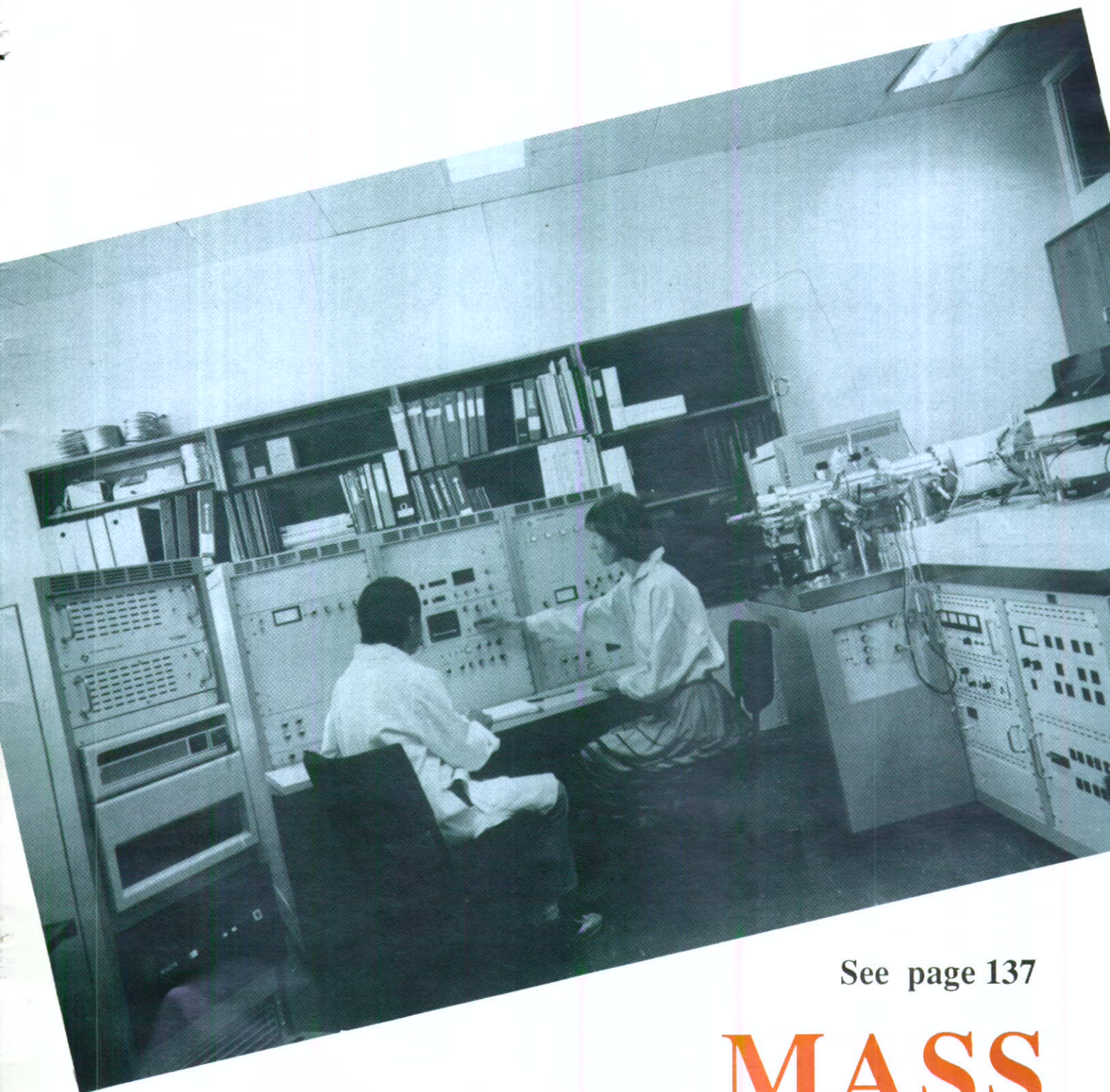




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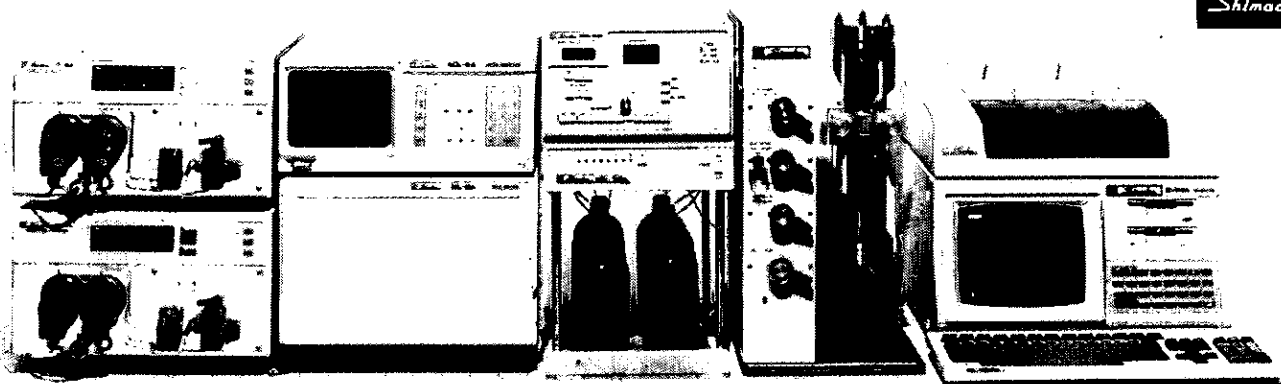
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See page 137

MASS SPECTROMETRY



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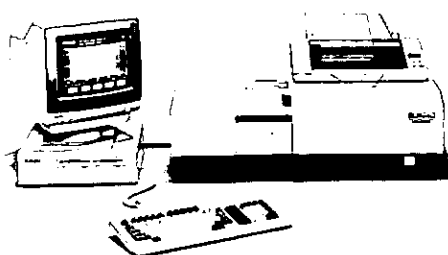
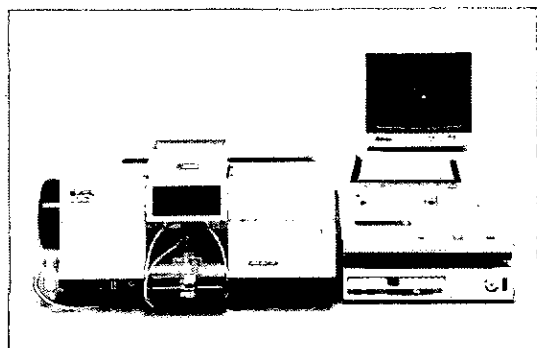
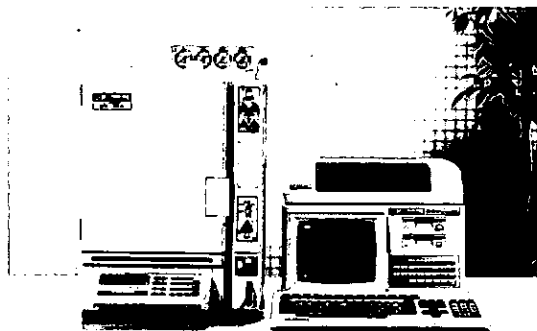
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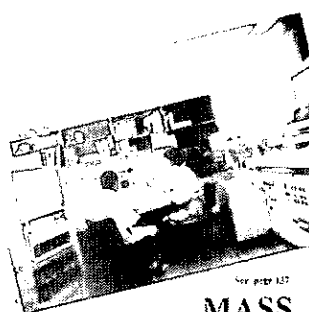




chemistry

in new zealand

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MASS SPECTROMETRY

FRONT COVER STORY

Mass Spectrometry Feature: A review from Harry Young and a directory of current instrumentation in New Zealand. See Page 139.

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Editor: Bruce Graham, c/- Dept of Health, 2 Edenvale Rd, Mt Eden, Auckland.

Branch Editors:

Auckland: Dr Roger Whiting, Auckland Technical Institute, Private Bag, Auckland.

Waikato: Nick Robinson, c/- Chemistry Dept, University of Waikato, Private Bag Hamilton.

Manawatu: Dr Cecil Johnson, Applied Biochemistry Division, Private Bag, Palmerston North.

Wellington: Dr Lawrence Porter, Chemistry Division, DSIR, Private Bag, Petone.

Canterbury: Dr Selwyn Maister, Christchurch Polytechnic, P.O.Box 22-095, Christchurch.

Otago: Dr Jim McQuillan, Chemistry Department, University of Otago, P.O. Box 56 Dunedin.

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Advertising Features

December:

The featured product is Mass Spectrometry, with an extensive "state of the art" review on page 139, by Harry Young, followed by a directory of current MS facilities in New Zealand and their areas of application. Some areas of applied mass spectrometry are included in our Product News.

February:

A major part of the magazine will be dedicated to biochemistry, with papers from the "Batt Symposium" held during the 1988 Annual Conference. The product feature will cover a range of equipment relevant to this subject area.

PEOPLE

1988 Shell Prize For Industrial And Applied Chemistry

Dr Ian Miller was employed by Chemistry Division, DSIR, from 1971 to 1986. In 1975, Dr Miller was made responsible within Chemistry Division for work related to the generation of energy from, and the recycling of, organic wastes. One project arising from this was the high pressure liquefaction of biomass.



Although from the scientific point of view this project was successful in that a route to synthetic fuel feedstock was identified (and potentially one of the cheapest of the synthetic fuels, and one, also, which does not contribute to the greenhouse effect), considerable development work remained, and the falling oil prices made the project unjustifiable.

A further project involved the recycling of plastics. A process was developed to the pilot stage by which mixed coloured plastics could be converted to pure white material similar to virgin material. Work was also carried out on designing a more simple wash system for recycling clear plastics, and material supply considerations led to the second option being followed by Plastic Granulators Ltd, and a plant was opened in Otaki in the early 1980s.

When the decision to proceed with the Motunui synthetic fuels plant was made, Dr Miller recognised that a byproduct of the process, durene, would be present in volumes not previously available. He identified durene as a feedstock for pyromellitic dianhydride (PMDA), which in turn is a raw material for a number of useful materials, including the polyimides, which are very high performance plastics at elevated temperatures. Following a significant publicity campaign by Dr Miller, the Government announced that it would accept tenders for the development of this resource. Dr Miller became the sole technical consultant for

a small entrepreneurial company Applied Chemistry Ltd, which made an initial submission to the Crown in April 1984. While none of the initial submissions were acted on, in the meantime, Applied Chemistry Ltd formed a Joint Venture with ICI NZ Ltd, and eventually this Joint Venture won the rights to develop the durene. A company, ICI Synchem Ltd, was formed (70% ICI NZ, 30% ACL), and this company is currently engaged in commercialising this opportunity.

In 1978, Dr Miller also published a paper demonstrating the basic conditions for improving the yield of agar from the New Zealand seaweed *Pterocladia*, which was the basis of an industry since 1944. Following this, he developed a new process for making high quality agar from *Pterocladia* mixed with extraneous seaweed, which had previously not been possible. These processes were offered to a Joint Venture between Excalibur Colloids Ltd and ICI NZ Ltd, and the latter process was the basis of a Joint Venture in the Wairarapa, under a company ICI Biocol Ltd. In the event, ICI NZ Ltd has recently withdrawn from this venture, but Excalibur Colloids Ltd intends to proceed with the venture, although performance the breakup of the joint venture will delay proceedings.

In 1986, Dr Miller left DSIR to form Carina Chemical Laboratories Ltd, a small private R&D company which operates both as a consulting laboratory and as a developer of ventures. In this time, Dr Miller has investigated a number of new technologies, with applicability in forestry, agricultural chemicals, hazardous chemical destruction (PCBs) new polymers and polymer raw materials, fine chemical production, and a number of other possible ventures. At present, financial considerations and the current economic climate have impeded these developments, but a number of patents are potentially available. These ventures are available for development should an appropriate Joint Venture partner emerge.

Dr Miller is currently Managing Director of Carina Chemical Laboratories, Technical Director of Applied Chemistry Ltd, Sunrise Chemical Industries Ltd, and Excalibur Colloids Ltd, is a Director of ICI Synchem Ltd, and was a Director of ICI Biocol Ltd.

New Registrar - Dennis Karl

After primary school at Paterangi and secondary education at Te Awamutu College Dennis commenced his working career at Ruakura Agricultural Research Centre (after a brief sojourn at Auckland University) in the Nutrition Section laboratories. This was followed by a period at the Meat Industry Research Institute of N.Z. Inc. During this period he gained his New Zealand Certificate in Science in Chemistry from Waikato Technical Institute.

After completion of NZCS he travelled with his wife to Britain for three years where he held positions at the City University, London and in the Beecham Products Research Laboratories, London. Upon returning from this trip he rejoined the Nutrition Laboratories at Ruakura for five years before moving to Auckland to take up a position in the General Analytical Services labora-

tories of T J Sprott and Assoc. When this practice was sold he left to set up his own consultancy and analytical practice, Scientific and General Consultants Ltd.

Dennis has been a member of the Auckland Branch Committee for a number of years, was a member of the Auckland conference committee and is currently on the committee of the Oils and Fats Group assisting with the organisation of the Fats For The Future II international conference on fats, oils and waxes to be held in Auckland in Feb. 1989.

In accepting the Registrar's position Dennis undertakes to try and assist as much as practical to speed the formation of the proposed permanent secretariat, which he sees as a big step in the direction of freeing members to concentrate on the intellectual side of the Institute rather than on the organisational nitty gritty.

EDITORIAL

Two months ago we had started on the draft of a 'farewell' editorial to go in this issue, on the assumption that a successor had been found to take over the reins in 1989. Alas, the only likely candidate changed her mind and the contract was not signed. It looks like all those other things we were planning to do in our spare time will just have to wait a bit longer.

(There is a theory that says lawns left unmowed will eventu-

ally cease to grow. This has not yet been demonstrated, however, and we may shortly have to consider taking sheep in lieu of honoraria.

Readers may not be surprised to learn that our sense of disappointment has caused a momentary block in the inspirational flow. We will therefore 'cheat' on the 500 word quota by using the remaining space for the message below. Help?

Bruce Graham

SITUATION VACANT - EDITOR

The current editor of Chemistry in New Zealand wishes to retire from the position at the end of the year and a replacement is therefore sought.

The position involves planning the content of each issue, soliciting of articles, liaison with Branch Correspondents and other regular contributors, selection, editing, and other preparation of material for publication, checking of galleys and page proofs, and so on. The editor acts as liaison between the publisher and the NZIC, and attends meetings of Council as appropriate. The editor is also convenor of the Editorial Committee, who are available to give advice and other assistance as required.

The work involved is a min-

imum of 20-30 hours per issue, with 6 issues per year. The position carries a modest honorarium.

It is preferable that the editor be a member of the NZIC and have a strong interest in chemistry generally, and in Institute affairs. As the magazine is currently published in Auckland an Auckland-based editor would be preferred, although with the ready availability of facsimile facilities operation from another part of the country should also be feasible.

Anyone interested in finding out more about the position should contact the present editor, Dr Bruce Graham, Dept of Health, 2 Edenvale Road, Auckland, ph 601-747 (bus) or 836 9472 (home).

1989/90 Nyholm Lectureship And Medal (RSC)

Robin J.H. Clark, who is professor of chemistry and dean of science at University College London, has been awarded the 1989/90 Nyholm Lectureship and Medal of the Royal Society of Chemistry (RSC). He is the first New Zealander to receive this award, which is made biennially for distinguished research in Inorganic Chemistry and is open to chemists of any nationality. The immediately previous recipient was Nobel-prize winner Roald Hoffmann. The lecture will be given at a specially convened conference in London as well as elsewhere.

Professor Clark is an old boy of Christ's College and the University of Canterbury (MSc first class honours 1958) and graduated PhD (1961), DSc (1969) and FRSC (1969) from University College London. He has lectured widely in Western and Eastern Europe, North America, Asia and Australasia and has acted as Visiting Professor at Columbia, Padua, Western Ontario, Bern, Fribourg, Texas A & M, Auckland, Odense, Sydney and Bordeaux. He was awarded the RSC's Tilden Lectureship and Medal for 1983/4. He is author of about 300 scientific papers and three books on inorganic chemistry/spectroscopy, editor of

seven monographs on inorganic chemistry, and co-editor of the widely praised review series "Advances in Spectroscopy", now in 16 volumes. Prof. Clark has also served on various UK Science and Engineering Research Council and RSC Committees, is currently a Vice-President of the RSC Dalton Council, and a member of the Senate of the University of London.



Prof. Clark's research interests embrace synthetic, structural and spectroscopic aspects of transition metal chemistry, most recently of mixed-valence, metal-metal bonded and one-dimensional materials. His seminal contributions to Raman and resonance Raman spectroscopy

1988 Easterfield Award

The winner of the 1988 Easterfield Award is Dr Gordon Rewcastle, a Senior Research Fellow in the Cancer Research Laboratory at the Auckland Medical School. Gordon is a graduate of the University of Auckland where he received his BSc, MSc (1st class honours) and PhD (1978) degrees. He was also the recipient of the 1978 L.H. Briggs Memorial Prize, which was awarded for his PhD research. After a period of post-doctoral research at Oregon State University he took up his present position with the Cancer Society in 1980. His work in the Cancer Research Laboratory has concentrated on developing general synthetic methods for the production of several classes of new antitumour drugs under study in the laboratory. More specifically, he has investigated unequivocal routes to the synthesis of individual isomers of polysubstituted tricyclic heterocycles, such as acridine, phenazine and xanthene. During the phenazine work he discovered a novel fluorine directing effect in

the reductive ring closure of 2-nitrodiphenylamines, and this proved to be of great synthetic utility. The tricyclic heterocycle theme was continued during a period of sabbatical leave at the University of Florida, where he investigated new methods for the synthesis of specifically substi-



tuted carbazole, phenothiazine and phenoxazine derivatives.

Gordon has been an active member of the New Zealand Institute of Chemistry, and was treasurer of the Auckland Branch for two years. He was elected to the Fellowship of the Institute this year.

Gordon will present details of his past and current research interests when he gives the Easterfield Address to the Hamilton conference of the Institute next August.

were recently recognized in his Chairmanship of the highly successful eleventh International Conference on Raman Spectroscopy (ICORS XI), held in London in September 1988.

1989 Branch Chairs (Part I)

Auckland - A.C.Herd

Tony Herd was educated at Otago, completing a PhD under the direction of Dr Chris Pope in 1970. For 18 months he held a post doctoral fellowship at the University of Tokyo, studying catalytic reactions via the infrared spectra of adsorbed species. Returning to New Zealand he worked for four years as a physical chemist at FMRA in Otara before joining the Auckland Technical Institute in 1976. He has been a senior lecturer at ATI since 1977 and is currently involved in several analytical chemistry courses including ACOL analytical chemistry workshops and a course leading to a Royal Society of Chemistry Certificate.

Tony was on the Auckland Branch Committee in 1972 and branch secretary in 1973. He edited Chemistry in New Zealand for two very long years in 1982 and 83 and has since

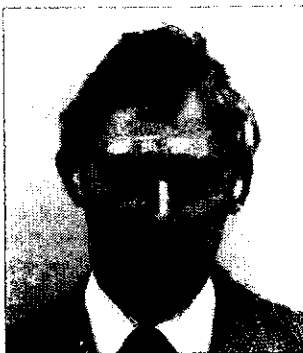
managed to fill the gap in his life with less painful pursuits like marathon running and worrying about rust in the mark 2 Jag.

Wellington - R Ledger

Rob Ledger is a Yorkshireman by birth who arrived in New Zealand ten years ago. Since he has now become a New Zealand citizen he felt it his duty to accept nomination as this year's Wellington Branch Chairman. He has been a tutor in the School of Pharmacy at the Central Institute of Technology since he came to New Zealand and is currently the Chairman of the Pharmaceutical Chemistry Group.

Dr Ledger was educated at the University of Sheffield where he obtained his BSc in Chemistry and Biochemistry in 1960 and PhD in steroid chemistry in 1963. On his first visit to the southern hemisphere he spent three years as a research scientist with CSIRO Division of Protein Chemistry where he discovered his

interest in protein structure. After a brief return to England as a research assistant at the University of Liverpool he accepted the



daunting task of setting up a new department in the Hacettepe University in Turkey. He spent an initially frustrating but ultimately rewarding three years in this position and is still in regular correspondence with his then graduate students from these years with whom he worked on the active site of serine pro-

teinases.

Between 1971 and 1978 Dr Ledger spent six years as a lecturer in medical biochemistry at the University of Cape Town Medical School where he worked on synergism in drug metabolism. During his career he has spent three years intermittently in positions at the College of Medicine of the University of Iowa working on aspects of protein folding. The hydrodynamics of protein unfolding and refolding continues to be his major research interest, though he has interests in affinity chromatography and in the biochemical interactions of food dyes. In his present position research interests are forced to take a back seat to teaching. In his teaching he has used structure activity relationships to bridge the various aspects of kinetics in the disciplines of biochemistry, physiology, pharmacology and pharmaceuticals.

OBITUARY

Joan McKenzie Parnell

Joan McKenzie Parnell became a member of the New Zealand Institute of Chemistry in 1976 when she returned from postdoctoral studies to take up a Lectureship in the Department of Nutrition at the University of Otago. She had been working at the United States Department of Agriculture's Nutrition Laboratory at Grand Forks in North Dakota during the tenure of a Postdoctoral Fellowship of the United States Department of Public Health, continuing her early research on zinc with special reference to its importance for the development of the brain.

She was an active member of the Otago-Southland branch of the NZIC, and contributed much to local activities. She served on the Committee from 1978 - 80, and 1982 - 85, becoming Secretary 1978 - 80 and Chairperson in 1984. This coincided with her term on the Council of the Institute (1982 - 84); and in 1986 she was elected to the Fellowship.

She was outstanding in her research, a meticulous and hard-working investigator, and a sympathetic, understanding, friendly and imaginative supervisor of projects. Her research was all essentially on trace elements, and she handled their many problems with initiative and infinite resource, as exemplified by

her success in persuading Bluff oystermen to collect metabolic samples for her. They were, of course prolific eaters of oysters and she needed to discover how



much cadmium they absorbed. Her use of herself as a subject for experiments of this kind led to some strange feats of eating - like 4 dozen oysters before breakfast - and to her having often to attend meetings elsewhere in New Zealand with the paraphernalia needed to collect her own metabolic samples. She came to be recognized internationally for her contributions to this field of nutrition. She had presented invited papers at conferences in Germany, Scotland and the USA, and was invited to give the Inaugural General Food Oration at

Deakin University in Australia.

In the University Joan was an outstanding teacher. The students admired her calmness and serenity, and found her available and approachable at all times as a wise counsellor in personal as well as academic problems. Indeed, she was so devoted to her graduate and honours students that the coincidence of timing sometimes faced her with a difficult decision - whether she should take part in their graduation or in the opening of the duck shooting season on her home territory at Seaward Downs. She was also outstanding in the quality of her relationships with her colleagues; she had their complete trust and for two years was their representative on the University's Senate. One junior lecturer said, "Joan was a model for me, in her academic achievements, in her integrity, in her relationships with people, and in her faith." She was promoted to an Associate Professorship in 1987.

Joan was indeed a quite remarkable person. She had entered the University in 1965 from Southland Technical College; and it was only after she had taken School Certificate - in Household Arts - that a perceptive Headmaster steered her towards chemistry and mathematics. Her first year at Univer-

sity was no mean challenge; but as with so many other goals she set herself, she made no fuss, but worked quietly and unswervingly towards the end in view. She often astonished everyone (possibly including herself) with what she accomplished in her quiet, determined way. In her humanity she made time for other activities too. For many years she led successfully, along with Colin Fitzpatrick, a tough youth group that no-one else dared to take on. She was an Elder, and Session Clerk at her local Church; and in her few spare moments she took to the hills to ski or tramp - and not only local hills - there was one eventful tramp in Nepal.

Throughout her life she was sustained by great faith and a tremendous sense of purpose. Even during the last few years of her life when she was fighting a tough fight against an unseen but remorseless enemy, she was a source of inspiration to us all, with her wonderfully positive attitude, her resilience, resourcefulness and determination to cope, with all the strength she had in her.

Dr Parnell is survived by her husband, and her parents, Mr and Mrs R D McKenzie of Seaward Downs near Invercargill.

Marion F Robinson

NOTICES

Chemical Education Trust

The Trustees of the NZIC Chemical Education Trust Fund record their grateful thanks for financial contributions during the period 1 May 1987 to 30 August 1988 from the following companies:

Abels Ltd, ANZ Banking Group (NZ) Ltd, Bayer NZ Ltd, Cable Price Downer Ltd, Caltex Oil (NZ) Ltd, Catoleum New Zealand Ltd, Chemby Industries Ltd, Chemcorp Holdings Ltd, Ciba Geigy New Zealand Ltd, Dominion Breweries Ltd, Hoechst New Zealand Ltd, Ivon Watkins-Dow Ltd, Mair Astley Ltd, NZI Corporation Ltd, NZ Pharmaceuticals Ltd, Petralgas Chemicals NZ Ltd, Rohm & Haas NZ Ltd, Westpac Banking Corporation Ltd, Wattie Industries Ltd, Wm. Scollay & Co. Ltd.

It is particularly pleasing to note that in addition to the above support, a considerable number of members of the Institute have made individual contributions.

The capital of the Trust now stands at approximately \$48,000.

A.W. Mackney
Chairman Of Trustees
National Chemistry Day, 30th June, 1989

A National Chemistry Day is planned for Friday, 30th June, 1989. A series of National competitions is being organised, but in addition, Branches are being asked to organise their own local activities. The theme chosen for the National competitions is "Chemistry, Working for the Modern World". Prizes will be offered for the best entries in the various competitions. It is hoped that schools will encourage either individual students or groups of students to attempt projects on New Zealand's chemical industries. The NZIC publication "Chemical Processes in NZ" vol. II edited by J.E. Packer and R. Whiting should be a useful starting point for these activities.

Dr Joyce Waters
1st Vice-President

University News

Waikato

Two topical speakers visited the University recently. **Dr Bob Mann**, Senior Lecturer in Environmental Studies at Auckland University, traced the New Zealand history of 2,4,5-T, and recommended a shift in emphasis in the way we manage our ecosystems. **Dr Mann** suggested we should move towards more traditional biological methods of eco-management. **Dr Doug Edmeades**, soil scientist of Ruakura, gave his account of the longest litigation court case (137 sitting days) in the history of the Commonwealth, that of *Maxicrop*, which eventually vindicated his claim that "Maxicrop does not work". Both talks were well attended and gave rise to lively discussion.

Victoria

Stuart Smedley has returned from a brief trip to Chicago where he attended and presented a paper at the Electrochemical

Society Meeting in that city. He also spent a couple of days at the Stanford Research Institute on the way home. Two visitors to the Department have arrived. **Professor C.K. Lee** from the University of Singapore has arrived to work over the next few months with **Professor Ferrier**. **Dr Franz Preilmeir** has come from the Institute fur Biophysik, Universität Regensburg to take up a postdoctoral position working with **Dr Speedy**. **Dr. Preilmeir** and **Dr. Peter Osvath** (from ANU) have recently given seminars in the Department.

Canterbury

Professor Kyle Bayes, Professor of Chemistry and Departmental Chairman at UCLA, visited the department as an Erskine Fellow during July and August. During March and April next year **Dr Eldon Ferguson**

Continued next page

GOVERNMENT DEPTS AND RESEARCH INSTITUTES

Forest Research Institute, Rotorua

Dr Roger Rowell, of the United States Forest Products Laboratory at Madison, Wisconsin was a visiting scientist with the Product Development group at the Forest Research Institute in Rotorua from mid-October to late November this year. Dr Rowell is Team Leader, Property Enhancement of Wood Composites at Madison and is internationally known for his research on chemical modification of wood as a route to new improved wood products. The Product Development group at FRI, led by Dr David Plackett, is investigating ways of chemically modifying radiata pine fibre or particles for use in new composite wood products. The Forest Research Institute maintains links with other scientists working in the field of wood modification through an informal network established by Dr Rowell.

Dr Rowell was one of the guest speakers at a symposium on composite wood products, held at the Forest Research Institute on November 9 - 10. The symposium also included speakers from FRI and other organisations covering such topics as the latest developments in adhesive systems for board products, new board manufacturing processes, and the preservation and finishing of composite wood products.

Chemistry Divn, DSIR, Christchurch

Dr Rob Lake was recently appointed to the Food Section of Chemistry Division. He recently completed a Post Doctoral Fellowship at the University of Canterbury in the marine natural products group. Dr Hamish Williamson was recently appointed to the Forensic Biology section. He comes from recent completion of a PhD at the Christchurch Clinical School.

Wool Research Organisation of NZ

Dr Campbell Page is on a 2 - 3 month trip to Japan and USA as a technical advisor on aspects of carpet yarn production and conversion into carpet. He is also attending the Ciba-Geigy labs in Switzerland for a crash course in dyeing technology. Mr Rex Stewart is currently in Argentina with the UN Industrial Development Organization in a consulting capacity and giving instruction to the wool scouring industry.

NECAL, Dept of Health, Auckland

Norman Thom has retired as Director of NECAL, after a career with the Health Dept spanning nearly 40 years. Norm joined the Department in 1952 as a trainee Health Inspector. He took a career change in 1960 to become Technical Officer, working specifically for the Auckland Air Pollution Research Committee, and then progressed to Chemical Inspector, then regional Air Pollution Control Officer, and finally Director of the National Environmental Chemistry and Acoustics Laboratory. During this time he has seen the lab grow from the original one-man establishment operating out of two converted army huts, to the present group of nearly thirty staff with the equipment and expertise to tackle a wide range of environmental problems. Over the last few years Norm has also been the senior scientist in the Health Department and has thus found himself increasingly involved in matters at the Head Office level. He will be continuing to work under contract to the Department in this role for at least the first part of 1989.

Norm's successor at NECAL is Dr Merv Jones, a chemical engineer who has been with the group since 1972.

BRANCH NEWS



Dr Robert Franich presents the Chemical Education Award to Mrs May Croucher

BOP Sub-Branch

Professor David Doddrell, Queensland Medical Magnetic Resonance Centre, Brisbane, visited BOP sub-branch on September 12th, and gave a talk on Magnetic Resonance imaging and *in vivo* nmr spectroscopy. The complexities of *in vivo* nmr were highlighted, not only from the standpoint of the sample (e.g. largely water, with overwhelming water signal), but also from the instrumentation, creating high intensity magnetic field gradients. Prof Doddrell was highly informative and entertaining. Roger Meder (FRI nmr spectroscopist) benefited particularly from the visit, with many user hints on running aqueous samples, an area of increasing demand.

The NZIC Chemical Education Award was presented to Mrs May Croucher. May has taught at Rotorua Girls High School for fourteen years, and is co-author of some well-received texts (e.g. *Chemistry Guide Book*, with John Packer). Her contributions to teaching chemistry in New Zealand were rewarded with the Chemical Education Award, presented to her at FRI, where she is now employed as a scientist (Organic Analytical Chemist) in the Analytical Chemistry Laboratory.

Manawatu

Why don't Americans like New Zealand beef and sheep meat? According to Professor Milton Bailey of the Department of Food Science and Nutrition, University of Missouri, our meat has a "grassy" taste and is thus unacceptable to many people. Professor Bailey described how he collected volatiles from heated fats and analysed them by gas chromatography-mass spectrometry. He showed correlations

of these results with those of organoleptic evaluations of the cooked meats. Professor Bailey claimed that, from these results, he could identify the feed (grain or forage) that the animals had been finished on. However the chemical identity of the objectionable "grassy-flavoured" compounds have not been ascertained yet. A lively question time followed Professor Bailey's presentation.

Mr Bruce Sinclair, a technician in DSIR's Biotechnology Division, gave his view of scientific research to a Branch meeting on 5 October. Mr Sinclair showed how his work over the past 10 years has ranged over many activities, most of which were not related to his initial area of study, namely chemistry. He emphasised that technicians must be adaptable to take on a variety of necessary research and that they must have a good working relation with their supervising scientist. This may include, at times, suppressing the scientist's natural excessive enthusiasm and thus stopping unprofitable lines of work. After describing the many and varied projects in which he had been involved, Mr Sinclair was closely questioned on his attitude to work and the position of technicians in the DSIR. This was a most interesting and informative presentation by an enthusiastic technical practitioner of science.

Wellington

At the October meeting of the branch Mr. P.G. Eastwood, Petrochemicals Manager of Shell Chemicals New Zealand Limited presented the Shell Industrial Prize to Dr. Ian Miller of Carina Chemical Laboratories. This prize was awarded to Dr. Miller for his work in the development of the Durene process.

University News Continued . . .

Erskine Fellow. Dr Ferguson was formerly director of the Aeronomy Laboratory of the National Ocean and Atmospheric Administration, Boulder, Colorado, and is now Director of Research, Centre National de la Recherche Scientifique, Université de Paris-Sud. Other visitors include Dr Jay Wimalasena, Senior Lecturer in Chemistry at the University of Sri Jayewardenepura, Nugegoda (near Colombo) who is working with Professor Leon Phillips for

twelve months, and Dr Lawrie Dunn of the University of Tasmania, working with Dr Robert MacLagan until early December.

Dr Ward Robinson will be overseas on an Erskine Fellowship from mid-January to mid-April next year, visiting crystallographic laboratories in the USA, Germany and China. Dr Bryce Williamson is on leave until mid-January working at the Synchrotron Radiation Centre, University of Wisconsin.

BOOK REVIEWS

PESTICIDES-MINIMIZING THE RISKS: Nancy N. Ragsdale and Ronald J. Kuhr ed. American Chemical Society, Washington DC, 1987, 175 pages. ISBN 0-8412-1022-5. \$(US)41.95.

This small volume is one of the ACS Symposium Series and was developed from an Agrochemicals Divisional meeting in 1986. The 13 papers summarise a broad range of issues in pesticide science including toxicology, chemical/pest interactions, chemical/non-target organism/environment interactions and hazard evaluation. The presentations are clear, concise and accessible to a general scientific audience. The volume can therefore be recommended as an antidote to many current popular misconceptions. Pesticide science is complex and many faced with the majority of R & D effort now focussed on minimising the various risks.

Statistics are quoted that current pesticide usage in the USA is about 500 million kg/year, principally herbicides. This \$3 billion annual investment has been estimated to increase crops yields by a value of about \$12 billion. Modern intensive cropping, including horticulture, is very dependant on pesticides but pastoral agriculture as practiced in New Zealand is not a heavy user. This book does not debate whether pesticides are a necessity to produce sufficient food for the world or the merits and problems involved in alternative control strategies. It focuses on the scientific and regulatory issues involved in development and use of pesticides, particularly their toxicological and hazard evaluation.

Stringent regulatory requirements now ensure objective risks to the general population from food residues or to the environment at large are very low from standard usage of modern pesticides. However significant hazards are still present for applicators of many pesticides, particularly in tropical countries where training and protective measures are often inadequate. Localised environmental problems also can arise from improper use or disposal. Development of resistant organisms (very common in fungi/insects/mites and increasingly documented in weeds) or accelerated microbial breakdown in soil also pose a great risk, this

time to the agrochemical industry itself. The possible loss of effectiveness of useful and safe materials well before their patent life expires is a highly significant threat to company profitability and to farmers' choice of control options. This has encouraged research into means to overcome problems using chemical, biochemical, and management strategies.

A paper of particular interest is by a journalist on the problems of science/media interactions. There is a need for a much greater effort to accommodate each group's special requirements if public understanding of pesticide risk is to be lifted from the current low level. There are useful messages here for all who are concerned with public perceptions of science and chemistry in particular.

Patrick T. Holland
MAFTech, Ruakura
Hamilton

INTRODUCTION TO MICROSCALE HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY: Diado Ishii ed. VCH Verlagsgesellschaft, Weinheim, Basel, Cambridge, New York, 1988, 208 pages. DM118.

The press release which accompanies this book indicates that it is written as a textbook, but it is also suitable for professionals who are new to microscale liquid chromatography (lc) or who are taking a short course in it. This reviewer is an experienced biomedical chromatographer to whom instrumentation is largely 'black-box', inexperienced in microscale lc.

The book is clearly written, well illustrated and indexed. It consists of seven articles (Introduction, Instrumental Requirements, Microscale Columns, Detection Systems, Hyphenated Systems Using Microscale Columns, Post-Column Derivatisation and Applications) and an appendix of available packing materials. The number of authors has resulted in a degree of repetition of material between chapters.

There is a distinction between conventional lc, semi-micro (columns 1/10 conventional volumes), micro (columns 1/100 volume) and fast lc. The theoretical aspects (eg calculation of maximum connection-tube volume for a specific increase in elution volume) are thoroughly covered. There are series of

chromatograms to illustrate most points (eg effect of increase of injection volume).

I finished reading the book feeling that the technology of micro lc requires a lot of input from the chromatographer, semi-micro lc less so as more columns and suitable equipment are commercially available. The 'what's required' aspects are well covered in the book but the 'how to do it' much less so. The applications given from the biomedical area include several (eg urine catecholamines, serum theophylline, serum urate, cough medicine active ingredients) in which the increased difficulty of the technique appears to offer no advantage over currently used conventional lc or biochemical methods. The potential for interfacing micro lc to mass detectors and to IR detectors for unequivocal peak identification will give micro lc great advantage in biological and forensic applications.

I think this would be a useful book for a technologically inventive chromatographer wanting to scale down a system, and for teaching the theory of smaller-scale systems, and to read to find out if micro-scale lc has applications to your area now.

Dianne Webster
National Testing Centre
University of Auckland
School Of Medicine

METHANE CONVERSION: D.M. Bibby, C.D. Chang, R.F. Howe and S. Yurchak. ed. Elsevier, Amsterdam and New York, 1988, 742 pages. ISBN 0-444-42935-2. \$(US) 197.25

This book is a record of the proceedings of an International Symposium on the production of fuels and chemicals from natural gas, held at Auckland in April 1987. It is published as Volume 36 of a continuing series, "Studies in Surface Science and Catalysis". The text is divided into four sections: "Methane conversion via methanol" (30 papers, 342 pp.); "Alternative routes to methane conversion" (24 papers, 144 pp.); "Zeolites and other catalysts" (13 papers, 102 pp.) and "Commercialisation of the gas to gasoline process" (5 papers, 78 pp.). It contains both the invited oral, and the poster contributions, but there is no record of any discussion resulting from the presentations.

Published conference proceedings always have to overcome a healthy scepticism on the part of prospective purchasers if they are to achieve significant sales. Isn't it true that the work is likely to be outdated, because conference organisers insist on abstracts so far in advance, and that the really interesting new science will be published in an extended form elsewhere? Also, bearing in mind that we have to buy journals in any case, won't the price be very high for what extra information is unique? Generally, I believe that these criticisms are justified. What, then, can be said in favour of this volume, which should persuade a research group or library to buy a copy?

I think that there are several factors which make this book worth careful consideration. In the first place, over half the volume is taken up with invited contributions. This ensures not only that the articles are authoritative, but also that they fit together to provide a coherent picture. In the more academic papers, there is a nice balance between reviewing the chosen topics and presenting current research results. The list of authors is very impressive, and each has considerable standing in their own field.

What is perhaps most unusual about the book is the coupling of pure science with an overview of the background, engineering, and commercialisation of a major industrial process, the conversion of methanol to gasoline in the Mobil process plant at Motonui.

I found this to be a stimulating mixture, and I have often been tempted to dip into articles which I perhaps would normally not have read.

In summary, I enjoyed reading this book, and expect to continue reading and using it for some years to come. It provides a very convenient, and easily accessible source for teaching programmes at the tertiary level which try to put the various facets of "Chemistry in action" together. It is clearly of particular interest to readers in New Zealand, but its appeal should certainly not be limited to this audience.

C.G. Pope
Chemistry Dept
University of Otago

Metal-Nitrogen Multiple Bonding in Transition Metal Complexes

A.J. Nielson, Chemistry Department University of Auckland

Al Nielson is the 1988 recipient of the ICI Prize. A graduate of Auckland University, he carried out post-doctoral work in London between 1976 and 1980 at Imperial College with Prof. Sir Geoffrey Wilkinson FRS and Prof. D C Bradley FRS at Queen Mary College. He has held various temporary teaching positions in the Chemistry Department at Auckland University since 1981, and will take up a tutorship in Physico-chemistry in the School of Medicine, next year.



INTRODUCTION

The formation of multiple bonds between nitrogen atoms and transition metals is implicated in many industrial processes, lab syntheses and enzymatic transformations. For example, the Haber process used at Kapuni to form ammonia for the production of urea, utilises a heterogeneous iron catalyst which is believed to involve bonding of this type. The ammoxidation of propylene to acrylonitrile is an important industrial process worldwide which is thought to involve transfer of an allyl group to a molybdenum-nitrogen multiple bond. Perhaps the best known lab synthesis is the osmium catalysed oxyamination of olefins where the unsaturated compound reacts directly with an organoimido ligand. Another example is the reduction of aryl azides by VCl_2 or copper to give arylamines. Many studies of the enzymatic reduction of N_2 by nitrogenase have been carried out in which metal-nitrogen multiple bonding is again implicated, especially in the form of ligated dinitrogen hydrides. Metabolic oxidation of 1,1-dialkylhydrazine by cytochrome P-450 (an iron porphyrin) is also known to involve bonding of this type.

When studying metal-nitrogen multiple bonding in transition metal complexes it is convenient to use the primary nitrogen moiety RN rather than an NH ligand as the R group provides a solubility factor and as well is a useful tag for NMR spectroscopy. This primary alkyl or aryl nitrogen ligand coordinates to the metal in various ways depending on the number of protons which remain attached to the nitrogen atom. Where two protons remain, the primary amine can only coordinate to a metal via the lone pair of electrons (amine ligand, see a below). With one proton removed, the amine becomes negatively charged and acts as an amido ligand (RNH^-). In this case the lone pair can donate to the metal involved or bridge with

another (b and c below). With both protons removed the amine acts as a di-anionic ligand (imido ligand) in which case the lone pair may either donate to the metal or form a bridge with another (d and e). The RN^{2-} ligand can also bridge two metals (f) and the lone pair donate to a third (g).

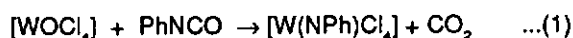
Each one of these bonding modes has been identified in a transition metal complex but only the early transition metals in the higher oxidation states exhibit the more interesting modes d and e. As the complexes are electron deficient they will seek electron density from any source, particularly from O_2 or H_2O , giving rise to oxo ligands which have electron pairs available to participate in maximising electron counts. Consequently most early transition metal complexes are air and moisture sensitive. However bench-top techniques for handling air sensitive complexes are now well developed so that there are few practical problems involved¹.

TERMINAL ORGANOIMIDO LIGANDS

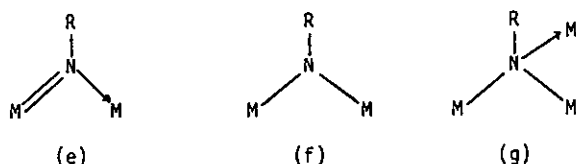
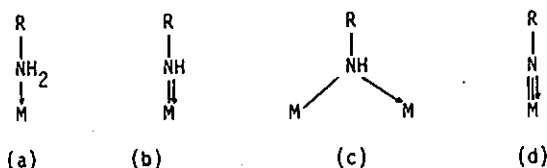
Our interest in multiple bonded nitrogen complexes arose in the early 1980s when a diverse chemistry was emerging for high-valent complexes containing multiple bonds to carbon. It appeared at the time that complexes with a terminally bound organoimido ligand ($\text{M} \equiv \text{NR}$) might participate in reactions similar to those being observed for the related alkylidene and alkylidyne complexes ($\text{M}=\text{CHR}$ and $\text{M}\equiv\text{CR}$) and that these reactions might lead to an understanding of the chemistry involved in processes where M-N multiple bonding was implicated.

Few organoimido complexes were known at this time that were suitable for further synthetic work, so that the initial studies carried out involved the development of preparative procedures. As oxo chemistry of the early transition metals was well developed, the synthetic approach taken was to exchange a terminal oxo group for an organoimido ligand.

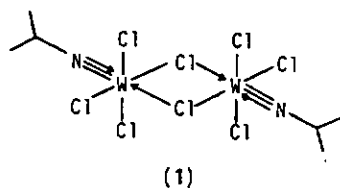
Tungsten oxytetrachloride was found to react with phenylisocyanate to form a simple but useful tungsten (VI) organoimido complex (equation (1))



Alkyl isocyanates performed the same reaction but t-butylisocyanate failed, presumably due to steric inhibition occurring during the exchange². Rhenium oxytetrachloride also reacted in this manner, but with molybdenum the reaction conditions caused reduction of MoOCl_4 , so that molybdenum organoimido complexes could not be prepared using this method. An X-ray crystal structure determination of the tungsten *iso*-propylimido complex showed a dimeric chloro bridge structure (1)

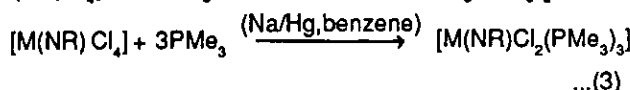
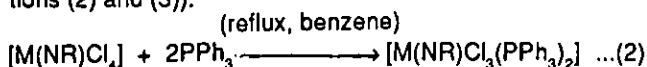


in which the imido ligand was terminal. The W-N bond length (1.72 Å) and W-N-C bond angle (176.8°) indicated that the nitrogen lone pair participated in bonding, so that the ligand was formally a 4-electron donor³.



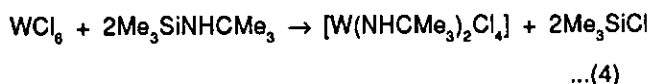
(1)

A variety of derivatives of the form $[W(NR)Cl_4(L)]$ ($M = W, Re$; $L = thf, pyridine, MeCN$, etc.) could be prepared from these dimers but the more interesting aspect of their chemistry was the particularly robust nature of the multiple bond which survived reduction of the metal to lower oxidation states² (equations (2) and (3)).

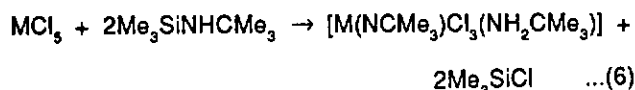


Further reduction of these complexes did not occur in aprotic solvents but in tetrahydrofuran, sodium-mercury amalgam reductions produced complexes containing the amido ligand ($-NHR$), the proton having arisen from breakdown of the solvent.

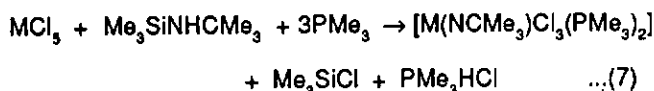
The failure of *t*-butylisocyanate to react with $WOCl_4$ to give a *t*-butylimido complex needed to be overcome as the *t*-butyl group was expected to be sufficiently bulky to provide a steric factor which could be useful for further synthetic work. A way round this problem was found using a reaction related to one known in metal-carbon chemistry whereby the steric bulk of five neo-pentyl alkyl groups around a tantalum centre had led to an alkylidene ligand by proton extraction. As NH protons are more acidic than CH protons, it appeared that proton extraction from one of two co-ordinated *t*-butylamido ligands ($-NHCM_e_3$) might generate the required imido linkage. Such a reaction would require the two amido ligands to coordinate *cis* to each other. The likelihood of this occurring seemed high as amido ligands involve a π -bonding component (bonding type b) and exert a high *trans* influence, so that a mutual *cis* configuration will always be preferred. A *trans* orientation would involve competition for the same π -bonding orbitals. On reacting WCl_6 with two equivalents of $Me_3SiNHCM_e_3$, it was found that a transient *bis*-amido complex formed which converted to an imido complex⁴ (equations (4) and (5)).



It was also of interest to develop routes to niobium and tantalum organoimido complexes as few of these were known. The pentachlorides are available commercially and these were found to react with $Me_3SiNHCM_e_3$ to produce *t*-butylimido complexes⁵ (equation (6)).



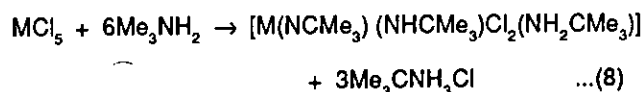
The reaction again involved the formation of two *cis* orientated *t*-butylamido ligands from which proton extraction occurred to give an amido and amine ligand. A similar reaction took place in the presence of trimethylphosphine whereby base promoted deprotonation of a single alkylamido ligand occurred (equation (7)).



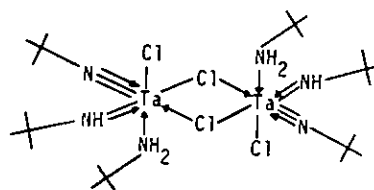
The $[M(NCM_e_3)Cl_3(NH_2CM_e_3)]$ complexes led to a variety of derivatives of the form $[M(NCM_e_3)Cl_3(L)_2]$ using phosphine and pyridine ligands but these were not always easily isolated. It was only when the bidentate ligands 2,2'-bipyridyl (bipy) and N,N,N',N' -tetramethylethylenediamine (tmed) were used that highly crystalline complexes $[M(NCM_e_3)Cl_3(bipy)]$ and $[M(NCM_e_3)Cl_3(tmed)]$ were obtained⁶. A similar set of reactions was found to occur for molybdenum starting with $MoCl_5$. When attempts were made to prepare isopropyl or ethylimido complexes from the pentachlorides and the relevant silylamine, solubility problems were encountered which prevented the proton transfer process from occurring.

MIXED BONDING MODE COMPLEXES

When the pentachlorides of niobium, tantalum or molybdenum were reacted with *t*-butylamine, further substitution of the chloro ligands took place than had occurred with the silylamine, resulting in a complex with imido, amido and amine ligands⁵ (equation (8)).



This complex was also formed when $[M(NCM_e_3)Cl_3(NH_2CM_e_3)]$ was treated with Me_3CNH_2 . Similarly reaction of MCl_5 with isopropylamine or ethylamine gave soluble $[M(NR)(NHR)Cl_2(NH_2R)]$ complexes. While $[M(NHR)_3Cl_2]$ is an alternative formulation for these complexes NMR spectral data indicated the presence of the different ligands. A crystal structure of the tantalum *t*-butyl complex showed the dimeric structure (2).

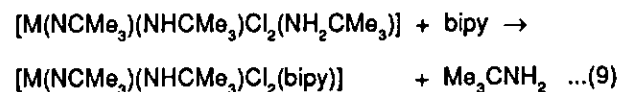


(2)

The structure was of particular interest as it was the first observed formation of the three possible bonding modes for the primary nitrogen moiety about the same metal centre. It had analogies to a complex prepared by Schrock containing the three possible bonding modes of carbon to a metal, namely alkylidene, alkylidene and alkyl ligands.

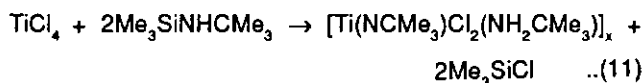
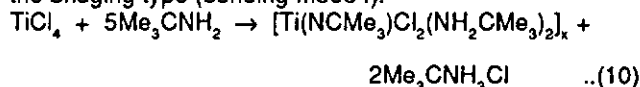
A feature of the reactions producing these mixed bonding mode complexes was the absence of intermediary complexes, particularly *bis*-amine species which were expected to be extremely reactive towards further deprotonation. If a close approach of a coordinated amine proton and a chloro ligand was a necessary prerequisite for dehydrohalogenation, it was expected that the process would be facilitated by steric congestion arising from *cis*-orientated amine ligands. On one occasion a stable *bis*-amine complex, $[W(NCM_e_3)Cl_3(NH_2CM_e_3)_2]$, was isolated and the X-ray crystal structure showed the amine ligands were orientated *trans*. The NH protons did not preferentially project towards any of the chloro ligands and no close contacts were made. As a consequence the NH and Cl moieties were unfavourably placed for further dehydrohalogenation to occur⁷.

As found for the other complexes prepared, the mixed bonding mode complexes reacted with bipy or tmed ligands to give highly crystalline solids (equation (9)) while phosphines gave products which were not easily identified⁶.



BRIDGING ORGANOIMIDO COMPLEXES

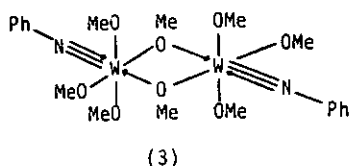
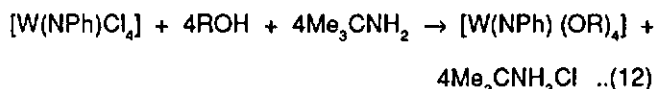
Having prepared imido complexes from WCl_6 and the MCl_5 species, it was of interest to establish how a tetrahalide would react. When $TiCl_4$ was treated with *t*-butylamine or $Me_3SiNHCM_3$, *t*-butylimido complexes were again formed (equation (10) and (11)) but these contained imido groups of the bridging type (bonding mode f).



iso-Propylamine and ethylamine reacted in a similar manner but gave polymeric complexes. The *t*-butylimido complexes reacted with bipy or tmed ligands to give complexes of the form $[Ti(NCMe_3)Cl_2(bipy)]_2$ and $[Ti(NCMe_3)Cl_2(tmed)]_2$ while unidentified polymeric species arose when similar reactions were carried out with the *iso*-propyl and ethylimido polymers⁸.

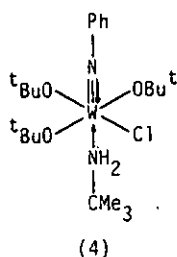
REACTIVITY STUDIES

Studies of the $M \equiv NR$ linkage reactivity showed that the multiple bond was surprisingly inert. Insertion reactions that were becoming common for the related alkylidene and alkylidyne complexes did not occur and the complexes did not react with unsaturated molecules as did organoimido complexes of osmium. However it was found that acids or hydride reagents readily cleaved the ligand to give amines. The general non-reactivity of the imido linkage appeared to arise from the lone pair on nitrogen remaining tightly bound to the metal so that the complex always achieved a maximum electron count. In attempting to overcome this problem, it was expected that a strong π -bonding ligand such as an alkoxide (RO^-) coordinating *trans* to the imido function might weaken the imido linkage if competitive π -donation from this ligand was sufficient to free the nitrogen lone pair. A series of alkoxide derivatives was prepared by exchanging the chloro ligands of the imido complexes using *t*-butylamine to remove the HCl produced⁹ (equation (12)).

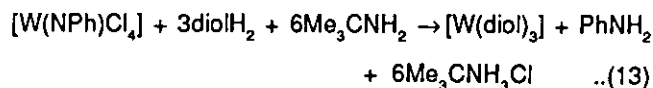


X-ray crystal structure determinations showed that the methoxy complex was dimeric (structure (3)) while the reaction with *t*-butanol formed a monomeric *trans*-alkoxy complex (structure (4)).

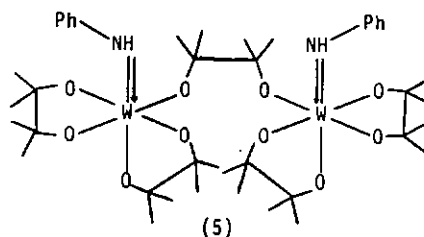
Although structure (3) showed an alkoxide in the desired *trans* position coordination, took place *via* a lone pair of electrons and this had no effect on the length of the W-N linkage.



When reactions were carried out using diols, it was found that three equivalents cleaved the imido function (equation (13)).

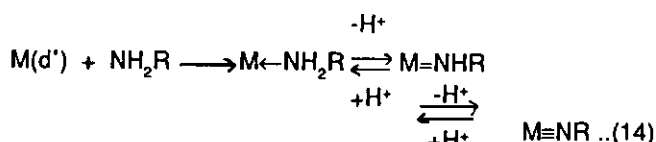


With two equivalents of $diolH_2$ $[W(diols)_3]$ was again formed but the reaction also produced another complex which was shown by X-ray crystallography to be the amido complex $[W_2(NHPh)_2(m-diols)(diols)_4]^{10}$ (structure (5)).



In this case the imido group had been protonated. Further reaction with $diolH_2$ and *t*-butylamine led to $[W(diols)_3]$ and $PhNH_2$.

The protonation reaction and those showing stepwise deprotonation of an amine indicated that under the right conditions formation of an imido group was reversible (equation (14)).



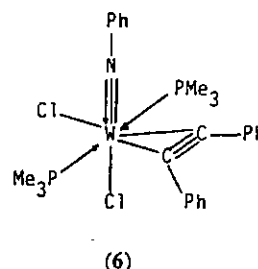
The apparent ease of protonation suggested that electrophiles such as CH_3^+ might also interact with the imido linkage to give an amido ligand ($-NR(CH_3)$). The strategy employed was to form an ionic complex which given sufficient density on the imido nitrogen would interact with the cation (equation (15)).



The imido complexes were however found to be surprisingly inert towards alkyl halides such as MeI, but ionic complexes were formed using trityl chloride (Ph_3CCl) and trityl ethoxide (Ph_3COEt)¹¹. The ethoxide ligand generated from Ph_3COEt was again expected to act as a strong π -bonding ligand *trans* to the imido function and release the nitrogen lone pair to allow interaction with the trityl carbocation. The ionic trityl complexes do react further but reliable evidence for the formation of an amido complex has yet to be gained.

All the d^0 organoimido complexes prepared showed little affinity for unsaturated organic compounds but the tungsten (IV) phosphine complexes (d^2 species) did interact with these molecules. Ethylene and propylene displaced one phosphine ligand from the $[W(NR)Cl_2(P)_3]$ complexes to give $[W(NR)Cl_2(CH_2=CHR)(P)_2]$ complexes¹². They could also be prepared by reduction of $[W(NR)Cl_3(P)_2]$ in the presence of the olefin. Similar complexes were also prepared from *cis* and *trans* 2-butene as well as from *iso*-butylene. With 3-methyl-2-butene and 2,3-dimethyl-2-butene the major product was $[W(NPh)Cl_2(PMe_3)_2]_x$ for which a dimeric imido bridge structure was proposed.

Acetylenes also displaced one phosphine ligand from $[W(NR)Cl_2(P)_3]$ to give complexes of the form $[W(NR)Cl_2(R'C \equiv CR')(P)_2]$. A crystal structure of $[W(NPh)Cl_2(PhC \equiv CPh)(PMe_3)_2]$ showed the imido and acetylene ligands lay *cis* (structure (6)).

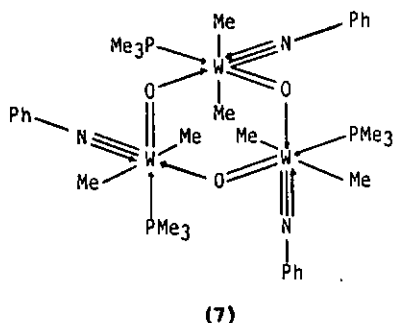


This was the expected orientation as theoretical calculations indicated that the two d electrons lie in orbitals associated with the equatorial plane so that stabilising π -backbonding to the unsaturated ligand would only occur with a *cis* olefin or acetylene. All the complexes were very stable and showed no tendency for the unsaturated ligand to interact with the organoimido linkage, even under strongly forcing conditions.

Very recent work has shown that the stabilising influence of the unsaturated ligand allows the preparation of some unusual complexes. In particular the reaction of $[W(NPh)Cl_2(PhC\equiv CPh)](PMe_3)_2$ with $PhC\equiv CCl$ gave $[W(NPh)(C\equiv CPh)_2(PhC\equiv CPh)(PMe_3)_2]$ containing both σ and π -bound acetylene ligands where normally polymerisation of the unsaturated molecules would occur.

COMPLEXES CONTAINING TWO MULTIPLE BONDED FUNCTIONS

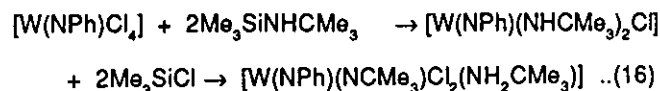
The early transition-metals in the higher oxidation states show a propensity for multiple bonding to a variety of ligands so that it was of interest to prepare complexes containing an organoimido ligand and another multiple bonded linkage. Our attempts to prepare imido complexes containing multiple bonded carbon were overtaken by the work of Schrock who prepared a variety of alkylidene complexes with *cis* orientated organoimido ligands¹³. We did however prepare oxo-imido complexes. Reaction of $WOCl_4$ with *t*-butylamine or $Me_3SiNHCM_3$ gave polymeric oxo-imido species which when treated with bipy gave $[W(NCMe_3)_2Cl_2(bipy)]$ and bipy-oxo complexes by a disproportionation reaction¹⁴. However reaction of $WOCl_4$ with $(Me_3Si)_2NPhMe$ followed by addition of bipy gave $[W(O)(NPhMe)Cl_2(bipy)]$. When $[W(NPh)Cl_4]$ was reacted with Me_2Mg , a small amount of the trimeric oxo-imido complex $[W(NPh)(m-O)(Me)_2(PMe_3)]_3$ was formed (structure (7)) in which the imido and oxo groups were orientated *cis* to each other¹⁵.



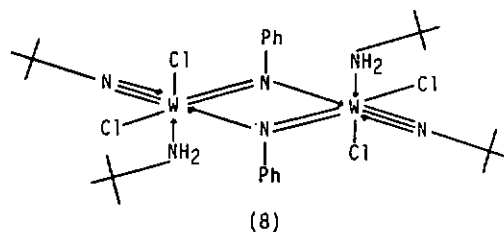
(7)

The oxo group apparently arose from breakdown of the 1,4-dioxane present in solutions of the dialkyl magnesium reagent.

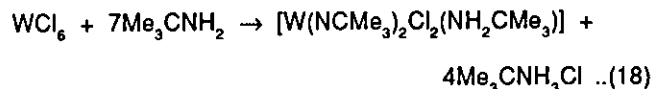
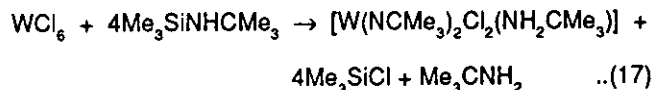
In attempting to form tungsten *bis*-organoimido complexes it was found that the reaction of dioxo species and isocyanates did not give imido groups. However, the strategy employed earlier, whereby proton transfer from two coordinated amido groups gave an imido ligand, was used to add a second imido function to a mono-imido complex⁴ (equation (16)).



An X-ray crystal structure of the complex¹⁶ (structure (8)) showed a dimeric molecule in which the *t*-butylimido group was terminal and the phenylimido group bridged to the adjacent metal *via* the nitrogen lone pair (bonding type e). It was subsequently found that a variety of *bis*-organoimido complexes could be prepared by varying the R group in the



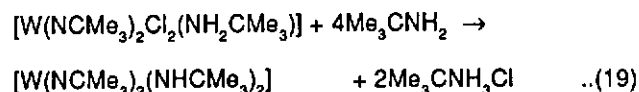
$[W(NR)Cl_4]$ complex and the silyl reagent Me_3SiNHR^4 . As the isocyanate reaction had not produced $[W(NCMe_3)_2Cl_4]$, a different method was employed to obtain *bis*-*t*-butylimido complexes (equations (17) and (18))⁴.



Of particular interest in these reactions was that with an MCl_6 species the interligand proton transfer process leading to an imido group occurred twice. This contrasted with the MCl_5 reactions mentioned earlier which produced an imido and an amido ligand.

The *bis*-organoimido complexes were found to react with a variety of ligands to give monomeric complexes of the form $[W(NR)_2Cl_2(L)_2]$ but again the bidentate ligand bipy gave the best results. X-ray crystal structure determinations of $[W(NPh)_2Cl_2(bipy)]$ ⁴ and $[W(NPh)(NCMe_3)_2Cl_2(bipy)]$ ¹⁴ showed the complexes were the imido analogues of the well known *cis* dioxo complex $[W(O)_2Cl_2(bipy)]$.

Attempts were also made to prepare *tris*-imido complexes (cf WO_3) using the methods outlined but further proton transfer did not occur in the *bis*-imido complexes. Reaction of $[W(NCMe_3)_2Cl_2(NH_2CM_3)]$ with further *t*-butylamine introduced two amido groups (equation (19)) but no proton abstraction occurred⁴.



When less bulky groups were present in the *bis*-organoimido complex the reaction did not proceed probably because there was insufficient bulk in the imido ligands to stabilise a tetrahedral coordination geometry.

CONCLUSION

A variety of organoimido complexes of the early transition-metals can now be prepared and these include mono-, *bis*-, oxo-imido, olefin-imido and acetylene-imido species. Initial studies of the imido linkage reactivity show an apparent inertness but this may well be put to good use in stabilising the high oxidation states of these metals for other specific studies. However it is envisaged that coordination of suitable ligands about complexes containing an organoimido ligand will cause changes in electronic states that will lead to a weakening of the metal-nitrogen multiple bond and thus allow further reactions to take place. In this way, further understanding of the processes involving the multiple bonding of nitrogen compounds to early transition metals will be gained.

ACKNOWLEDGEMENT

The initial studies of the organoimido complexes were carried out at Queen Mary College, University of London, London, England, under the supervision of Professor D.C. Bradley F.R.S. and supported by the S.R.C. The author gratefully acknowledges the early impetus provided by Professor Bradley

and continuing support and enthusiasm he has shown during the progress of this research.

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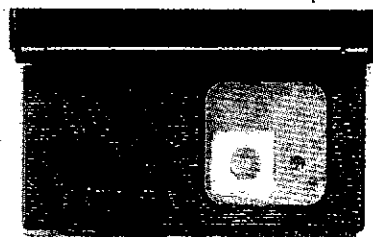
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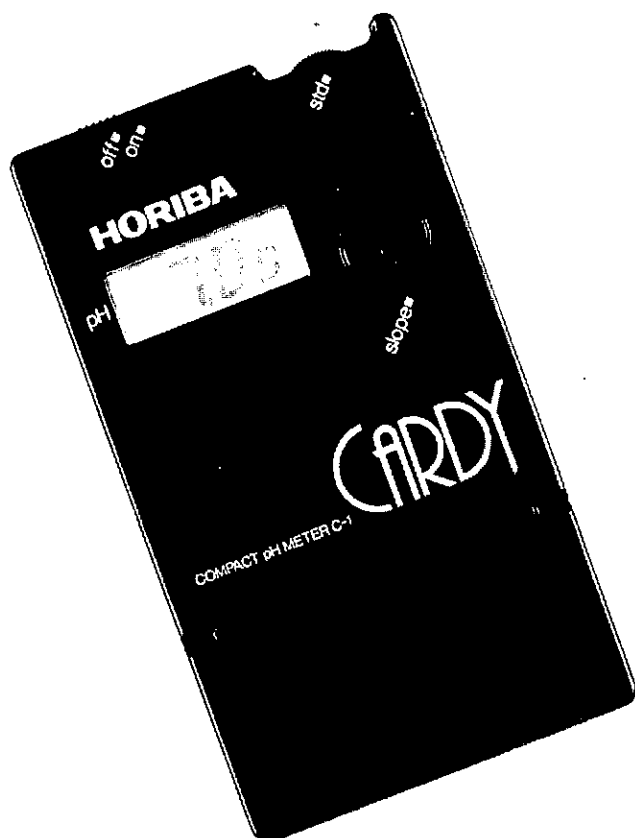
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The Horiba Cardy pH Meter C-1: Some Field Experiences

K.A.Rodgers, C. Cantrell and R.J.Sims, Geology Dept, University of Auckland



The Horiba Cardy pH meter is different! For starters it is small measuring only 95 x 95 x 9mm and weighing in at 40g. It is self contained. There is no probe. The sole accessories are two 10ml bottles of buffers, a 25ml bottle of distilled water, a pad of non-acid tissue squares and a plastic tweezers/screwdriver.

We've recently subjected two Cardys to rigorous field use in the central Pacific. We used them outside and in, all day and everyday and in all weathers. They came through with flying colours with but one reservation.

The conventional glass electrode has been modified and combined with the reference to form a sensor pad placed in a protected recess at the front of the instrument. Measurements and calibrations are made by placing a square of acid-free tissue across the pad and moistening it with a few drops of buffer or sample fluid. No more than 150 μ l is required. Temperature corrections are obtained by adjusting the pH settings of the buffer solutions with slope and calibration controls. Readings are to 0.01 pH units via a digital readout.

Measurements could be made directly on the sensor pad with moist materials such as fruit and fish, while soils, algae and mineral scrapings were measured by placing a few milligrams on a tissue square and then saturating the sample with distilled water. In this way results were easily obtained from very small amounts in contrast to the quantities normally required using conventional probes.

We found that stability was most quickly achieved and greatest precision obtained when a tissue was thoroughly saturated. In some instances when less moisture was available a distinct drift occurred, presumably due in part to the sample being measured equilibrating with atmospheric CO₂.

With soils and sediments quick stability required sufficient water to be added until the sample glistened.

However, the more the meter was used the more stable it became and it was found an advantage to pre-treat the sensor pad for some hours before use by leaving a tissue kept saturated with distilled water on the glass. On the whole the machine remained stable whether or not it was switched off, as long as it was used regularly.

Our initial reaction to the meter was somewhat cautious. It seemed a little bit gimmicky. On the one hand we needed something light and portable, on the other we wanted reliability, versatility and ease of use. In all but one area, the Cardy fulfilled our requirements.

Why o' why is the Cardy not supplied with a robust, protective carrying case, something serviceable that could, perhaps clip on your belt or dangle round your neck? Something too in which the buffer bottles and their tiny tops could stand and which would make field usage that much easier. The cardboard and polystyrene packaging the meter comes in does not take kindly to tropical downpours or salt spray but was found useful to contain and protect the equipment. In the end we kept meter and manual in one zip-up plastic bag and the bits and pieces in another. That criticism aside, the meter is a masterly piece of miniaturised instrumentation.

We used two meters, one in Fiji, the other in Tuvalu. The Tuvaluan meter proved a little disconcerting when we opened its factory sealed case; the pad recess was full of fluid. The manual tried to reassure us that "there appears sometimes some liquid ... but it is not uncommon." Despite this and a further note that "you can use after clean up", we were not entirely happy as the pad continued to fill after every aircraft flight! Nevertheless we continued to get consistent results from this meter which agreed closely with those of narrow-range Merk test strips we had taken along by way of security. However, there was a trap for young players with this fluid. We became somewhat blasé about it, using the meter "after clean up". In so doing, we once or twice overlooked the fact that after a good flow, some drops could be found adhering to the pad cover. If we then closed the cover during a reading, these drops could become transferred to our sample. The Fiji instrument afforded no problems in this area.

The first readings made with the Tuvalu meter were obtained at home in the days preceding the trip. One of us has a medical student son who, on being asked for "something to test", promptly produced drops of blood from his finger and saliva swabbed from his mouth. Two quick, sensible measurements left him convinced that this was just the thing for A & E. The ease with which the samples were handled and disposed is worth noting in these days of hepatitis and AIDS concerns.

In the end we measured body and plant fluids, soils, sediments, plants and their fruit, sea, river, lagoon and tank waters, rain drops caught on a tissue in-flight, drips from stalactites, puddles, moisture films on rocks and window glass, algal scrapings, cements, natural alumina gels, sludges, sump pit contents, tea, coffee, Coke, gin and tomato sauce. Our results fell in the range 3.5 to 12.5. Duplicate analyses on soils and sediments gave a reproducibility of better than 0.1 pH units. Liquids were generally half of this again, the qualification about

saturation, equilibration with atmosphere and consequential stability notwithstanding.

And we found too that for the conservator this gizmo couldn't be better. Paper or textile are laid directly across the pad, instead of the tissue and a few drops of water added for a rapid result doing no harm to the item in question.

The compactness of the meter has much to offer the field worker. However the method of measuring using tissues presents real, but not insuperable difficulties when working in windy conditions. We recommend calibrating the meter under shelter, unless you are blessed with the patience of Job or a highly dexterous field assistant. It is not easy to keep the tissue on the pad and uncontaminated, while at the same time saturating it with buffer - all in a Force 8 gale. However, we are able to give assurance that it can be done! Again a thoughtfully designed carry case could enhance the usefulness of the instrument in this respect.

Workers with gritty materials such as soils and sediments are

advised to keep a largish supply of distilled water to hand. The electrode pad is easily damaged. And in this connection DO NOT try to scrape off the annoying bits of adhesive which remain attached to the electrode after removal of the factory seal. That glass really does shatter very easily and the plastic surround is susceptible to some solvents! And ensure you have a good wad of conventional tissues on hand to blot the electrode clean between samples. The special tissue squares provided will vanish like the melting snow if used for this purpose. (Toilet paper proved insufficiently absorbent.)

For the lab worker who has everything, the Horiba Cardy comes not only in sensible black, the sort of thing you can put down in the field and never find again, but also delightful pink, and little boy blue. Unfortunately, at last time of asking the local New Zealand agents had only sensible black in stock. However, we are told that similar specific ion and salinity meters are now on the market. Can't wait to try 'em. Colour-coded field work could be all the rage this summer.

Organic Mass Spectrometry: An Update

H. Young

Division of Horticulture and Processing, DSIR, Private Bag, Auckland.

In the last 10 years or so mass spectrometry (MS) hardware has undergone dramatic changes and has matured into one of the recognised pieces of equipment found in all sorts of laboratories, be it biochemistry, physiology, forensic science, diagnostic medicine, or any in which chemistry is involved. Ten years ago analytical MS was almost exclusively carried out with electron impact (EI) ionisation. Now, EI is but one of the many methods, such as chemical ionisation, fast atom bombardment, thermospray, negative ion MS, and a host of other techniques, used in analytical MS. The early mass spectrometers, with their temperamental nature, have been replaced by stable, "almost user friendly" machines with much greater capabilities. The upper molecular weight limit has been extended to thousands, tens of thousands in specialised instruments, of daltons. Instruments with resolution in excess of 1×10^6 are commercially available. The increased performance however does not come free. Unlike computers, mass spectrometers are still beyond the reach of most laboratories although MS may be the most cost-effective, or the only practical way of solving a problem. A single beam Kratos MS30 mass spectrometer with its single electron impact source and maximum resolution of 10,000 cost \$60,000 in 1974. The modern replacement with what would now be considered as essential options like computer control, resolution to 50,000, multiple sources, etc, would cost over \$0.75M.

Some areas in which MS has played an important role include elucidation of biochemical pathways in plants and animals, the study of environmental pollutants at sensitivities and selectivities previously impracticable, the study of genetic and metabolic diseases in medicine and the sequencing of peptides and oligosaccharides.

This article is only an overview of what modern MS has to offer. Those wishing to get a better insight into modern MS for organic analysis should consult the excellent book by Chapman¹.

VACUUM SYSTEMS

A feature of modern mass spectrometers is the vastly improved pumping speed of the vacuum system, especially in the source region. Many of the new techniques impose a considerable gas load on the source pumping system. Tur-

bomolecular pumps with their high pumping speed and clean vacuum are replacing the traditional oil diffusion pumps in many systems. The advantages of faster pump down from a cold start, slightly lower water and power consumption and absence of backstreaming, however, must be balanced against the much higher maintenance and repair costs.

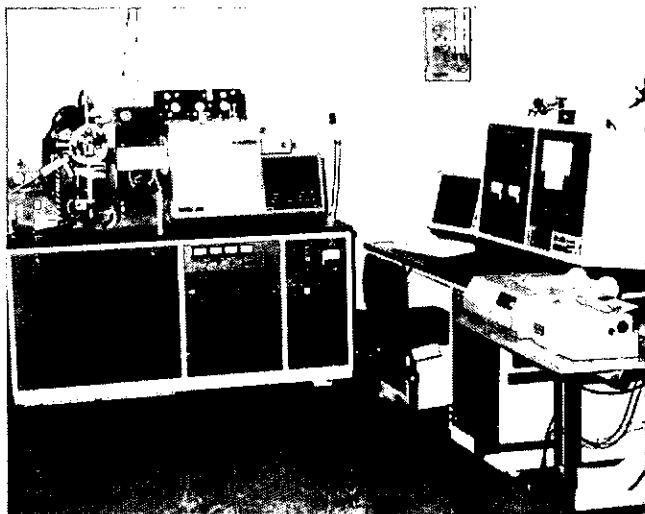
MASS SPECTROMETER TYPES

The sector instruments are still the most versatile analytical mass spectrometers currently available, despite the considerable improvements that have been made to the significantly lower cost quadrupole mass spectrometers. Practically all sector instruments sold now are of the double focusing design (an energy focusing electrostatic and a mass focusing magnetic sector). A high performance mass spectrometer for general purpose use should have a variety of sources and inlet systems and should be capable of resolution of 50,000-100,000* EI and chemical ionisation (CI) sensitivity should be of the order of 10^{-7} - 10^{-8} coulombs/ μ g for the molecular ion of methyl stearate. Alternatively, in terms more familiar to chemists, with the mass spectrometer set at 1000 (low) resolution and 1 second/decade scan rate, the molecular ion peak of 1 ng of methyl stearate introduced via a capillary GC column (peak width 3 seconds at 1/2 height) should have a signal-to-noise ratio greater than 1500. For comparison these figures are approximately 200 x better than those for the MS30 of 14 years ago. It should be noted that "chemical noise" is the limiting factor to sensitivity in most practical situations. Chemical noise arises from contamination of source and/or sample, carry over from previous samples, and in the case of gas chromatography - mass spectrometry (GC-MS), from column bleed.

Historically, sector instruments are notorious for their difficult operation. Much of this was attributed to magnet hysteresis, thermal drift, unstable calibration, slow scanning speed and tedious manual tuning. The hysteresis problem has been greatly reduced with the laminated core magnets now used, while water cooling has reduced the thermal magnet drift to an acceptable level. Together with interactive computer control

* Unless otherwise stated resolution refers to the 10% valley definition.

the new magnet designs have eliminated many of the drawbacks. For example, for low resolution spectra the VG-70SE mass spectrometer can be calibrated in less than 5 minutes and the calibration remains valid for several days. Scan cycle time over a mass range of 1 decade (e.g. 50-500) is down to 0.2 second. The major automation hurdle yet to be overcome in sector machines is tuning for resolution. Even with the new instruments it is still somewhat of a black art and can be very tedious.



An innovative sector instrument is the VG Tritech TS-250 with its hysteresis-free air-core magnet and double electrostatic sectors. Tuning is carried out from the keyboard as fixed slits are used. The stability of the system is such that accurate mass measurement is possible without using an internal standard. Maximum scan speed is 0.1 seconds/decade, mass range is up to 4000 daltons, and maximum resolution is 7500. Sensitivity is an order of magnitude lower than models with laminated magnets. This mass spectrometer would appear to have many of the convenient features of the quadrupole mass spectrometer, but without the limitations. If the technology can be improved to get better sensitivity, resolution and mass range, it could rival the laminated magnet sector machines as the preferred analytical mass spectrometer.

Quadrupole mass spectrometers have enjoyed considerable success, especially for routine analyses, because of their much lower cost (less than half that of sector instruments) and their ease of operation. However, apart from the tuning aspect, many of the operational advantages they have over the sector instruments have diminished. Despite the high mass discrimination inherent in quadrupole mass spectrometers, the mass range has been pushed up to 2000 daltons. Quadrupole mass spectrometers are capable only of nominal mass resolution and are less sensitive than the sector mass spectrometers. Nevertheless, their small physical size and relatively low cost have spawned basic bench-top models specially adapted for use as GC detectors.

The ion trap mass spectrometer (IT-MS) manufactured by Finnigan may be the beginning of a trend to have a MS detector on every GC in the laboratory. The IT-MS is a low cost (ca \$70,000), low resolution mass spectrometer with mass range of 20-650 daltons and scan speed compatible with capillary column GC. Ions are generated under conventional EI or CI conditions. However, with this device ion formation and mass analysis are performed sequentially. In order to get good dynamic range, the ionising electron beam is switched on for a variable (<0.1 to 25 msec) period of time, depending on sample size. It is difficult to compare sensitivity between the IT-MS and the conventional mass spectrometers because the available sensitivity specification is based on different compounds and conditions from that used for sensitivity measure-

ments in sector mass spectrometers. With small modifications to the electronics and changing the control software the IT-MS could be made to function in the MS/MS (see below) mode².

Fourier Transform MS (FT-MS) represents the other extreme in cost (\$1.5M+) and performance to the IT-MS. These mass spectrometers are based on the ion cyclotron principle and are the only mass spectrometers to incorporate superconducting magnets. Like the IT-MS the analysis is performed in time rather than in space, with the timing sequence fully computer controlled. High resolution is achieved by a change in the acquisition parameters, there are no mechanical adjustments such as slits to be altered or lenses to re-tune. Resolution of over 1×10^6 is possible. More importantly sensitivity actually increases with resolution. Thus high resolution accurate mass measurements on limited quantities of sample (ng or less) are feasible. The superconducting magnet is sufficiently stable for accurate mass determination without the use of an internal calibrant. FT-MS operates best at pressures of 10^{-8} torr or less. In early instruments, ion production and mass analysis were carried out in the same cell, making analysis of many samples (e.g. in gas chromatography-mass spectrometry) difficult if not impossible. In more recent versions a differentially pumped 2-cell design is used, with one cell used for the ionisation chamber and the other acting as the analyzer. This has made GC-MS and CI possible without compromising the analyzer vacuum and hence performance. Like the IT-MS, MS/MS experiments can be carried out simply by changing computer software. At present the cost excludes these instruments from the analytical laboratory, but like NMR, one day they will no doubt be just as common.

SOURCES AND INLET SYSTEMS

One area of MS which has undergone the most development in recent years is the method of ionisation. While the traditional EI source is still the workhorse, many new types of sources, each having its own special merits, are offered by the instrumental manufacturers. Some of the more important ones for the analytical laboratory are:-

1. The EI and CI sources are still the most widely used and are usually combined (EI/CI) into a single unit. It should be noted that the EI/CI source is a compromised design. A source that is dedicated EI or CI will usually out-perform the EI/CI source, e.g. an EI only source from VG Analytical is 5 x more sensitive than the EI/CI equivalent. These sources are easy to use and are applicable to a wide range of compounds, the main limitations being volatility and thermal stability of the sample. Typically, 1 ng of a compound would give reliable, interpretable spectra.

These sources generally come with several inlet ports to allow simultaneous installation of different inlet systems. The simplest inlet is the heated reservoir with a capillary leak. This inlet is only suitable for gases and low boiling liquids and is commonly used to admit calibration and reference compounds. For pure high boiling liquids and volatile solids the most convenient method of sample introduction is the direct insertion probe. The insulated probe tip has low thermal mass, a built-in heater and is frequently water (or other coolant) coolable. A temperature programmer to control the heating, typically at rates of 0.5-100 degrees C/sec, is highly desirable. The sample is transferred into the source in the gaseous state, albeit only over a distance of 10-15mm. In other cases where the sample is insufficiently volatile or is thermally unstable a modified technique may be used to advantage. In this technique the sample is coated on to a fine wire attached to the end of the probe. The coated wire is inserted into the source to within 1-2mm of the path of the electron beam. Electrical connections are provided so that the wire can be resistively heated. The best results are obtained with rapid heating which tends to

favour evaporation over thermal decomposition, but it does depend on the sample. Spectra of compounds with extremely low volatility such as underivatized sugars have been obtained by this method. The low energy ionisation of CI with its inherent low fragmentation of the molecular ion, is particularly suited to this "in-beam" technique. When used in conjunction with CI it is given the name **desorption chemical ionisation (DCI)**.

The direct insertion probe is really only suitable for single component samples, although with careful temperature control some crude fractionation of mixtures is possible. A better approach to mixture analysis is to link a chromatographic device to the mass spectrometer. The most common device-coupled to an EI or CI source to date is the GC for **gas chromatography-mass spectrometry (GC-MS)**. In the early days when only packed GC columns were available, separators were used to remove a proportion of the carrier gas (and inevitably some of the sample) since the high gas flow rate of the GC and the high vacuum of the mass spectrometer are largely incompatible. Typically 15-40% of the sample was transferred from GC to mass spectrometer. Today the flexible fused silica capillary columns can be connected directly to the source. The vacuum inside the source does not appear to degrade column performance. The fast pumping system fitted to modern mass spectrometers easily copes with the 1-2 ml/min of carrier gas (usually He) but the flow rate should be kept as low as consistent with good chromatography, since high source pressure does depress sensitivity. A 2 ml/min flow of He can reduce EI sensitivity by as much as 90%. In some source designs a portion of this can be regained by operating at reduced electron energy (28 eV vs 70 eV) to suppress the ionisation of the He, and hence reduce the space charge effect. We have found that with short (<15m), and with wide bore (>0.32mm id) columns a restrictor between the column and mass spectrometer improved dramatically the reproducibility of retention times.

Capillary GC allows excellent separation of mixtures but it is limited to volatile (derivatized if necessary) samples. The recent development of **super critical fluid chromatography** should address some of the drawbacks of GC for analyzing less volatile mixtures using the EI/CI source.

The interfacing of HPLC to mass spectrometers using a moving belt and a differentially pumped vacuum inlet has been superseded to a large degree by newer types of sources (see below).

2. **Fast atom bombardment (FAB) MS** has revolutionized the mass spectral analysis of very large or involatile molecules. Compounds ranging from simple salts to biopolymers with molecular weights of thousands of daltons have been successfully analyzed. Spectra of compounds with molecular weight greater than 10,000 are becoming relatively common. Examples of compounds which have been studied by FAB are oligosaccharides, peptides, nucleotides and organometallic compounds. In FAB analysis the sample dissolved in a high boiling solvent is applied to a metal target and when inserted into the FAB source it is bombarded with a beam of fast moving neutral atoms. A feature of FAB spectra is that often there is a very high background of peaks from the solvent, including some dimer and trimer ions. This can make interpretation of the spectra very difficult. The difficulty of controlling the rate of evaporation of the solvent and the fact that FAB is a surface phenomenon make it almost impossible to get a reliable spectrum for background subtraction. A recent variation is to introduce the sample as a continuous flow, the so called dynamic FAB. This mode of operation gives much cleaner spectra with few interfering peaks from the solvent matrix. A logical extension of this method would be to use the eluate from micro-bore column HPLC.

Xenon atoms are commonly used as the beam of neutral atoms but much less expensive argon may be used when



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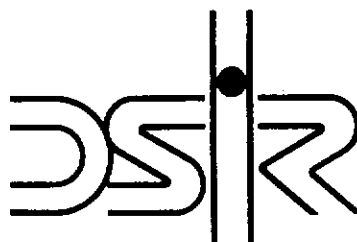
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sensitivity at high masses is not a problem. Neutral particles were at first thought to be essential but it has been found that charged particles may also be used. Recently FAB sources fitted with Cs ion guns have become available. The heavier Cs ions give better yields especially at higher masses, and have extended the usable mass range for FAB to over 15,000 daltons³. The most widely used solvent is glycerol but various solvents including triethylglycerol, DMSO, triethanolamine and even phosphoric acid have been reported in the literature⁴. Modifiers such as acids or bases may be added to enhance ionisation.

When using FAB for quantitative analysis, isotopically labelled internal standards must be used because spectral intensity is very sensitive to the presence of other species in the sample, e.g. traces of detergent from inadequately rinsed glassware could completely suppress the sample spectrum.

As a general purpose ionisation method for involatile molecules FAB has largely taken over from field desorption (FD), an experimentally demanding technique. Note however that FAB, although good at producing intact molecular ions, is not a soft ionising method like FD. FAB spectra generally exhibit fragmentation ions.

3. **Liquid chromatography-mass spectrometry (LC-MS)** is a rapidly expanding area in MS. One of the first methods of coping with the very high gas load imposed by LC is to deposit the LC eluate on to an endless polyimide conveyor belt which enters the source via a series of heated vacuum locks during which the solvent is removed. Ion production for this method is by EI, CI, and more recently by FAB.

Direct introduction via a pin-hole in a diaphragm has been used successfully but the thermospray source is now probably the most commonly used method for LC-MS. As the name implies the LC eluate is introduced into the ionisation chamber as a spray of micro-droplets produced by a combination of the source vacuum and a heated (ca 200 degrees C) metal

capillary inlet jet located at the source. A rotary pump, attached directly to the source opposite the inlet jet, removes the bulk of the solvent vapour. The analyte is ionised by a desorption mechanism due to an electric field induced on the droplets. A wide range of compounds can be analyzed by the thermospray LC-MS, amino acids and peptides, auxins, nucleosides and nucleotides to name a few. Unfortunately, for the thermospray mechanism to work, the eluent must contain at least 20% water and a volatile electrolyte (commonly ammonium acetate). To overcome these limitations a modified thermospray type source has been developed. In this system the capillary jet is maintained at 600 volts below the source block potential. The effect of this is to produce a glow discharge between the jet and the source, producing positive ions from the solvent molecules. These ions act as the reactant ions for ionising the sample. Not only is the water and the electrolyte no longer required in the mobile phase, but the source is more sensitive and inherently less noisy. The spectra show more fragmentation.

LC-MS has come a long way in the last few years but unless there are good reasons for using it, GC-MS would still be the method of choice for dealing with mixtures, even if the sample has to be derivatised. GC-MS is more convenient to use, has better chromatographic resolution, and can be used with the EI source for which there are large established databases of spectra for identifying unknowns.

4. Some of the more exotic ionisation techniques for dealing with large molecules are **laser desorption** and **plasma desorption** (using the high energy particles emitted by $^{252}_{98}\text{Cf}$). They are particularly useful for ionising large bio-molecules such as peptides in the 20,000 + dalton range⁵. These are highly specialised techniques and are available only in specialised laboratories.

DETECTORS

Detectors for mass spectrometry have shown few fundamental changes in the last 20 years. Electrical detection based

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on the electron multiplier is now almost universally used. One recent innovation is an electron multiplier with oxygen-insensitive and cost-saving replaceable dynodes*. Recently we have seen the electron multiplier being replaced by a photo multiplier system which does not suffer from loss of sensitivity on exposure to oxygen and does not discriminate against negative ions. A diode array detector with some of the better features (e.g. fast acquisition at maximum sensitivity) of the old cumbersome photoplate detection method, is now available commercially.

SPECIALISED MS TECHNIQUES

Accurate Mass Measurement. Before the days of computers mass measurements for determination of atomic composition were done by a manual peak-matching technique. This method is more accurate than the computer method but is time consuming, therefore wasteful in terms of sample requirement, and is recommended only for a limited number of ions. With computer scanning it is practical to accurately measure the masses of all the peaks in the spectrum.

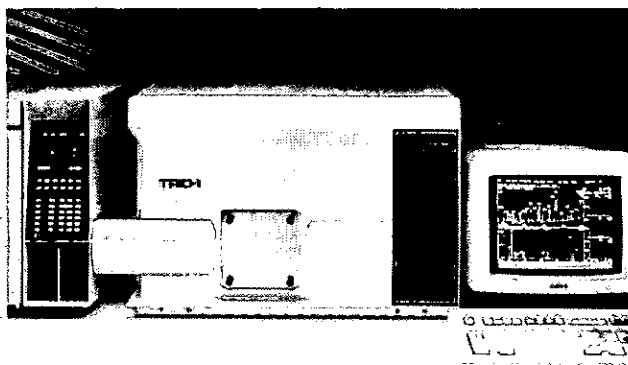
Accuracy of mass measurement is a function of the stability, rather than the resolution of the mass spectrometer, providing the resolution is sufficient to resolve the targeted peak from interfering peaks. Traditionally, mass measurement experiments were always carried out at resolution of 5000 or more because the stability of the mass spectrometers of the day was such that a calibrating compound had to be run at the same time as the sample. The resolution was necessary to resolve sample and calibrant peaks with the same nominal mass. On modern sector instruments accurate mass measurements may be made at low resolution by correcting for small drifts in the mass scale by including one or two peaks with known masses from a suitable lockmass compound such as tetraiodoethane. The only restriction is that the lockmass peaks and those of the sample do not interfere with each other. For limited mass ranges the more stable accelerating voltage scanning may be employed. Increasing the resolution slightly to 2000-3000 often increases the accuracy of the measurement dramatically as even with pure compounds there are usually interferences from background ions. This technique is frequently used for accurate mass measurements under CI conditions for which there are no compounds suitable for use as a general purpose internal calibrant (like perfluorokerosene with EI), and during capillary GC-MS since high scanning speed and high resolution are incompatible, except with FT-MS.

Negative Ion MS. Traditionally, organic MS was restricted to analyzing positive ions, even though in some instances with electron capturing molecules the yield of negative ions is much greater than the yield of positive ions. Furthermore, negative ion formation is a more selective process than positive ion formation so may be of advantage in analyzing mixtures. Negative ion MS is commonly used with CI or FAB. Most modern mass spectrometers are designed so that the negative ion mode can be easily selected. Indeed, positive and negative spectra may be acquired automatically on alternate scans. Even a combination of alternate EI, CI, positive and negative ion scans is possible.

Metastable Spectra. With double focusing sector mass spectrometers some fragmentation occurs in the field-free regions to give rise to metastable peaks which can be used to associate daughter ions with parent ions. In the days (not so long ago) when spectra were recorded on chart recorders, metastable peaks were recognised by their broad diffuse shapes. Data systems have eliminated these useful peaks from the normal printout. With computer control this information can be more conveniently obtained using "linked scan-

ning" in which the electrostatic sector voltage and the magnetic field strength are maintained in some fixed relationship to each other. Linked scanning may be used to record spectra of all daughter ions from a specified parent or vice versa, or to identify all the ions formed by the loss of a specified neutral species e.g. HCl. If desired, all three functions can be recorded from a single experiment in alternate scans. The scanning is fast enough to cope with wider capillary GC peaks.

Metastable ions in normal scans arise by unimolecular reactions or by collision with background gas molecules, **collision induced dissociation (CID)**. A collision gas (e.g. He) may be deliberately introduced into the field-free region to enhance the fragmentation process.



VG Trio-1 Benchtop GC-MS

Selective Ion Recording (SIR). SIR or Multiple Ion Monitoring is a technique whereby the mass spectrometer is used as a number of highly selective detectors, usually for GC or LC analysis. The mass spectrometer is stepped to the m/z of the selected ions in a cyclic fashion instead of continuously scanning over the mass range. Each mass channel monitored is analogous to a different GC detector and the results treated similarly, e.g. integrated for quantitation purposes. The time spent "looking" at each m/z value is increased from an average of less than 2 msec (scanning from m/z 500-50 at 1 sec/decade) to around 80 msec (allowing for settling time after each step) when monitoring 10 ions with a 1 second cycle time. Depending on the nature of the compound, sensitivity of 1 pg or less is easily achievable. With computerised mass spectrometers the setting up and calibration for SIR runs are relatively quick and straight-forward. SIR may be used in any of the ionisation modes and can also be implemented using metastable ions.

The selectivity may be further enhanced by operating the mass spectrometer at higher resolution to eliminate interferences from co-eluting contaminants with ions of the same nominal mass. This reduction of chemical noise may actually increase the detectability of trace components. A good example of this is the analysis of dioxins at 10,000 resolution on a sector mass spectrometer. Despite the reduced ion transmission (ca 5% of that at 1000 resolution), quantitation at the pg level is achieved. At 1000 resolution, though signals are 20 x larger, quantitation is difficult due to masking and distortion of the chromatographic peak by contaminants.

With quadrupole and ion trap mass spectrometers the stepping of the analyzer over the whole mass range poses no special problems since voltage changes only are involved. Stepping the magnetic field on sector mass spectrometers is slow (seconds) and lockmasses must be used to get reproducible results. Therefore for small m/z ranges the stepping is done by changing the accelerating/electrostatic sector voltages. Changing the accelerating voltage results in an unacceptably large loss of sensitivity if the ratio of the highest and the lowest mass is greater than ca 2:1. To overcome this a combination of voltage and magnetic field changes are used when large mass ranges are involved. On the assumption that

* ETP Pty Ltd, Australia

MAF New Zealand

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A laboratory with over 25 years experience of analytical chemistry is now able to offer a GC-MS service.

Instrument: HP5970, mass selection detector with electron ionisation, GC with autosampler.

Experience and expertise, particularly in the field of veterinary drug residue and pesticide testing (insecticides, anthelmintics, sulpha drugs, antibiotics, etc.) and vitamins, nutrients, trace metals, etc.

For further information on the services offered and the cost involved contact:

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Ruakura Agricultural Centre
Private Bag, Hamilton
Ph (071) 62839 Fax (071) 385012

MAF Tech Ruakura

at any one period of time only a narrow range of masses needs to be monitored (as is the case with GC-MS), these masses are organised into groups with narrow mass ranges. Voltage stepping is used within each group while the magnetic field is changed to effect the large mass changes between groups. This sequence actually helps to increase sensitivity since the dwell time at each mass can be much longer than if all the masses are monitored as a single group.

Tandem Mass Spectrometry. MS/MS, as the technique is frequently called, is the mass spectrometry of ions. A gas collision cell and a second mass analyzer are added in series with the conventional mass spectrometer. Ions emerging from the first analyzer are subjected to CID and the daughter ions analyzed by the second analyzer. This is in fact a more sophisticated approach to the linked scanning described above for double focusing sector instruments. Common instrument configurations for MS/MS are the triple quadrupole, the double focusing sector instrument plus double quadrupole hybrid, and linking two double focusing sector instruments. CID takes place in the first of the additional quadrupole sections. The higher sensitivity and resolution of the sector MS/MS instruments are advantageous in some instances, despite their higher initial cost. By a suitable choice of scanning modes between the two mass analyzers, daughter ion, parent ion and constant neutral loss spectra are easily obtained at high sensitivity. An obvious use of MS/MS is in the study of ion/molecule reactions but it is being increasingly used in analytical MS for analysis of mixtures since it provides another element of selectivity, and therefore more certainty in the result, or less clean-up of the sample is required.

With the ion trap and the FT-MS, MS/MS is implemented by tandem-in-time compared with the tandem-in-space requirements of the sector and quadrupole mass spectrometers. The significance of the tandem-in time arrangement is that only minor additions to the hardware are required to do the MS/MS experiment and the transmission losses of the conventional analyzers are avoided. Briefly, the sequence of events is ion formation, ion selection, CID and analysis of the product ions, all of which can be readily automated. In principle MS/MS/MS... can be implemented with the FT-MS without the cost and alignment problems of the multiple quadrupole and sector configurations. Ion trap MS/MS is still in the development phase but it offers exciting possibilities for affordable GC/MS/MS. A very readable article on the ion trap and ion trap MS/MS is found in *Spectra*.²

Currently there are no MS/MS instruments in NZ.

DATA MANIPULATION

Today data systems are an integral part of a mass spectrometer. The use of the computer system for controlling the hardware for some of the more complicated scan functions such as SIR and linked scanning have already been mentioned above.

In the days of chart recorders a scan was only taken when we decided that there was a spectrum of interest. With data systems the mass spectrometer is usually operated in a cyclic mode. A large amount of data can be acquired with each sample. In GC-MS runs it is not unusual to end up with as many as 4000 scans. Computer manipulation of the data has greatly enhanced the presentation and simplified the interpretation of the results. Such manipulation may simply involve plotting the spectrum with annotated m/z values along the x-axis but those who have manually annotated the mass scales of spectra recorded on UV chart paper would appreciate the advantages of a data system.

A plot of the sum of the intensity of all (or a selected range) of the peaks in the spectrum, the **total ion current (TIC)**, against time, is very useful in selecting the optimal scan. In GC-MS, this plot is the chromatogram obtained using the mass spectrometer as the GC detector. SIR may be simulated by

plotting the individual intensities of each ion. Such plots, also called **fragmentograms**, are useful in locating a particular compound, classes of compounds, or to check the homogeneity of GC peaks, especially with complex mixtures. Various **spectral enhancement** programs are available for extracting interference-free spectra from partially resolved GC peaks but their usefulness appears to be of limited value. There is just no substitution for good chromatography.

Background subtraction is particularly useful in GC-MS analysis. It can be a simple one to one subtraction or a known background peak can be used to correct for variations in mass spectrometer sensitivity, such as the result of source pressure changes during the elution of an intense GC peak. We have found that with the high sensitivity of the modern mass spectrometer it is seldom necessary (or indeed desirable) to use so much sample that source pressure changes are a problem. In practice, simple background subtraction usually gives a better result. Automatic selection of the background scan should be used with caution since it does not always retrieve the most appropriate scan.

Averaging of spectral intensities often enhances the quality of low intensity, low resolution spectra. Quality accurate mass data should be the average of several scans and include statistics on the variations. For very low intensity peaks an alternative method to get good accurate mass measurements is to record all data points taken, rather than just the usual centroid and intensity of the peaks. Multiple scans are summed together before calculating centroids and intensities. Accelerating voltage scanning is used in order to minimise mass-scale drift between scans.

For data acquired using SIR mode the usual GC type software for peak integration, quantitation, reporting, chromatogram overlaying for comparisons, etc are available.

The identification of compounds from MS data has been greatly simplified by the use of library searches. Large databases are available, especially for EI spectra and are incorporated in almost all MS data systems. There are also off-line systems available (e.g. the CIS database or the Cornell University STIRS system). As with all database searches the search parameters must be set with care and experience, and even

more important, the results used intelligently. It is well known that mass spectra are very dependent on the source conditions and analyzer type, and can vary from instrument to instrument.



CONCLUSION

Finnigan Ion-Trap Detector

I hope I have been able to give a brief overview of the type of analyses and results that could be accomplished on modern mass spectrometers. Versatile MS instrumentation is expensive and it would not be cost-effective for every laboratory in NZ with a need for mass spectral analysis to own one. If you would like to find out if your problems can be solved more effectively by MS there are several laboratories in NZ (see list elsewhere in this issue) which are equipped with modern mass spectrometers and staffed with specialist personnel who would be happy to help.

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MASS SPECTROMETRY FACILITIES IN NEW ZEALAND

A listing of current installations, application areas, and commercial availability.

MAGNETIC SECTOR INSTRUMENTS

Biotechnology Divn, DSIR, Palmerston North (contact: Dr John Shaw). VG70 - 250S, double focussing magnetic sector instrument with solids probe, GC and HPLC for sample inlet, EI, CI, Cs ion, thermospray, and plasmaspray ionisation modes.

Chemistry Division, DSIR Christchurch (Contact: Dr D Winter) - refer University of Canterbury entry.

Chemistry Division, DSIR, Wellington (Contact: Dr Lawrence Porter) VG70-250S, double focussing magnetic sector instrument with septum, solids, DCI and FAB probes, and capillary GC for sample inlet, EI/CI, +/- ion, Cs ion gun for liquid SIMS (FAB), ACE, and linked scans for parent/daughter ions.

Specialists in trace analyses by GC SIR and accurate mass CI. Available for contract work.

Division of Horticulture & Processing, DSIR, Auckland (Contact: Dr Harry Young). VG - 70SE, double focussing magnetic sector instrument with solids and DCI probes, and capillary GC for sample inlet, FAB source, MW up to 16,000, SIR, and metastable scanning. Current applications include

all aspects of GC/MS and general organic analysis. Available as a commercial service.

MAFTech, Ruakura Agricultural Research Centre, Hamilton (contact: Dr Patrick T Holland). Kratos MS80RFA high resolution magnetic sector instrument with solids. DCI and FAB probes, and capillary GC for sample inlet, accurate mass assignment (incl. for capillary GC/MS), +/- CI, library search facilities, and MS/MS via linked scanning.

Current applications include pesticide residues and environmental screening, mycotoxins, biomedical stable isotope dilution studies, and general compound identification by elemental composition determination. Available as a commercial service.

University of Canterbury (Chemistry Department) Christchurch (Contact: Dr G J Wright). Kratos MS80RFA and MS902 double focussing magnetic sector instruments with DIP/DEI/DCI/FAB probes, and GC and gas inlets, EI, CI, ACE, DEI, DCI, +/- ionisation, FAB, and metastables monitoring, plus DS90 data system.

Current applications include general organic mass spec-

trometry, forensic toxicology, illicit drug analyses, environmental and industrial analyses. Available as a commercial service through Government Analyst, DSIR, Christchurch.

QUADRUPOLE SYSTEMS

Chemistry Division, DSIR, Christchurch (contact: Dr D Winter).

(i) Hewlett Packard 5970 MSD, with HP5890 GC with autosampler, HP9000 series computer with UNIX, and HP-UX MSD ChemStation.

(ii) Hewlett Packard 5982A GC/MS with EI, CI, gas inlet and DIP/DEI/DCI probe.

Current applications are in forensic toxicology and illicit drug analyses. Available as a commercial service.

Chemistry Division, DSIR, Wellington (Contacts: various - see below).

(i) Extranuclear Laboratories spectrEL quadrupole MS, gas inlet mode, coupled to temperature programmed desorption furnace. Specialises in gas analysis. Available for contract work (contact: Mrs Linda Parker, Dr Donald Rogers).

(ii) Hewlett Packard 5970B MSD with capillary GC and autoinjector. Specialises in food components, pesticide residues, and environmental contaminants. Available for contract work (contact: Dr D J Hannah, Mr J A Toucher).

(iii) Hewlett Packard 5970B MSD, with capillary GC. Specialises in drugs and toxicological compound identification and analysis (contact: Dr Alan Stowell, Mr Eric Cairns).

(iv) - in conjunction with Industrial Processing Divn, DSIR. Hewlett Packard 5970B MSD with capillary GC. Specialises in organic and trace organic analysis and identification. Available for contract work. (contact: Dr Lawrence Porter.)

(v) Shimadzu QP1000 capillary GC/MS, with EI/CI and pyrolysis furnace inlet. Available for contract work (contact: Dr Richard Furneux, Ms Jennifer Mason).

Forest Research Institute, Rotorua (contact: Dr Robert A Franich). Hewlett Packard 5985B quadrupole GC/MS with DIP, and direct/open-split capillary GC inlets, CI/EI, pyrolysis GC inlet for polymeric solids and liquids, and data system. Complementary information can also be provided by GC-FTIR.

Current applications are numerous, and include wood preservatives, wood/polymer composites, adhesives, chemically modified wood and fibre, and wood pulping by-products. Available as a commercial service.

Ivon Watkins-Dow Ltd, New Plymouth (contact: Mr Dave Wills). Hewlett Packard HP5995, with GC and DIP inlets.

Current applications in environmental monitoring. Available as a commercial service.

MAFTech, Chemical Residues Laboratory, Wallaceville (contact: A D Sheppard). Hewlett Packard HP 5970 MSD with capillary GC, and 100 place autosampler. Available as a commercial service.

New Zealand Dairy Research Institute, Palmerston North (contact: Dr Owen Mills) Shimadzu QP1000 GC/MS with capillary GC direct inlet, solids inlet, and NBS/NIH/EPA spectral library.

University of Otago, Dept of Pharmacy, Dunedin (Contact: Mr D M Schmierer). Hewlett Packard HP5970 MSD with 5980 capillary GC. EI, with data manipulation facilities.

Current applications include routine structural analysis, prostoglandin metabolite quantification, head space analysis and drug analysis. Available as a commercial service.

Victoria University, Chemistry Dept, Wellington (contact: Dr J T Craig). Hewlett Packard HP5995 GC/MS with capillary GC (open split) and solids probe.

ISOTOPE MASS SPECTROMETERS

Grasslands Division, DSIR, Palmerston North (contact: Dr J R Crush). VG SIRA 9 stable isotope ratio analyser with microprocessor controlled inlet system.

Institute of Nuclear Sciences, DSIR, Wellington (contact: various - see below).

(i) VG Micromass 602D stable isotope ratio mass spectrometer, with gas source and dual inlet, and semi-automated double collector. Used for measurement of D/H ratios in waters and other fluids applied to hydrological and geothermal studies. (contact Dr M K Stewart).

(ii) VG MM30B solid source isotope ratio mass spectrometer, with single collector, automated filament control and peak switching. Used for Sr isotopic ratios in geological dating by the Rb/Sr method, and precision isotope ratio and element abundance measurements (eg Sr, Nd). (contact: Dr I J Graham).

(iii) VG Micromass 1202E, stable isotope ratio mass spectrometer, with gas source, dual inlet and semi-automated double collector. Used for S-isotope ratio measurements of sulphide and sulphate minerals, sulphur in rocks, oils, geothermal and volcanic gases and waters. (contact: Dr B W Robinson).

(iv) AEI MS10 (modified) isotope ratio mass spectrometer, with gas source, static mode, and on-line system for extraction of argon from rocks. Used for low level inert gas isotopic ratio measurements, mainly Ar, for geological dating by the K/Ar method. (currently 500,000+ yr, but younger system under development). (contact: Dr C J Adams).

(v) Tandem Accelerator Mass Spectrometer, with Cs sputter source. Presently operating as a radiocarbon research facility for C-14 dating of small samples in archaeology, atmospheric, oceanographic and earth science studies. (contact: Dr R J Sparks).

(vi) Nuclide RMS 6-60 stable isotope ratio mass spectrometer, with gas source dual inlet, and fully automated triple collector. Used for oxygen isotope ratio measurements of waters, carbonates, silicates and sulphates in hydrology and the earth and atmospheric sciences. Also carbon isotope ratio measurements of organic material, oil and natural gas, geothermal gas, and carbonates in environmental, geochemical and medical studies. (contact: Dr J R Hulston, Dr C A M Brenninkmeijer, Dr G J Llyon)

All of the above instruments at INS are available as a commercial service.

Membership Changes

The following were approved at the November meeting of Standing Committee:

Fellow:

Gainsford, Graeme, Scientist, Chem Div, DSIR, Wellington.

Johnstone, Howard James, Reader in Chemistry, Victoria.

Member:

Somerville, Robyn Faith, Scientist, Chem. Div. DSIR, Auckland.

Causser, Jay Elizabeth, Tutor, Christchurch Polytech.

Anderson, Bryan Frederick (Dr), S.R.O, Massey.

Norris, Gillian Ema (Dr), R.O. Massey.

Associate:

Hue, Wen (Ms), PhD Student, Canterbury.

Naidu, Rajinda, Graduate, Research Assistant, Univ. South Pacific, Fiji.

Waller, Andrew Grant, PhD Student, Canterbury.

Kilmartin, Paul Andrew, BSc Hons Student, Victoria.

Reinstated:

Roper, Jack (M), o'seas.

Resignations:

B.Crane (Waik), H.J. Van Eckenvordt, M.J. Queree (Welln), D. Winter (Cant), J.M. Duncan. (nee Mackintosh), J.S. DeCourcy (o'seas).

Obituary:

The following deaths are noted with regret: Sir F.J. Llewellyn (o'seas), Prof. J. Vaughan (Cant.), A.J. Sutherland (o'seas).

MASS SPECTROMETRY

Applied Mass Spectrometry From Leybold AG

Leybold AG offer a wide range of mass spectrometer systems for a variety of applications including leak detection, gas analysis and process control. A recent addition to the line is the first portable MS gas analyser with built-in intelligence for generating instantaneous results in the field.

A sampling of the Leybold instrumentation in this area is given below. Details of the full range may be obtained from the New Zealand agents, Javac (NZ) Ltd.

QUADREX 200

Quadrupole Mass Spectrometer for the mass range 0-200 amu, with the following applications:

- residual gas analysis
- process gas analysis
- process gas control
- evaporation plants
- sputtering plants
- plasma etching plants
- all types of vacuum systems.

Quadrex 200 - an instrument which offers everything expected from a modern quadrupole mass spectrometer. Various display formats enable the user to get a clear picture of the measured values that really count in the process environment. Data on the real partial pressures for routine analysis, bar graph displays or analog spectra for residual gas analysis, timed monitoring of single components for fault analysis, selected gases displayed for process control, dynamic changes displayed during process runs - to name just a few of the many standard features the QUADREX 200 offers - and without any additional help from an external computer. And not to be forgotten, the built-in Spectra Library that lets the user compare real-time spectra with the stored reference spectra. The Spectra Library is preprogrammed with 34 types of gases that most frequently occur in vacuum processes. Plus, six of the user's own typical spectra can also be entered.

Easy operation, simple-to-read displays, and easy-to-follow interpretation guides by using the built-in library even allow a beginner in mass spectrometry to make use of the advantages the Quadrex 200 offers in gas analysis and process control within a short time. And for those who want a more in-depth analysis of the reliable results they get from the Quadrex 200, the standard RS 232 interface is available

which allows the Quadrex 200 to be joined up with an external computer.

For further information please circle no. 8 on reader reply card.

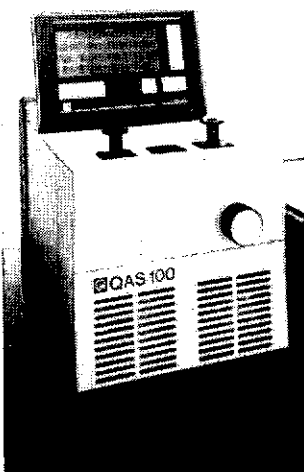
QAS 100

The first portable gas analysis system with built-in intelligence.

The Leybold QAS 100 mass spectrometer sets new standards of modern mobile gas analysis.

Gas analysis problems - especially in response to progressively stringent legislation - are increasingly demanding mobile gas analysis systems that can be rapidly applied on-site. They should be easy to operate and meet the different requirements concerned. The QAS fulfils all these requirements, being transportable in any truck or van and powered by a stand-by unit, so that measurements on-site are possible at all times without mains connection.

The first analytical results are available within a few minutes.



The QAS 100 is well-suited to the following applications of environmental analysis:

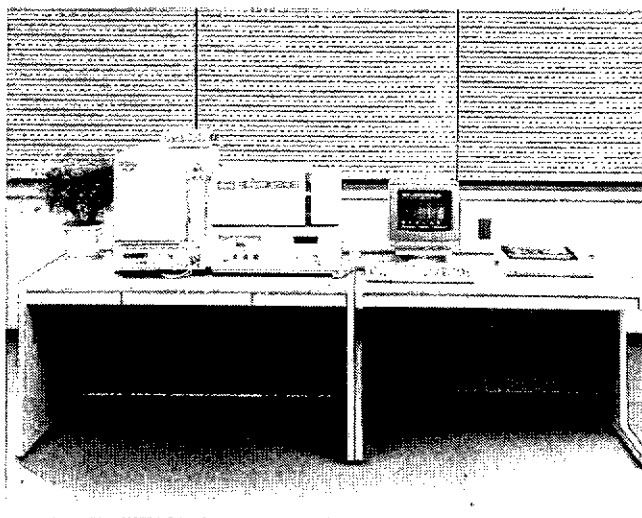
- ground air analysis during refuse dump probing
- identification and assessment of gaseous contaminants
- gas analysis of internal combustion engines (on-line)

For further information please circle no. 6 on reader reply card.

Ultratest UL 500 - Intelligent automatic helium leak detector

The Ultratest UL 500 is an intelligent helium leak detector of a generation, whose equipment makes it suitable for both vacuum and overpressure leak detection.

Its primary application area is



Shimadzu GC/MS

Shimadzu Corporation are pleased to announce the international release of their Bench Top Gas Chromatograph/Mass Spectrometer System, The QP2000.

This features their latest Gas Chromatograph, the GC14A, specifically designed for Capillary Column GC, directly coupled to the Mass Spectrometer. The MS includes a Variable Temperature E.I. Ion Source, high performance Hyperbolic Quadrupole Mass Filter and a 16 Stage Off Axis Electron Multiplier. The evacuation system includes Turbo Molecular and Rotary Pumps.

quality control of components in the:

- electrical industry
- electronic industry
- refrigeration and air-conditioning industry
- vehicle industry
- tank construction
- packaging industry
- precision engineering
- R&D

The instrument is used to test components whose integrity is a precondition of durable operation and safety, starting with the smallest specimens of some mm³ in volume all the way up to large components with volumes of several hundreds of litres. It is also just as suitable for one-off tests as for the leak-testing of large numbers of items within short cycle times.

The Ultratest UL 500 features a virtually hydrocarbon-free inlet system, making it the correct instrument for specimens or systems of the highest purity. Through the application of two microprocessors, the instrument features unique leak detection know-how available to the operator on an applications-tailored

The whole system is under the control of the Built in Micro Processor, complete with 20MB Hard Disc and 1MB Floppy Disc. 12" Colour VDU, full keyboard and Dot Matrix Printer.

Software includes Auto Tuning, Automatic Data Collecting, Self Diagnostics, and Single Ion Monitoring.

Full Data Processing is provided with Mass Spectral Plots, total ion and Multi Ion Chromatograms and background subtraction.

Options include Library Search and Direct Sample Inlet Probe.

For further information please circle no. 7 on reader reply card.

A completely new vacuum concept produces extremely short pumpdown and response times, the latter typically of the order of <1 s at volumes as high as up to 10 l.

The provision of a low-temperature trap with the aid of liquid nitrogen is no longer necessary. The instrument can also be easily handled by untrained operators. On a large synoptical LCD-display, leakage rates between 2.10⁻¹⁰ and 100 mbar.l.s⁻¹ can be easily read in analogue or digital form even from 5 m distance. The entire automatic start-up procedure is completed in less than 5 minutes after switch-on. Even site-to-site applications could not be easier: simply switch off the instrument and, without waiting, run it over to the next application on its large easy-running castors. A testpiece can already be leak-checked from atmospheric pressure and from 100 mbar in a precisely quantitative form.

For further information please circle no. 9 on reader reply card.

AMTAC
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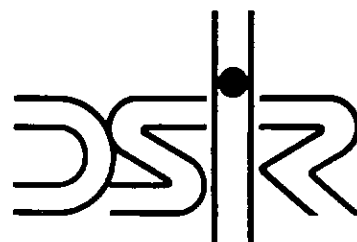


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CHEMISTRY DIVISION FELLOWSHIPS

Chemistry Division is offering annually up to four fellowships in Chemistry to Honours or Ph.D graduates of New Zealand Universities. The intention is to sponsor chemistry research in New Zealand; encourage new graduates to stay (at least temporarily) in New Zealand; to provide more graduates with job experience in DSIR and to attract good research staff to the Division. The fellowships will be tenable for periods of 6 to 12 months, and will comprise salary and travelling expenses. Further information may be obtained from:

**Mrs K Martin, Personnel Officer,
Chemistry Division, Private Bag, Petone.
Telephone (04) 690-321**

**GOOD
NEWS FOR
VACUUM USERS**



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PRODUCT NEWS

MGA 2000 Respiratory Gas Analyser

A versatile mass spectrometer designed for today with tomorrow in mind whatever the application...

Airspec is dedicated to the design and manufacture of instruments for respiratory gas analysis. The technical team includes physicists, chemists, engineers and computer experts. Their considerable combined expertise is continuously directed towards satisfying customers measurement requirements.

The MGA 2000 is particularly appropriate for use in the following applications:

Lung function testing, exercise testing, sports medicine, aviation medicine, intensive care monitoring, anaesthetic monitoring, bronchoscopy, hyperbaric respiratory monitoring.

The exceptional design concept of the MGA 2000 allows the user to choose how data is selected, processed and presented, to obtain the optimum analysis for each application. Up to 4 analogue signals from other transducers, such as flow and heart rate monitors, can be acquired and processed through the analyser to provide a single source of all relevant data for computation of physiological parameters.

The analyser can be configured through its customised keypad or its RS232 port from another computer as part of an automated system.

Airspec is able to provide a range of IBM PC applications software support, from high speed data acquisition and disk storage, to full system software in its PULMOLAB 2000 products.

The MGA 2000 affords the elegance of a single technique for the analysis of all respiratory gases. The quadrupole analyser provides precise multigas analysis, combined with exceedingly fast response times, at low sample flow rates.

Designed for those who need to see and record continuously...

- The dynamic change of up to 8 gases in a mixture.
- At response times as fast as 40ms (0-90%).
- With a complete analysis every 20 ms for all gases.
- Taking only 4 ml/min sample
- With large screen real time analogue displays.
- On line to any computer.

For further information please circle no. 5 on reader reply card.

Vacuum Equipment Joint Venture

Users of vacuum equipment will gain considerable benefit from the recent formation of a joint venture company in New Zealand.

The partners in the venture are Javac Pty Ltd, an Australian company, and Levingston Bros Ltd in Auckland, who, until recently, were selling agents for Javac Pty Ltd. The new Company is Javac (NZ) Ltd.

The advantage of the new venture to vacuum users lies in an increased product range, greater availability of products ex-stock, and easier access to international technical expertise.

Javac (NZ) Ltd will be marketing a complete range of vacuum equipment for laboratories and industry, including Javac and Leybold vacuum pumps of all types, freeze dryers, leak detectors, cryogenics, partial pressure analysers, mass spectrometers, and more. The company will confine its activities to vacuum equipment with a view to becoming the country's leading specialists in the field.

Javac Pty have on their staff experts in vacuum technology drawn from around the world, and this pool of knowledge will be available to the local company for providing advice and service to their customers.

Javac (NZ) Ltd will, at least for the time being, operate out of Levingston Bros Ltd premises in Penrose. Further information is available from Chris Price, Sales Engineer (Vacuum Technology), Javac (NZ) Ltd, 348 Church Street, Penrose. Phone (09) 590 450. Fax (09) 591 196.

For further information please circle no. 4 on reader reply card.

Covering The Field In Analytical Instruments

Varian manufactures two ranges of UV-Visible and NIR spectrophotometers - the DMS single monochromator UV-Vis spectrophotometer Series and the CARY double monochromator research UV-Vis and NIR spectrophotometer Series.

The DMS Series consists of five low to mid priced instruments which offer excellent performance at moderate price. All are double-beam, microcomputer controlled to ensure high performance and inherent system reliability, says Wilton Instruments, New Zealand agents for Varian.

All models allow high accuracy measurements to be performed with wavelength scanning if desired and operate over the range 190 nm to 900 nm.

DMS 300 is the new top-of-the-line DMS model, providing all the capabilities of the popular DMS 200 Series and much more. With applications modules, user programmability and on-line final result calculations, the DMS 300 provides unmatched flexibility for routine and research applications. DMS 200 and 100S are optimised for real time presentation of spectral data, spectral computation and hard copy report generation. With the addition of the DS-15 Data Station their flexibility is extended with powerful applications software and data storage capability.

DMS 90 offers many similar features to DMS 200 and 100S but without video facility, producing spectra on X-Y or strip chart recorders. The DMS 90 is an excellent general purpose spectrophotometer with facilities for scanning, single and multi-wavelength analysis and kinetics studies.

DMS 80 is most suitable for single wavelength measurements, and forms high performance analytical systems with the Routine Sampler, Routine Sampler/Batch Sampler and Programmable Cell Changer accessories.

For further information please circle no. 3 on reader reply card.

A & D Electronic Balances

A & D Company Limited of Japan manufacture a comprehensive range of balances, with so many capacities and resolutions that there is a model to suit any need from 0.01mg to 60kg.

The EK and EW series balances offer accuracy of 1:12000 and 1:3000 respectively. Both are available with internal battery pack for complete portability, and RS-232C interface.

EP series precision industrial balances are ideal for bulk weighing and counting, up to 200,000 pieces at a time, 60kg total maximum.

ER-A series laboratory standard balances have advanced design features with high resolutions. Standard features include glassed-in weighing chamber, underhook, and moisture and dust-proof keyboard. ER-A series balances calculate to 10 decimal places.

FX and FY series balances can perform all your weighing



needs in any of 13 versatile models featuring 10 measurement units.

A&D complete the range with a precision compact moisture content balance, a compact printer, calibration weights and options for all models.

A&D balances are available in New Zealand from Gough Technology.

For further information please circle no. 2 on reader reply card.

THE GREAT WRITTEN DOWN STOCK SALE

Genuine Dollar Savings on top of the line instruments, Accessories, Consumables, suppliers, reagents and spare parts.

Every year, as part of it's company policy, Wilton Instruments is forced to write down the value of items which have been in stock for some time.

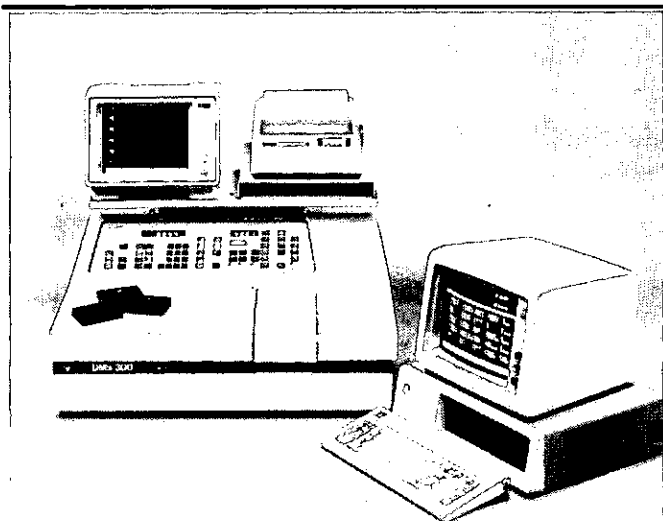
As a result, Wiltons has on offer a whole range of products, many below original cost and all many times below their replacement value.

These have been combined in one comprehensive listing which contains accessories and supplies for instruments which are no longer current models and a number of instruments, at never to be repeated prices.

Examples include: Sartorius Balances, Microscopes from Reichert and American Optical, SGE-Syringes and Supplies for Chromatography, Varian GC and HPLC Accessories and Supplies, Handheld Refractometers from Kyowa Optical, Iatron Supplies for Iatroscan, Electrochemistry Products from Metrohm and many more.

If you would like a copy of the "Great Written Down Stock Sale" and to see these bargains for yourself, please contact: Ian Albers, Wilton Instruments Division, Salmond Smith Biolab Ltd, PO Box 31-044, Lower Hutt, Ph (04) 697-099, Fax: (04) 697-240.

PRODUCT NEWS



Spectrophotometer With Optical Performance Of Research Instruments

Plug-in application modules further improve DMS 300's performance for scanning, kinetics, and quantitative analysis.

The DMS 300, is the newest model in Varian's family of routine UV-Vis spectrophotometers. According to the company the DMS 300, a double beam instrument with dual optical gratings, achieves a level of performance comparable to the finest research instruments.

Providing exceptional price/performance value, the DMS 300 is well suited for application in a wide range of industries says Wilton Instruments, New Zealand agents for Varian.

These include: pharmaceutical, quality control, chemical processing, plastics, petrochemical, biotechnology, medical research, food processing, and fertilizer and farm chemicals.

A number of features contribute to the spectrophotometer's high performance. The DMS 300 is able to make accurate measurements above 5 absorbance, a capability generally found on more expensive research instruments. Its ability to work with highly concentrated, minimum dilution samples, allows the chemist to work with "as is" samples. The instrument can also analyze light scattering samples and measure small absorption differences accurately.

The DMS 300 offers a range of plug-in application modules. These provide an application-specific, soft-key menu from which the chemist establishes data and mathematical routines to be used in calculating, online, final results for scanning, kinetics, and quantitative analysis. Once a user has developed an

application program, it can be recalled with a single keystroke.

The application modules eliminate time consuming manual transfer, entry, and interpretation of data. Built-in scanning functions include spectral subtraction/addition, absorbance ratio, peak area, multi-wavelength, and dilution correction calculations.

Kinetic capabilities include slope and activity calculations for zero and first-order rate reactions for up to 5-cells, slope window calculations on user-defined windows, and the ability to average slopes. This can be performed on short-term (secs), kinetic, or long-term (hours) runs.

The built-in concentration functions include calibration for up to ten standards and five replicates in a choice of photometric modes, including first to sixth derivative, choice of curve fits, weight correction, and identification of out-of-range sample concentrations.

An IBM PC can enhance the DMS 300's application module system by greatly expanding method and spectral data storage. The IBM PC provides unlimited storage capacity of methods, command programs, and data.

In addition, the PC allows application of user-developed spectral manipulation routines and third-party software. Such packages include spread sheet and graphic packages for trend analysis, graphic presentation, and documentation of UV-Vis data.

For laboratories with a larger workload, a batch sampler accessory makes it possible to produce final results for up to 175 samples completely unattended.

For further information please circle no. 1 on reader reply card.

NOTICES

Australian Journal of Chemistry

Professor R.C (Con) Cambie, University of Auckland, who is a member of the Editorial Board of Aust. J. Chem, has requested that the following information be brought to readers attention.

Some recent developments with the Australian Journal of Chemistry are as follows.

Papers In Chemical Technology. Papers in this vital area are now being received. In addition to regular contributions, a special issue devoted to this topic will appear in 1989.

Invited Reviews. Invited review papers by eminent Australian chemists have been instituted, and will appear every few months.

Staff Changes. Dr Robert Leydon left the Journal on July 22, after many years of service. Bob joined the Journal in 1966, and has served as one of our Managing Editors since 1985. He has given devoted service to the Journal, and through it to the Australian and international chemical community. We wish him well in his future career. A new position entitled Production Editor has been created for the Journal, to work under the Managing Editor, Dr John Zdysiewicz. Dr Catherine Greenwood will commence work in this position in August.

Production Delays. Because of a number of managerial changes instituted by CSIRO, Aust J Chem has suffered delays in production over the last few months. This is principally due to the closure of the CSIRO

print shop and temporary manpower shortages. All printing is now being done externally, and our manpower is back up to full strength. The Journal production will be back on schedule by October. Apologies are owed to the Australian and international chemical community as a result of this problem; however, these managerial changes have led to a Journal which will be produced more economically and efficiently in the future. On a positive note, the Journal is now sent overseas by surface/air lift, which means that overseas readers receive it some months earlier than they have previously.

Chemdraw. This structure-drawing package is now being used by the Journal; authors are also encouraged to submit Chemdraw structures (on disk or camera-ready on paper) with their manuscripts. Instructions to Authors will soon contain technical information about this, which will lead to increased efficiency and productivity.

Budget. The budget for Aust J Chem has been approved for the next year. The Journal is a major earner of overseas currency, and although at present operating at a small loss, will turn into a profit-making publication by 1990. This financial position, and the CSIRO/Australian Academy of Science publication agreement, ensures the future well-being of the Journal.

Stranks Issue. The special issue in honour of the late Don Stranks will appear as issue 11, and will also be offered for sale as a separate publication.

Fume Cupboards - New Standards

The standards Association of New Zealand has promulgated NZS-7203 "Safety in Laboratories - Fume Cupboards". This standard is in effect and is, in fact, the New Zealand adaption of the Australian Standard 2243.8-1986.

Specific attention is drawn to the retro-activity clause 1.2 of the standard. In order to comply a three-phase approach should

be determined at the field level:

- any new fume cupboards installed should meet NZS-7203;
- any fume cupboards modified should so be to meet NZS-7203.

-local priorities for other fume cupboards should be set to meet the requirements of NZS-7203.

In respect of fire safety there exists NZS-4208 for fume cupboards and compliance with such remains a requirement as is current practice.

New Agents for Hewlett Packard Equipment

Wellington, 19 September, 1988: - Announced today by Bob Cattell, Country Sales Manager of Hewlett-Packard (NZ) Ltd, was the appointment of Medtec Products Limited as the New Zealand distributor of Hewlett-Packard's medical and analytical products and systems.

These products were previously distributed by Northrop

Instrument & Systems Ltd.

Hewlett-Packard is a worldwide leader in the manufacture of medical products and systems which are used for adult and neo-natal patient monitoring, cardiology and obstetric applications. Hewlett-Packard's analytical products are used by chemists and analysts in the areas of environmental, petrochemical, medical and forensic applications.

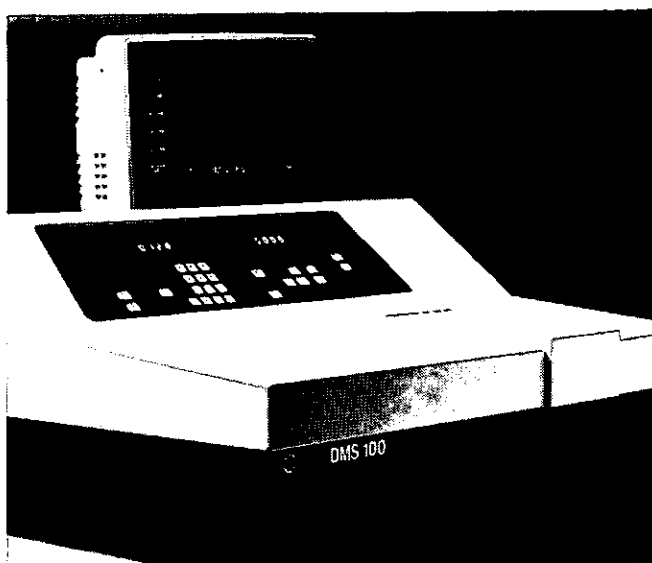
Covering the field in analytical instruments

Varian 3000 series Gas Chromatographs ▶

Models 3300, 3400, 3500, 3600

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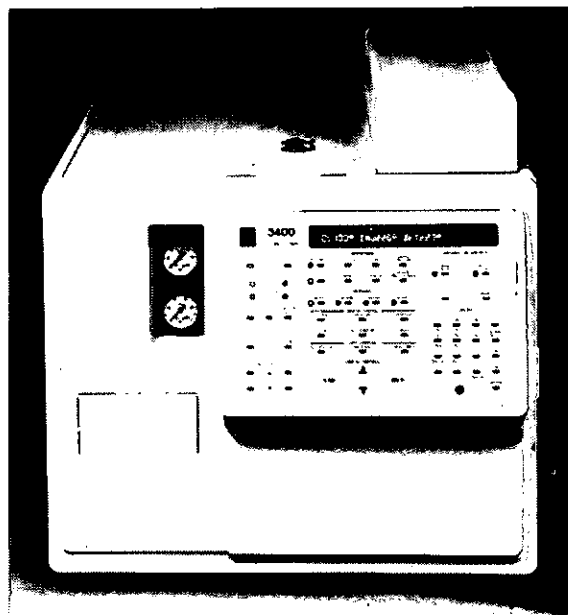
- Standard or automated systems.
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SpectrAA Series Atomic Absorption Spectrophotometers ▶

Models 10/20, 30/40, 300/400

Single and double beam. Choose from a range of flame controls, lamp turrets and background correction, levels of data handling from routine inboard to advanced PC operation, even Zeeman systems. All from the company that pioneered atomic absorption – Varian.

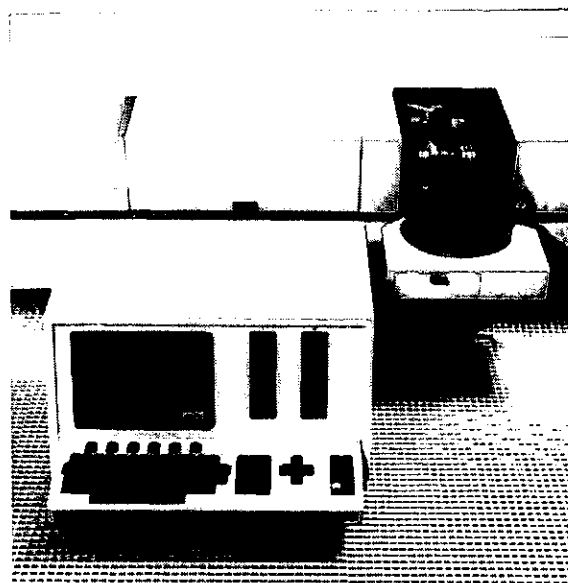


The 3000 Series has a model and configuration to suit you.

◀ DMS Series UV-Visible Spectrophotometers

Models 80, 90, 100, 200, 300

A range of double beam instruments for all applications from routine to research. Buy only the features you need and choose from a wide range of accessories and sample-handling systems. Apply the power of advanced data handling, graphics displays and applications packages.



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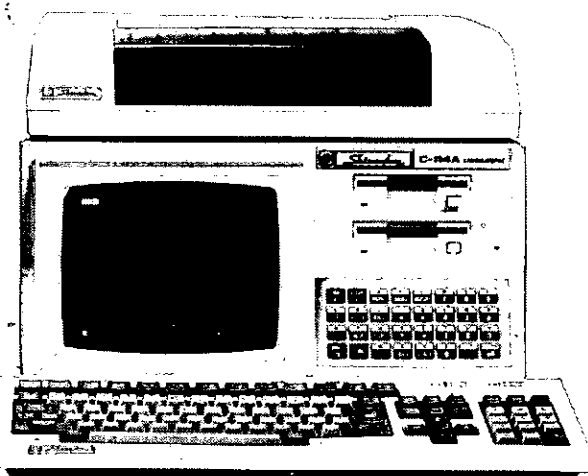
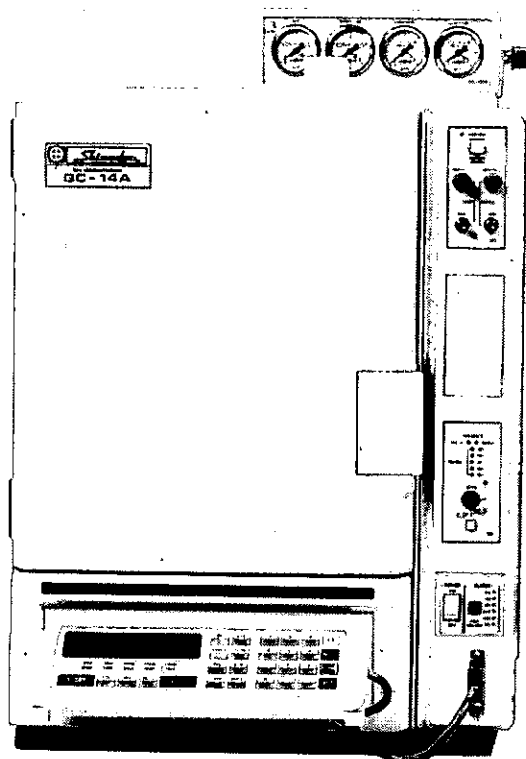
SHIMADZU

GAS CHROMATOGRAPHS

Shimadzu's wide range of GC's progresses from the Model GC8 Series basic quality control and teaching instruments, through the new compact high performance GC14 Series, to the top of the line GC15/16 Series.

Shimadzu's new design ovens provide accuracy, precision and reliability in capillary GC.

Shimadzu Chromatographs are designed not only for independent use but as core instruments for multi-dimensional GC.



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