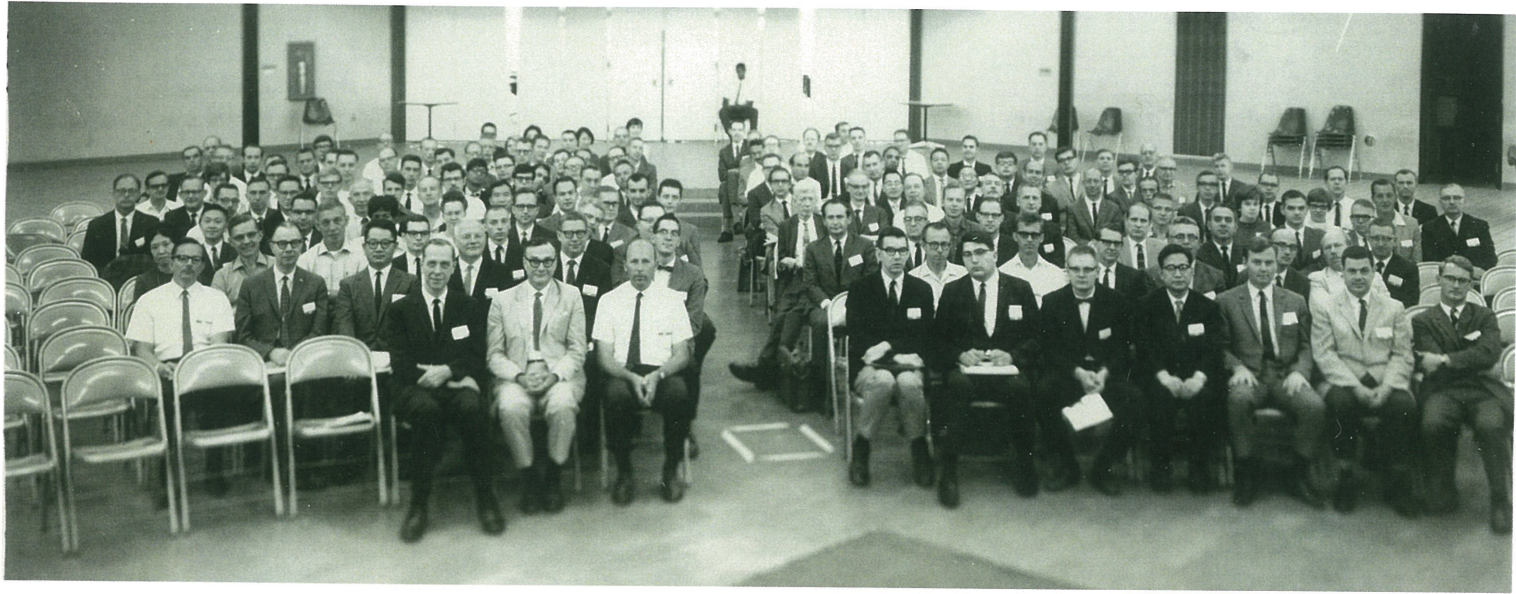
Huddersfield CTS group: L → R: Sarah Goodhall, Jim Rooney, Hayley Markham (2005).



IUPAC Chemical Thermodynamics Conference, Exeter: 1965.
 Notable attendees included Max McGlashan, Hank Skinner, Roger Irving.



Parton Chemical Thermodynamics Conference, Auckland: 1982.
 Attendees included Jim Pearce and John Hill.



23rd CALCON Conference, Midland, Michigan: 1968.

Notable attendees included: Hank Skinner, Max McGlashan, Ingemar Wadsö,
Cecil Vanderzee, Reed Izatt, Jim Christensen and Delbert Eatough.





'6th ITAS', New Delhi, 1987.





'9th ICTAC', Jerusalem, 1988.

L → R: Jeff Dunn, Slade Warne, John Hill, Unknown, Mike Brown, David Morgan.



'9th ICTAC', Jerusalem, 1988. L → R: John Hill, Edith Turi.



'9th ICTAC', Jerusalem, 1988. L → R: Mike Brown, Unknown, Shmuel Yariv, Jeff Dunn, John Hill, Unknown.



'9th ICTAC', Jerusalem, 1988. L → R: Ted Charsley, John Hill, David Morgan.



'9th ICTAC', Jerusalem, 1988. Sightseeing in the 'Old City' (Tour guide at extreme right).



'9th ICTAC', Jerusalem, 1988. L → R: David Morgan, Ted Charsley, Jeff Dunn, John Hill.