Chemistry in new zealand

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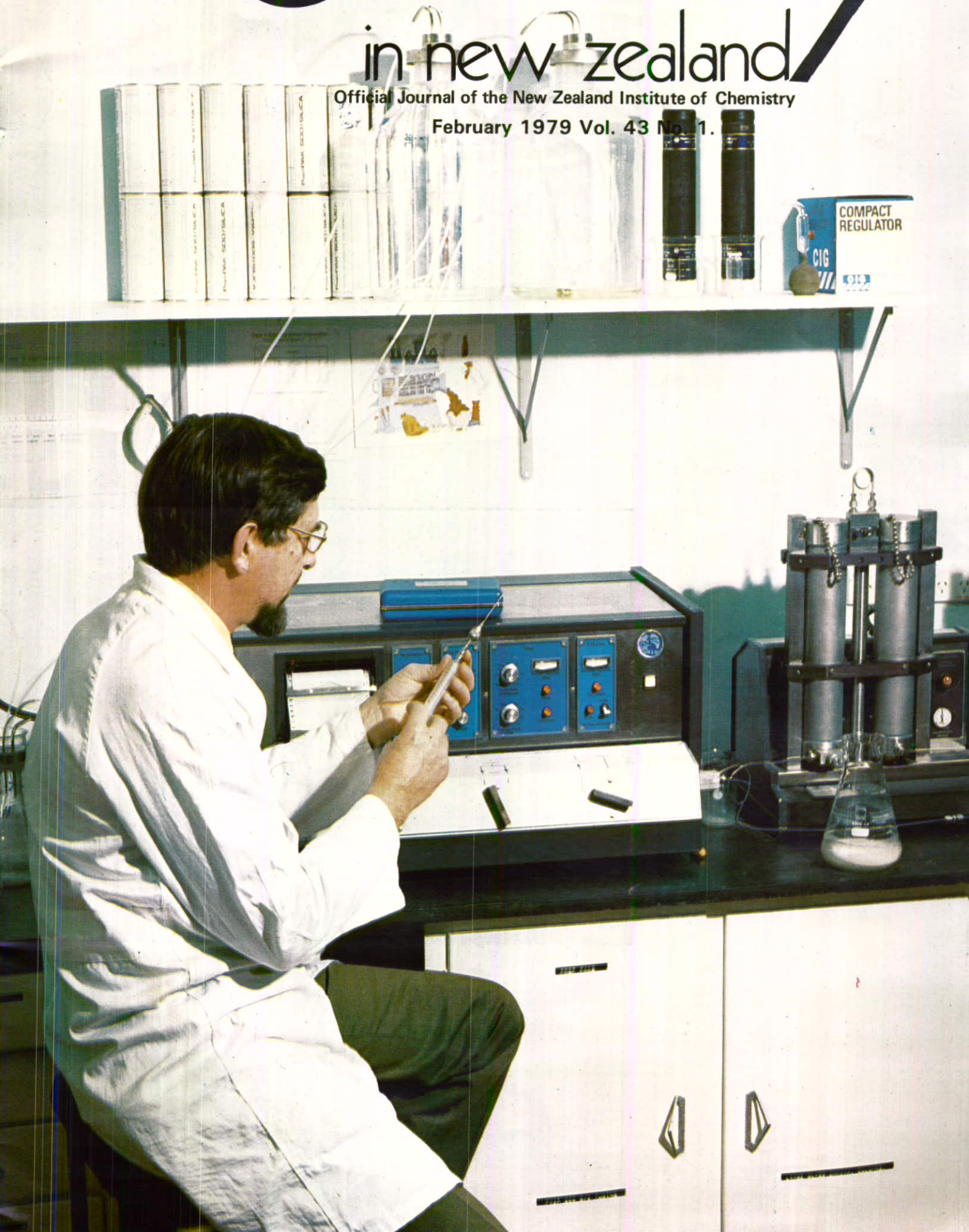

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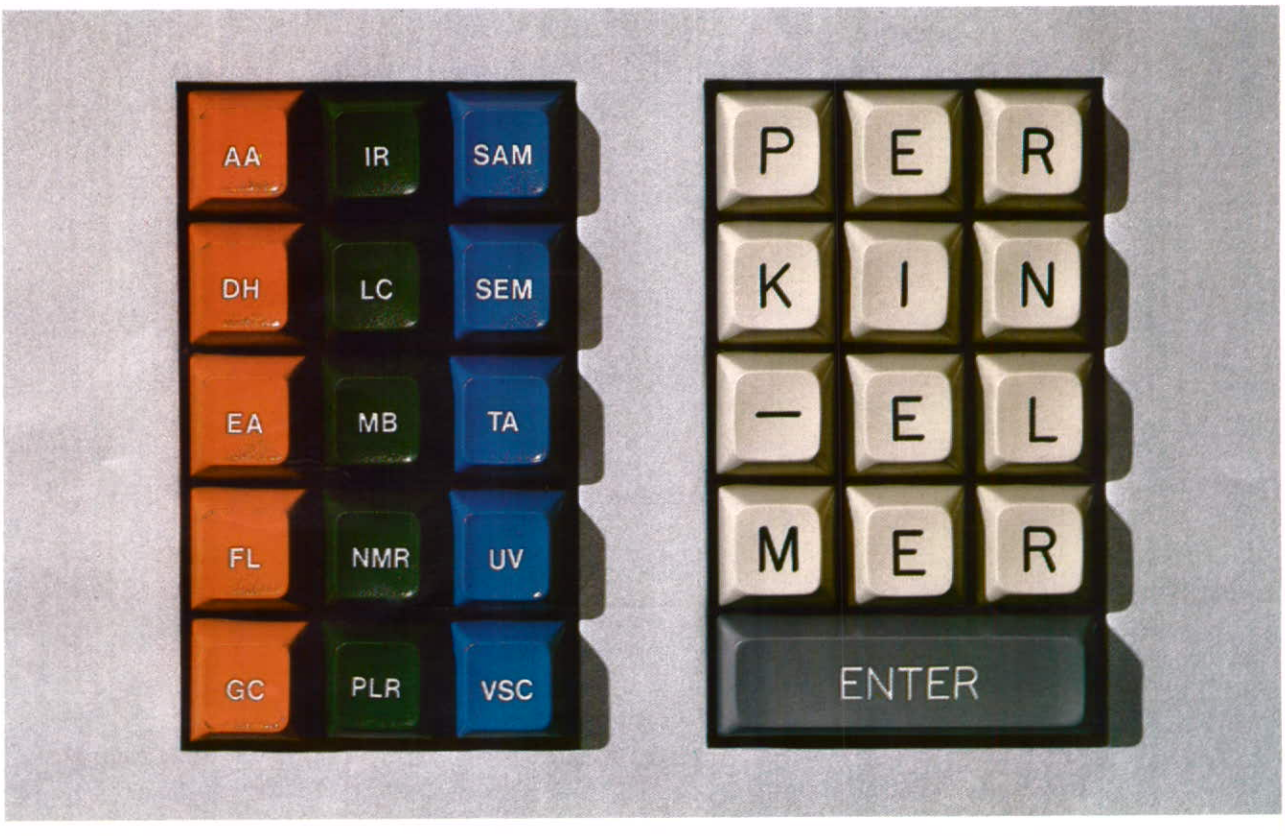

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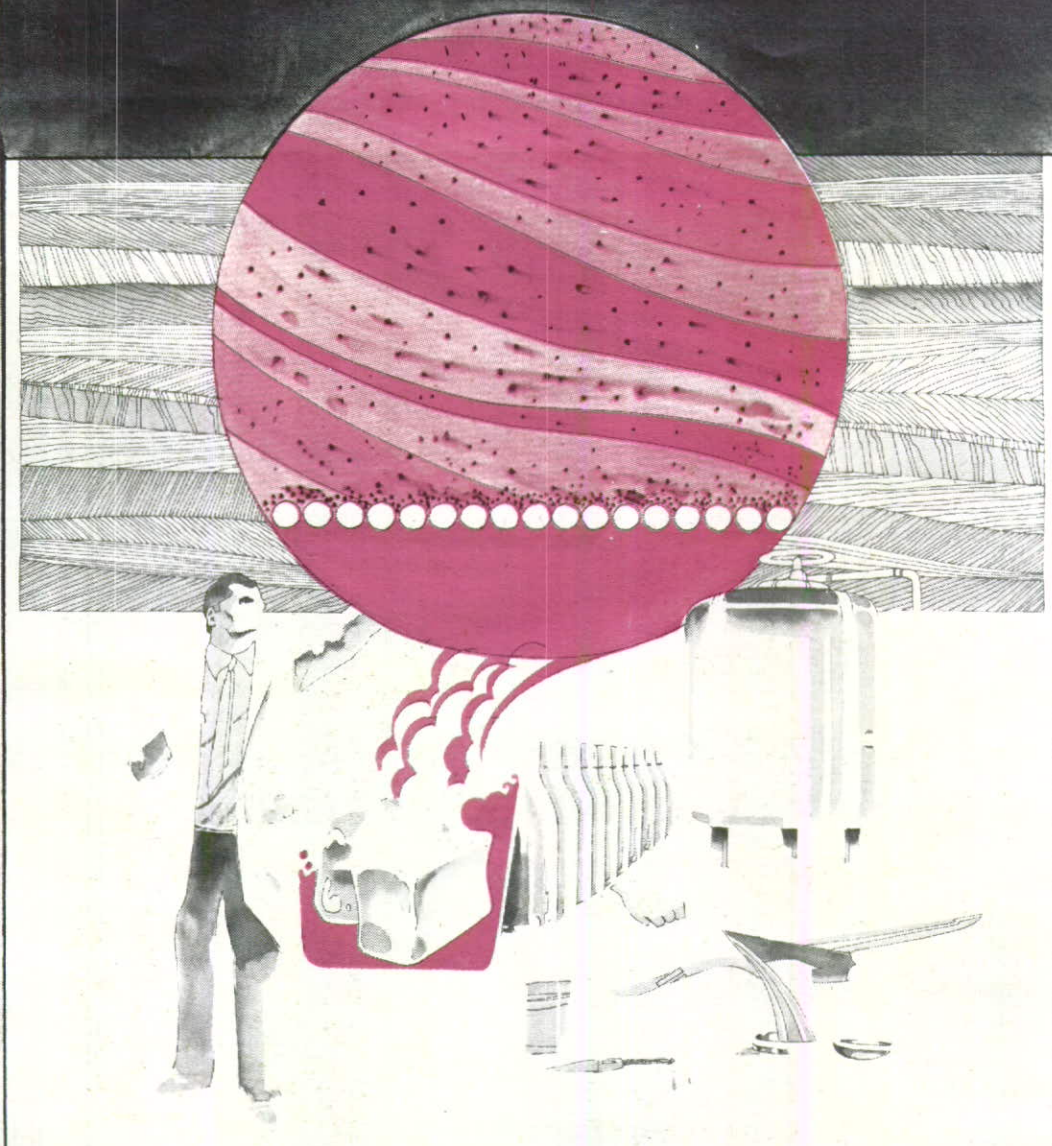

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CONTENTS

Minister's Pledge: "I will Play My Part"	**5**
Comment	**6**
What's Happening	**9**
CHROMATOGRAPHY FEATURE: 12 Papers covering aspects of the use of chromatography	**13-28**
Outstanding Service Culminates In Institute Leadership	**30**
UK Chemists' Plight Causes Concern	**30**
1979 Branch Chairmen — Profiles	**32**
Branch News	**34**
1979 Branch Officers	**36**
The Register	**38**
New Products, Services	**39**
For Your Diary	**40**

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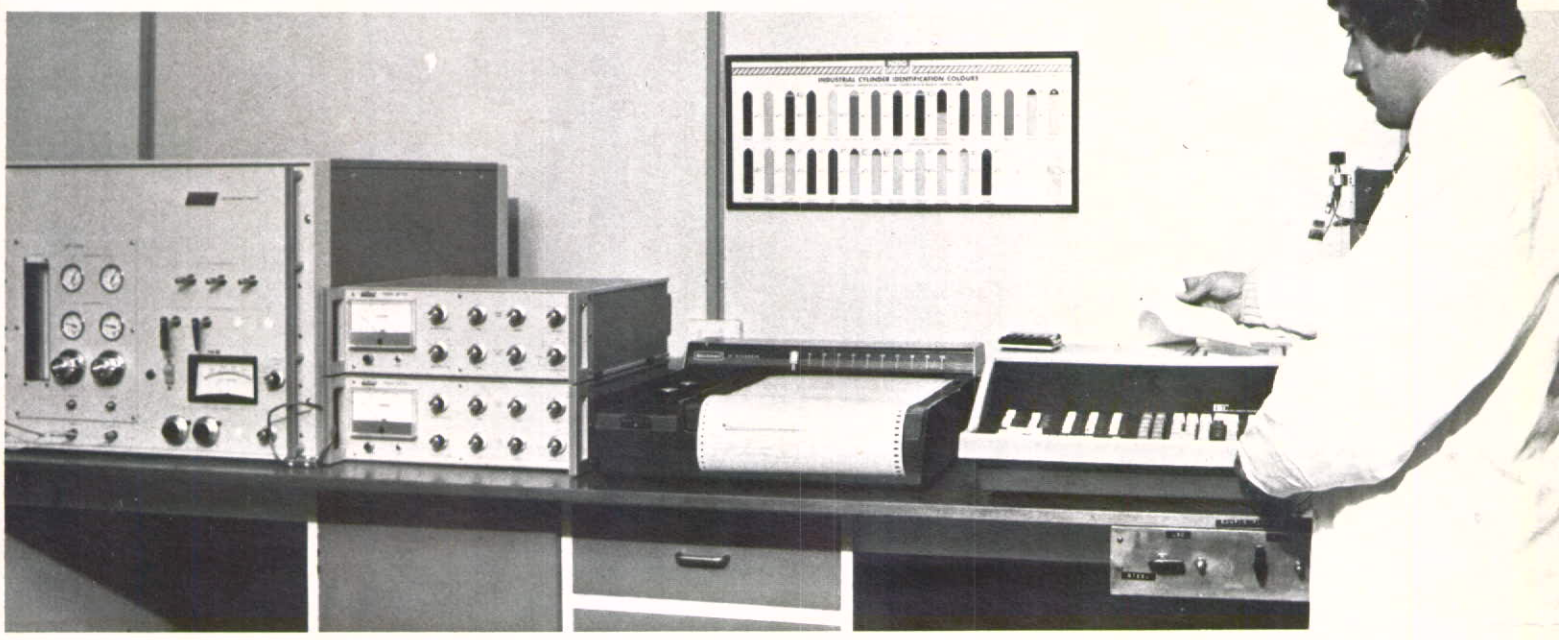

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New Minister's Pledge:

"I Will Play My Part"

Mr W F Birch, Minister of Science and Technology, who wrote this message following his promotion to Cabinet.

It is becoming something of a platitude to repeat that NZ is today faced with major problems in restructuring its economy to overcome the present imbalance in our overseas trade. As Minister of Science and Technology, Minister of Energy and Minister of National Development, I am acutely conscious that in all three areas of responsibility the success of our efforts will depend in large measure on the way in which we are able to bring the skills and experience of our scientists and technologists to bear on our problems.

To build an export industry requires many skills in the fields of management, finance and marketing. It is nevertheless true that in today's world, our success in exporting will increasingly depend on the use of advanced and innovative technology. The best salesman cannot for long successfully market an obsolete product or one produced by outdated and expensive processes. Some of our Asian neighbours have shown us what can be done by a determined policy of building industry on the most modern technology available. In this I am thinking not only of Japan, but of countries such as Singapore and Korea, whose industrial capacity is expanding at a remarkable rate.

The New Zealand Institute of Chemistry constitutes the largest single professional group of scientists in the country. Perhaps, to cater for the well-known ecclesiastical bias of your editor, I should refer to them as the "Chosen People" for this task! I am mindful too, that Doctor K.L. Sutherland, president of the recent ANZAAS Congress in Auckland and a New Zealander by birth, is a chemist who has risen to the top of the science profession and I commend to you the very relevant comments about priorities in science contained in his speech to the Congress.

There can be no doubt about the many, varied, complex and challenging tasks that call for the urgent and dedicated endeavours of your members. Some of these tasks may require the direct importation of overseas technology, but I am very much aware that this policy has, in the past, brought some disastrous failures as well as some outstanding successes. I am therefore convinced of the need for our own scientists and technologists to be intimately involved with such importations, to ensure that the technology is suitable for NZ conditions — and if not, that it is modified to be compatible with our raw materials, markets, working conditions and scale of operations; clearly any needed modification should take place before commissioning, not after costly unsuccessful trials.

In the energy field, the proposed petrochemical industry to be based on Maui gas will rely heavily on chemists and chemical engineers. While the technology required for this kind of industry will inevitably have to be imported, it is my hope that local scientists and technologists will be involved to the maximum extent possible.

The effort to increase our export earnings will continue to rely heavily on the agricultural sector. For many years we have given lip service to the concept of adding value to our agricultural and pastoral exports by additional processing. Our present situation demands that we convert this philosophy into practical reality. Meat, wool, dairy products and timber continue to dominate the scene, and we have already made some moves to increase processing — for example, exports of carpets and carpet yarns, and an increasingly sophisticated range of dairy products.

I expect this trend to continue, but I am also impressed by the potential for the development of new land-based industries. Kiwi fruit exports are the glamour development of recent times. I believe our horticultural industry is capable of greatly increasing the volume, range and quality of products in the field of fruit, nuts, vegetables and herbs. I am also convinced that there is scope for the development of high-value processed products based on agriculture. The newly-established industry producing solasodine from the native plant Solanum aviculare is an excellent example, and I am sure that Kiwi ingenuity and inventiveness, coupled with scientific training and outlook, will bring more such products to commercial reality in the future.

Processing of indigenous minerals and protection of our environment are other important areas which will increasingly call on the services of Institute members.

Many of these projects will call for the dedicated efforts of our chemists, whether in industry, Government or Universities. I wish you well in your work, and assure you that I for my part will do all I can to ensure an environment in which scientific endeavour and originality can flourish.

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Comment

PURE AND APPLIED CHEMISTRY

Unfortunately Andrew Brodie's questionnaire in Bulletin 16, inviting views on the Journal, arrived so late that it deterred 1300 members of the Institute from replying — but the other 48 did! Their replies indicated a strong interest in work done by NZ chemists and in chemical developments in this country. There was a general opinion that short articles were the "in" thing, but also considerable interest was shown in the salary survey and wider social issues, together with a plea for less academic material. Easily the most popular recent scientific article was the paper by Adams on High Pressure Liquid Chromatography in the issue for November 1977: your new Editor attributes the insight which led him to feature chromatography in this issue long before he saw the results of the questionnaire to the ecclesiastical tradition in which he was nurtured.

The replies also indicated a need for more attention to applied chemistry, which we think means chemistry in industry and in the marketplace (though cognoscenti in the Royal Society of NZ may think differently). The Institute's Council, with foresight rivalling our own, has appointed an editor with an industrial background; he slipped into industry on margarine in 1936 and is still in it. One of the great fields of industrial chemistry was opened with the discovery of the effect of vulcanising of rubber by Goodyear, which made possible its use in tyres. A more recent technology has given us the process of retreading of tyres and in NZ about two tyres are retreaded for every three new ones produced.

The Council's wisdom and leaning towards applied chemistry has extended to retreading an editor, who last held the post in 1953. Most people in the rubber industry would consider this a fairly old tyre to be retreaded, but the questionnaire showed members interested in social questions, which could extend to archaeology. This gives us courage to refer back to two things we said in our first editorial in 1949: we compared ourselves then to Columbus, who didn't know where he was going, but we now have Peter Reaves to provide a compass. We also pleaded for reactions of any order from our readers, but both the halo and the bulletproof vest gathered dust. We hope for more reaction in 1979.

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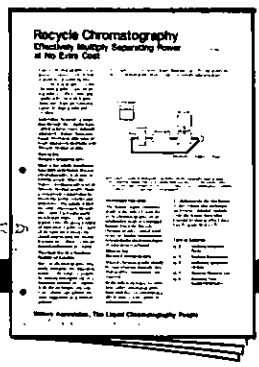
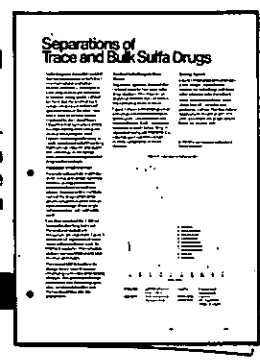


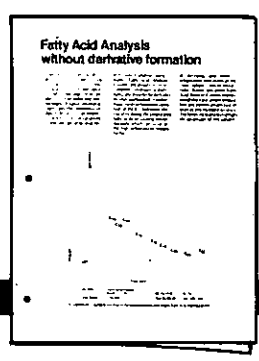
Recycle Chromatography Effectively multiply Separating Power at no extra cost

10 pgs. Describes the theory and operation of Recycle Chromatography. Examples include verification of Compound Purity, confirmation of Compound Identity and data on reduced L.C. operating costs. *Ask for N 70.*

Separations of Trace and Bulk Sulfa Drugs

Details method for the L.C. separation of nine Sulphur drugs in seventeen minutes. Reverse phase technique with detection limits less than 5 micrograms. *Ask for H 10.*





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4 pgs. Shows typical separations achieved on new Fatty Acid Analysis column. Separates underivatized free Fatty Acids, Fatty Alcohols, Amides and the C18 Isomers from a wide range of samples. *Ask for D 62.*

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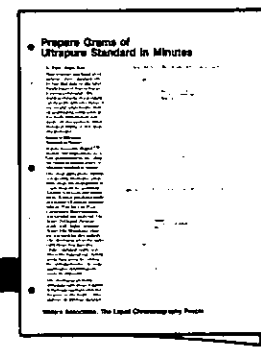

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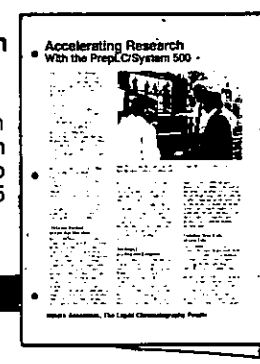

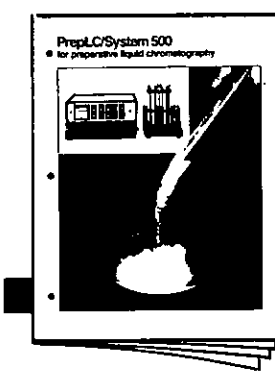

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What's Happening

The President, Dr W.E. Harvey, will be attending the combined American Chemical Society-Japan Chemical Society meeting in Honolulu, April 1-9. Dr Harvey has a copy of the programme, which runs to 70 pages, and he is prepared to supply a photocopy of appropriate sections to anyone interested who writes to him at the Victoria University of Wellington.

A recent visitor to NZ was Dr M. Cresswell of Wellcome Reagents, UK. Dr Cresswell was invited here to address a course on "Standards of Laboratory Performance", in Christchurch. He designed and developed the Wellcome Quality Control Scheme which is used extensively in this country as a check on the precision of pathology laboratories. As part of his lecture tour Dr Cresswell is visiting Australia and USA. During his stay in Auckland Dr Cresswell visited L. Eyres who used to work with him in UK.

The Secretary of the NZIC has been advised of the arrival, in NZ last month, of Dr Frans Robert Visser, who is looking for a position related to the food industry. He is 38, married with two children and has had considerable experience with food flavours, starches, gums and other food hydrocolloids. Enquiries should be directed to the Dutch immigration office in Wellington.

Dr R.W. Harman, former general manager and director, Colonial Sugar Refining Co., died recently in Australia. He was educated at Hamilton High School, and Auckland University College, where he gained a 1851 Exhibition which took him to University College, London. Here he gained a D.Sc. in 1925. He joined CSR in 1936 to head the newly formed research department, and rose to become general manager in 1951. He was President, RACI in 1942. On retiring in 1957, he came to live in NZ, but later returned to Australia, where he died.

The Editor is preparing an index to the Journal running through from Vols 1 (1937) to 42 (1978). He would like to know who are interested in having copies, or in assisting in the project.

The recent ANZAAS Congress in Auckland attracted 4200 participants. Of these, 1200 were Australians, while a further 100 came from other countries. In our next issue, and possibly subsequently, we shall be reviewing or publishing some of the more interesting papers presented.

'79 Conference Will Explore Industry Prospects, Perspectives

NZIC 1979 conference theme will be "The NZ Chemical Industry: Prospects and Perspectives". Venue is Victoria University of Wellington, August 20-23.

Plenary lectures will reflect this theme in the general areas of carbonaceous resources [both fossil and renewable], inorganic and organic based industries [including health-based industries] and prospects for biochemical technology.

In addition, there will be the traditional specialist sessions incorporating student papers, current research papers, review lectures and an opportunity for business and social meetings of specialist groups. As usual, the conference will form a joint meeting with the NZ Biochemical Society.

A one-day symposium for Friday, August 24, on Forensic Science is being organised in conjunction with the main conference.

Social functions will include an informal get-together, and the conference dinner.

There will be the customary trade and book displays.

Registration forms and details regarding plenary lectures, specialist sessions, submission of papers, and accommodation available will be published in the next issue of the Journal.

In the meantime, any enquiries should be sent to the Conference Secretary:
J.T. Craig,
Chemistry Department,
Victoria University,
PB, Wellington.

(Our photos show Prof. Neil Curtis, Conference Committee chairman (left), and Dr John Craig, secretary, draped around sculpture in the foyer of the Cotton Building, Victoria University, where the 1979 conference will be held.)

"Flammable" or "inflammable"? The Professional Bulletin of the RIC has received advice from the Health and Safety Committee which, after consulting with other bodies, has brought down a ruling in favour of "flammable" — "Unhesitatingly and unanimously" according to the letter received by the RIC. This brings British practice in line with American, although it is pointed out that there are many legal and other documents still in force with the old term.

Implementing a remit to the 1978 AGM recommending that the NZIC establish a specialist group on Industrial Chemistry and investigate means of increasing the numbers of members from industry, an inaugural meeting was held in Auckland late November.

Organised by Auckland branch secretary Mr Milton Gibson, the meeting attracted 28 members and following an address by the General Secretary, several ideas were mooted; lunchtime meetings were favoured with topics of general interest to industry.

A steering committee was formed. It comprises: Chairman, Mr J. Yolland (Materials & Testing Laboratory. Ph. 545-717); committee, Messrs R. Macleod (Mair Marketing. ph. 771-657); C.W. Harland (Farmers Fertilizer Co. ph. 543-189) and Dr L. Eyres (Abels Ltd. ph. 548-145).

Tentative programme ideas suggested for 1979 are:-

February — Lunchtime meeting
April — Works visit
June — Evening symposia
July — Lunchtime meeting.

The NZ Fertiliser Manufacturers' Association, representing all the domestic fertiliser producers, has expressed concern at recent news media releases implying that NZ may soon have difficulty in gaining access to suitable supplies of phosphate rock for the manufacture of superphosphate or that great price increases are to be expected.

These releases emanate largely from ill-informed academic sources and accompany suggestions relating to the use of dubious or unproved alternatives, says the NZFMA, which regards these statements as completely erroneous and irresponsible in that they can cause quite unnecessary concern to farmers who are so dependant on the product.

In reality, it comments, phosphates are widely spread in nature and are one of the

few major minerals where supplies are secure for centuries. There are enormous resources in USA, USSR, Africa, Australia and other countries and new deposits continue to be found and will be developed as the need arises. Many of these are available to NZ as normal commodities of world trade and there is absolutely no need for a search for questionable substitutes.

Recently, for instance, the Association arranged the supply of over 50,000 tonnes from other than the traditional sources.

The death has occurred of Brian Ogilvie Jones, chemist, Packaging Research Department, NZ Forest Products Ltd. He was 54.

He was an active member of the NZIC and of the NZ section, Australian Pulp and Paper Industry Technical Association (APPITA) for many years. Apart from a break between 1958-60, he spent 25 years with NZFP in a variety of positions. He also worked for Wilsons (NZ) Portland Cement, Waitomo Portland Cement and Caxton Paper Mills Ltd.

He is survived by his widow, a son and two daughters.

The end-of-year function of the Polymer Group was held late November in Auckland when Mr Keith Boyer addressed it on "Adhesives". He summarised the different categories of adhesives and illustrated some different chemical types. A discussion on the design of the adhesive joint and surface wetting followed. The address concluded with a consideration of testing and the various standards used. A lively question time followed with cyano-acrylate adhesives and tack measurement prominent.

An informal election of office bearers followed. Mr Arthur Kennett, the driving force in the group's formation in June 1974 and inaugural chairman, stepped down. Mr Neil Edmonds was elected chairman and Mr Michael Cochrane secretary, both unopposed.

A discussion on activities for 1979 followed. The chairman undertook to attempt to arrange a repeat of the "Aspects of Polymer Science" course successfully run in 1978 with 55 participants completing it.

Dr Lawrie Creamer, Dairy Research Institute, Palmerston North (above) who filled the position of Journal Editor with distinction during 1977-78, has now relinquished this responsibility.

He will be remembered for his numerous innovations (not least the crossword puzzle), but less obviously to many of our readers the almost superhuman amount of work put in. It is not surprising that the Council has turned to a different system which has put a less onerous burden on an honorary editor.

Well done, Lawrie, and thank you from all in the Institute!

The NZ Association of Clinical Biochemists held a 2-day workshop on "Standards of Laboratory Performance" in Christchurch in November.

The organisers clearly intended that discussion concentrate on the reality of running busy medical laboratories generating data which would give to the user (the clinician) what he needed to know to enable him to properly manage the care of his patients and producing this data reliably with well defined uncertainties. A fairly optimistic programme certainly, but one that revealed a very healthy attitude among the clinical biochemists, medical technologists and pathologists who attended.

The programme included speakers who discussed clinical requirements, analytical limitations, quality control procedures and interlaboratory trials, laboratory accreditation, centralised laboratories for sophisticated assays and education for laboratory staff. Each presentation was followed by lengthy and vigorous discussion.

Each presentation and discussion is worthy of separate reports but until such time as the proceedings are available this is not possible. My personal response to this programme is to commend the NZACB for its intiative and concern in this area which can truly be described as laboratory management and perhaps suggest to chemists in other industries that attention to some of these areas of management would be most fruitful.
J.A. GILMOUR

NOVEMBER COUNCIL BRIEFS

New System For Collecting Subscriptions: As from May 1, 1979, all subscriptions, including branch subscriptions, will be collected by the Registrar, and members will receive computer printed accounts soon after that date. Subscriptions will be increased to $25 (including $3 for the local branch) reducible to $23 if paid by August 31, 1979.

Technicians Certification Authority: Dr Harvey reported that the Authority was seeking a new Act under which the name would be changed to one more in keeping with its broader responsibilities — "The NZ Authority for Advanced Vocational Awards".

New Appointments:
Membership Committee — Dr J. Rogers.
Publications Committee — Mr S. Gray.
Energy Committee — Dr A. Metcalfe.
UNESCO — Dr G. Burns.
Royal Society Member Bodies Committee — Dr I.D. Watson.

Institute Golden Jubilee, 1981: A report was received from the second vice-president, Dr Ellis, on suitable ways of celebrating this event. It was decided to approach the Post Office about a commemorative issue of stamps. It was also agreed that the venue of conferences be altered so the 1981 conference will be held in Auckland and the 1980 gathering at Palmerston North.

Industrial Chemistry Group: It was reported that a meeting was being held in Auckland as reported elsewhere.

New Legislation: It was noted that Council had taken out a subscription to Common Concern, Wellington, a body which keeps a watching brief on legislation before Parliament. Matters concerning chemists include Liquid Fuels Trust, Restricted Drugs, Pesticides and Toxic Substances, (the Massage Parlours Bill could be of peripheral interest), which came up during the 1978 Session.

Committee on Materials and Energy: Copies of an official news release were available at the Council meeting. Members wanting further information should write to Prof. A.G. Williamson, Dept. of Chemical Engineering, Canterbury University.

New Men At The Helm

The new team running "Chemistry in New Zealand". From left Stan Brooker, Institute Editor, Dr Bill Denny, Associate Editor, Dr Lawrence Eyres, Publications Committee, NZIC, and Peter Reaves, TRICOM. Note that the new editor is the only one that thinks he can do the job without taking his coat off! Dr Tony Herd, an ex-secretary of the Auckland Branch, has since joined the committee, and a further addition will be a person with a special interest in biochemistry.

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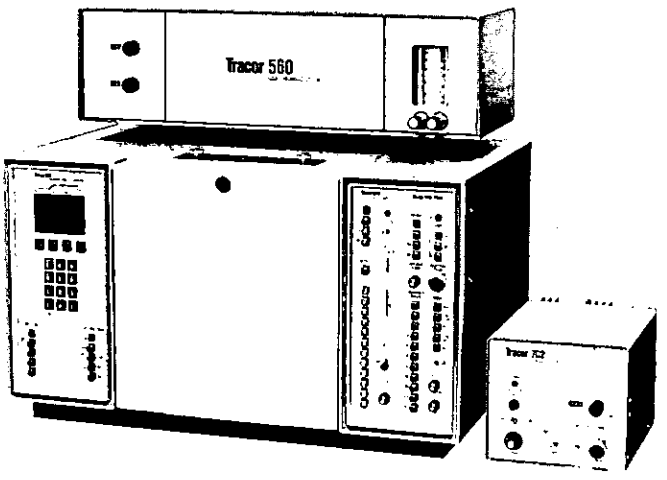
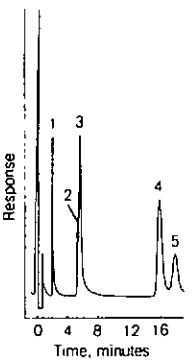


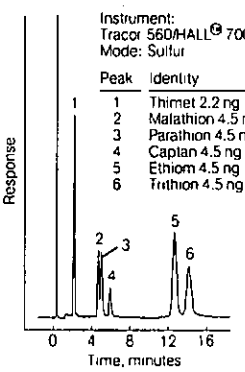
702 NPD
Phosphorous



Instrument: Tracor 560/702 NPD

Peak	Identity
1	Thimet—1.8 ng. ea.
2	Malathion—3.6 ng. ea.
3	Parathion—3.6 ng. ea.
4	Ethion—3.6 ng. ea.
5	Trithion—3.6 ng. ea.

700A HECD
Specific Sulfur Mode

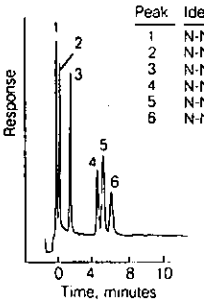


Instrument:
Tracor 560/HALL® 700A
Mode: Sulfur

Peak	Identity
1	Thimet 2.2 ng
2	Malathion 4.5 ng
3	Parathion 4.5 ng
4	Captan 4.5 ng
5	Ethion 4.5 ng
6	Trithion 4.5 ng

The 700A detector provides improved linearity and selectivity for sulfur over the FPD.

Specific Nitrogen Mode
Nitrosamines



Instrument:
Tracor 560/HALL® 700A
Mode: Nitrogen

Peak	Identity
1	N-Nitrosodimethylamine
2	N-Nitrosodiethylamine
3	N-Nitrosodipropylamine
4	N-Nitrosodibutylamine
5	N-Nitrosopiperidine
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CAPILLARY GAS-LIQUID CHROMATOGRAPHY AND GAS-CHROMATOGRAPHY — MASS SPECTROMETRY IN NATURAL PRODUCT CHEMISTRY; SEPARATION OF PLANT WAX SECONDARY DIOLS.

R.A. Franich, Forest Research Institute. Rotorua.

In the field of natural product chemistry, the separation of components of plant epicuticular waxes presents a challenge to the chromatographer. While the separation of members of a homologous series of compounds commonly found in epicuticular waxes (e.g., fatty acids, hydrocarbons, alkanols) can be readily achieved using conventional packed-column gas-liquid chromatography (GLC), difficulty is encountered when separation of compounds having different positions of unsaturation or substitution within each homologue is desired. The simplest solution to this problem is to attempt high resolution chromatography. Wall-coated open tubular (WCOT) glass columns offer the higher resolution in GLC, and these have been employed in epicuticular wax composition studies.

The epicuticular wax of *Pinus radiata* needles contains a complex mixture of alkyl esters, estolides, fatty and diterpene acids, secondary alcohols, and secondary diols (1). The last-named class of compounds are little known as plant was components, and therefore warranted critical examination.

Packed column temperature-programmed GLC (1.5m x 2mm, SE 30) showed that the diol fraction, analysed as the bis-trimethylsilyl derivative, apparently contained three poorly resolved C_{29} compounds, a small amount of C_{27} and traces of C_{25} and C_{31}. However, when the same sample was analysed on a 20m x 0.2mm OV-1 WCOT column, six C_{29} compounds were resolved, though not all with baseline resolution. The resolution obtained using WCOT capillary column is partly depentent on the manner in which the sample in injected. In this case the sample, in dichlorolethane, was injected into a Grob-type splitless injector while maintaining the column oven at ambient temperature. As a result, the sample becomes re-concentrated on the column and, as soon as the solvent peak has emerged, the temperature programme is initiated. The C_{29} compounds were eluted at an oven temperature of ca. 265o (ca. 31 min after injection), and the smallest separation time between adjacent peaks was just under 10sec.

Investigation of the diol composition using combined capillary column GC-MS (computer-controlled quadrupole filter instrument with data acquisition to allow repetitive, fast scanning) in which the capillary column was coupled directly to the ion-source of the mass spectrometer, gave mass spectra and, therefore, structural information for all six C_{29} diols. Examples of the mass spectra obtained for the smallest peak No 7, Fig. 1), and the largest (No. 8, Fig. 1) are shown in Figs. 2 and 3 for comparison.

Use of WCOT capillary column GLC therefore enabled separation of and, when combined with GC-MS, structural information on components within this homologous series of long-chain diols (2).

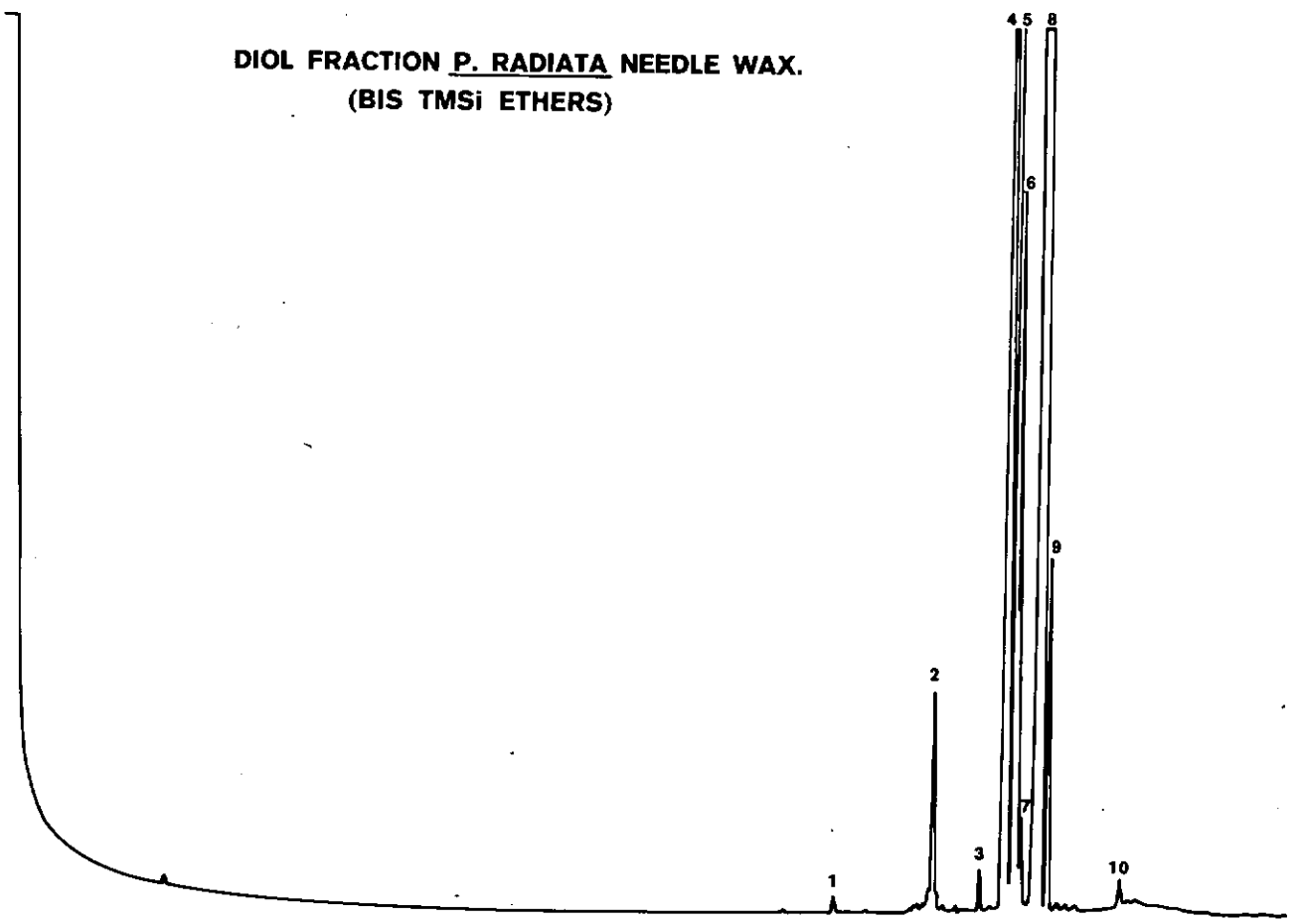

Fig 1 Gas liquid chromatogram of P. radiata secondary diol bis-TMSi derivatives. The peaks numbered 4 to 9 are those of the C_{29} diols, viz : 4=10,13; 5= 6= 7,10; 7= 6,10; 8= 5,10; 9= 4,10.
Mass injected c. 50ng.
Temp. program. 150-270° at 3.5o min $^{-1}$. Attenuation 16x1.

Capillary GLC (Cont)

ACKNOWLEDGEMENTS

The work described here was carried out at the Organic Geochemistry Unit, University of Bristol, Bristol, UK. The author thanks Prof. Eglinton, FRS, University of Bristol, for use of the OGU quadrupole filter GC-MS-DS, and the Royal Society for a Commonwealth Foundation Bursary.

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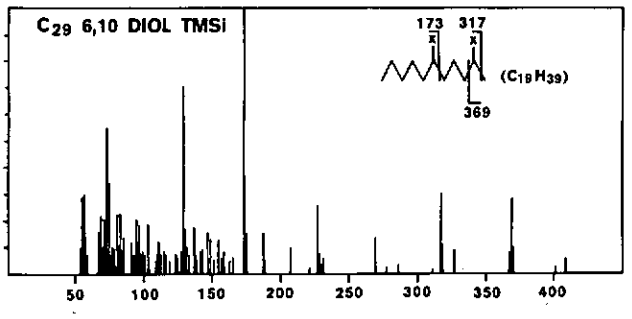

Fig 2 Mass spectrum of C_{29} 6,10 - diol bis TMSi ether.

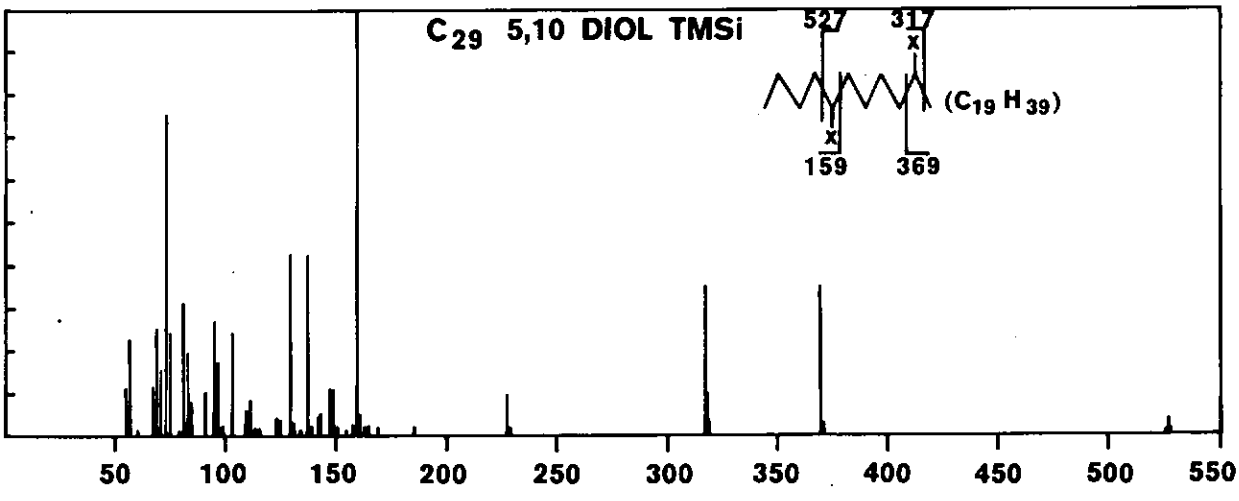

Fig 3 Mass spectrum of C_{29} 5, 10 - diol bis TMSi ether.

HYDROPHOBIC LIGANDS FOR PROTEIN PURIFICATION.

Alistair G.C. Renwick, Department of Biochemistry, University of Auckland.

The deliberate use of hydrophobic ligands for chromatographic separations is a recent innovation and the technique has been described as "hydrophobic interaction chromatography" In essence, mixtures are separated by virtue of the strengths of their hydrophobic interactions with hydrophobic groups attached to an uncharged matrix.

Hydrophobic ligands have so far been mainly used for protein purification. Er-el et al. (1972) were among the first to explore this field when they synthesised a homologous series of hydrocarbon coated agaroses that differed in the length of their alkyl side-chains. A 100-fold purification of glycogen phosphorylase b was obtained in one step on a small column of butyl Sepharose.

In studies of the affinity chromatography of steroid-transforming enzymes, Renwick et al. (1979) found that p-(phenoxypropoxy) aniline covalently-linked to Sepharose 4B was effective in adsorbing an estradiol-17β dehydrogenase from a partially purified extract of avian liver, and that a cortisone reductase was preferentially retained when mixtures of estradiol-17β dehydrogenase, cortisone reductase and a 3(17)β-hydroxysteroid dehydrogenase were applied to small columns bearing the ligand. Lactate dehydrogenase from rabbit muscle was also adsorbed in a manner similar to that found on affinity chromatography with 2',5'-ADP-Sepharose.

p-(Phenoxypropoxy) aniline offers promise for the purification of steroid-transforming enzymes where elution with substrate of expensive cofactor is not wanted and the ligand may be of service in the purification of receptors for hormonal steroids.

Because hydrophobic interactions between ligand and solute are affected by several factors that include pH, temperature and ionic strength of solvent systems, this form of chromatography allows considerable flexibility in design and execution of protein separations.

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THIN LAYER CHROMATOGRAPHY AS A TOOL FOR SCREENING FOR ABUSED DRUGS IN URINE.

R.A. Richardson, School of Medicine, University of Auckland.

Screening urine samples in an analytical toxicology laboratory for the presence of drugs is required for two types of patient - those who abuse drugs for "kicks" and those who deliberately take overdose quantities of drugs.

Thin layer chromatography has been the major tool for rapid screening of these patients. Because the interpretation of the thin layer chromatograms can be difficult particularly in the drug overdosage area a manual has been prepared to assist technologists in reporting results especially in urgent after-hours work. Samples were obtained from overdosed animals and human patients and the results of their screens put together in usable form. The methodology used to prepare these chromatograms and the final drug and metabolite patterns will be discussed. These procedures illustrate the versatility of thin layer chromatography as a valuable tool in this field.

AN AUTOMATED ANALYSER FOR THE DETERMINATION OF PARAFFINS, NAPTHENES AND AROMATICS IN GASOLINE FRACTIONS WITH 200°C FINAL BOILING POINT

I.R. Clark, N.Z. Refining Co. Ltd., Marsden Point, Whangarei.

Introduction

The difficult separation of paraffins from napthenes plagues analysis of hydrocarbons in the naphtha boiling range. However, gas chromatographic separation in a combination of 3 columns permits paraffin/naphthene/aromatic (PNA) compositions to be determined with surprising rapidity.

An automatic analyser was devised by H. Boer and P. van Arkel of Shell Laboratories, Amsterdam, to provide rapid and quantitative analyses of straight run as well as processed streams in the boiling range of 0-200°C. The analyser gives the percentage of P,N, and A per carbon number within 2 hours of unattended operation. This analyser is now manufactured and marketed by Packard Becker Ltd., of Delft, as the Becker PNA Analyser Model 411.

Principle of Operation

Following injection of a naphtha sample the instrument performs a sequence of separations on a polar, a non-polar and molecular-sieve 13Xcolumn using flow switching and hold-up of fractions of the sample. Both the polar and non-polar columns are kept in the oven at 160°C., while the 13X column is mounted on the cover of the main oven.

The temmperature of this column is kept at 70°C. or programmed up to 450°C. The polar and non-polar columns are connected by a component trap also mounted on the main oven cover. The temperature of the trap is maintained at -80°C.(using dry ice). The temperature of the trap is raised quickly to some 250°C. for re-injection of the trapped components.

In order to obtain accurate quantification of all component types, it is imperative to carry out all separations on a single sample, in a closed system and to measure the components with a single detector. This principle is adhered to in the analyser. The column specifications are given in Table 1.

Reasons for Procedure

The instrument can be looked upon as consisting of 2 sections: the PN from A and the P from N sections. The PN from A separation by gas chromatography is a problem that is usually underestimated. With ultra-polar liquid phases the carbon-number selectivity may amount to 6 or 7, which means the benzene elutes closely after n-Tridecane. However, practical samples such as naphthas contain naphthenes and binaphthenes. These behave differently from paraffins in that their carbon number selectivity is generally 3 or 4 units lower. This seriously limits the permissible boiling range of the naphtha sample for a single stage PN from A separation on a polar column.

Even using OV-275 liquid phase, naphtha samples with a FBP of 200°C require a 2-stage fractionation procedure in which an additional non-polar column is used to limit the boiling range of the sample that is injected on to the polar column. This non-polar column can also then be used to quantitate the aromatics by carbon number.

Although the technique and the background of the P from N separation on 13X molecular sieves is not completely understood, proper selection of operating conditions as applied in this analyser warrants a fair separation in a remarkably short time and yields excellent quantitative results. This partly stems from the extremely pure hydrogen carrier gas used (palladium diffused). Helium could be used, but the requirements on purification may be less easily met than with hydrogen.

The Instrument

The analyser features a number of interesting provisions that are not commonly encountered in gas chromatography.

The in-line trap is imperative in order to maintain good peak resolution throughout the PN from A separation procedure, by providing sharp re-injections on both the non-polar and polar columns. Since benzene is the lowest boiling component to be trapped, cooling with dry ice is adequate. The great advantage of dry ice is that the coolant need not be removed while the trap is being heated. The trap is heated to about 250 - 300°C. in 1min by passing a heavy current through the walls of the trap.

The 13X column is also heated by passing a heavy current through the walls of the column. Proportional control takes care of maintaining the column at 70°C. and the rapid heating to the initial temperature (225°C.) of the temperature programme as well as accurately following the temperature programme up to its final temperature of 450°C.

The analyser functions entirely automatically. Automation is accomplished by a real-time digital programmer which performs all the functions that are necessary for unattended operation. The programme is easily set by means of contact pins in the timing matrix. The valve switching is accomplished by using pneumatic actuators which are triggered by the digital programmer.

The chromatogram has to be interpreted as a collection of chromatographic bands rather than as separate peaks. The individual bands, particularly those from the 13X column, tend to vary considerably in shape and area. For this reason an analogue integrator is used instead of a digital integrator. The electronic analogue integrator fitted to this instrument provides a continuous read-out on a dual pen recorder. Automatic scale expansion provides ample sensitivity for small peaks and allows the instrument to operate unattended.

Conclusion

The analyser operates under constant conditions throughout the analytical cycle, and uses a single FI detector. Therefore, taking into account the rigid automation of the method, it is no surprise that the results obtained are very repeatable as well as reproducible. Although the analyser is normally a dedicated gas chromatograph, it is possible to use it for other analyses. For instance, both the polar and non-polar columns can be used independently for any analysis that can be carried out isothermally.

TABLE 1 - COLUMN SPECIFICATIONS

COLUMN	DIMENSIONS	LIQUID PHASE	SUPPORT	MESH SIZE
Polar	3m x 4mm I.D. Stainless steel	OV-275	Chrom-P	60 - 80
Non-Polar	4m x 4mm I.D. Stainless steel	SE-30	Chrom-P	80 - 100
13X	0.6m x 2.5mm I.D. Stainless steel	13X Mole. Sieves		60 - 80

SPORIDESMIN METABOLITE SEPARATION AND PURIFICATION BY HPLC.

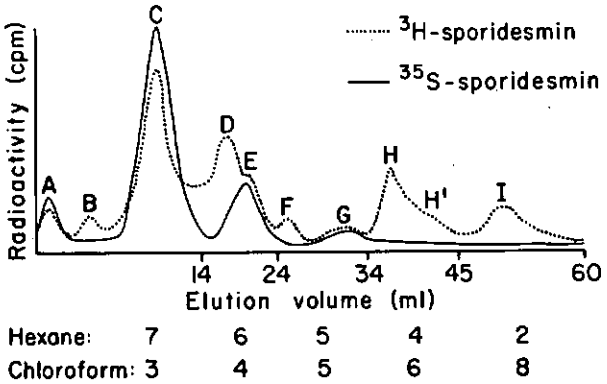
D.R. Lauren, Ruakura Agricultural Research Centre, Hamilton.

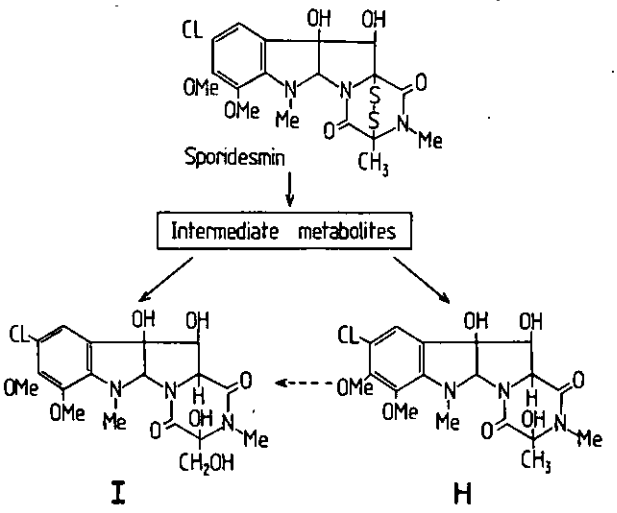
Sporidesmin is the fungal toxin responsible for facial eczema, a severe threat to livestock in some areas. Some animals are more susceptible than others, and differences in the rate of sporidesmin metabolism by the liver seems to be one factor determining resistance or susceptability in sheep.

To establish the pathway for sporidesmin metabolism, ^{3}H and ^{35}S labelled sporidesmin are incubated with liver microsomes in in vitro tests [1], and the product mixture fractionated by various techniques. Fig 1 shows the Lipidex 5000 fractionation of the ethyl acetate extractable metabolites. Tentative structures of 2 metabolites are shown in Fig 2.

Some fractions from the Lipidex column (D,E,F and H^1) were shown to be mixtures by t.l.c. These were examined by HPLC to determine their complexity, and then pure samples of the major components were separated micro-preparatively on the analytical column. A cyano bonded phase column was used. The four fractions, well separated on Lipidex, gave components with a range of overlapping R_f's on the cyano column.

Fraction D (five components, one major) was purified using 85:13.5:1.5 hexane/$CHCl_3$/MeOH at 1 ml min^{-1}. Compound D had an R_f of 18 min.





Fraction E (nine components, two major including compound D) was also separated preparatively using 85:13.5:1.5 hexane/$CHCl_3$/MeOH at 1ml min^{-1}. Compound E had an R_f of 32 min.

Fractions F (two major components) and H^1 (one main peak) were separated using 4% i-PrOH/hexane and 8% i-PrOH/hexane respectively, both at 1ml min^{-1}. The R_f values were 28 min (Fa), 37 min (Fb) and 16 min (H^1).

Compounds D,E,Fa and Fb were obtained in $\geq$ 95% purity, but the peak separated from fraction H^1 revealed two roughly equal components when further examined on an ODS reverse phase column.

Points of interest noticed during the purifications included:

a) a leading shoulder developed on some peaks as the sample size increased. This shoulder, presumably an overloading effect, occurred in a range of solvent systems and more readily with some compounds than with others.

b) lowering the solvent strength did not always simply space out the peaks; some moved faster than others, leading to overlapping peaks and changing elution patterns. A gradient system allowing precise small changes in solvent composition is thus invaluable for examining a complex mixture.

c) a reversal of the elution order of the components of fraction E occurred when changing from an i-PrOH/hexane solvent system to a hexane/$CHCl_3$/MeOH solvent system.

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SPORIDESMIN PURIFICATION BY PREP HPLC.

J W Ronaldson, Ruakura Agricultural Research Centre, Hamilton

Facial eczema has been traced to sporidesmin, produced by the fungus, *Pithomyces chartarum*. Pure sporidesmin used to be obtained from cultures, after chromatography on alumina, partition chromatography with carbon disulphide followed by adsorption chromatography on silica gel with benzene - 3 weeks' work.

To produce sporidesmin for extensive animal studies Prep HPLC was adopted whereby decomposition of the toxin by alumina, and large volumes of flammable and toxic solvents might be avoided. After a number of runs with crystalline sporidesmin and then with partly processed concentrates in different solvent systems it was found that chloroform, with about 1% v/v ethanol yielded ca.1g crystalline sporidesmin at a k' of 3. 50ml chloroform completely dissolves ca. 20g concentrate.

To obtain the quantity (3 l/run) and quality of chloroform routinely, it is circulated through a 34mm column of 340g calcined (500°) alumina inserted between the Prep HPLC and the solvent reservoir. This system lowers the concentration of ethanol, removes the water and any yellow pigments which move off the column subsequent to a run. When the column of alumina is in the circuit, the Prep HPLC pumps the solvent at 150ml/min. An hour's run is sufficient. When a porosil column has reached equilibrium with the solvent - recorder trace- the alumina column is removed and the concentration of ethanol corrected according to the i.r. At ca. 3600cm^{-1} is the O-H stretching peak for ethanol in chloroform, and at ca. 1600 is one for water. Any solvent system might hade been successful had it not been for the presence of sporidesmolides. These are non-u.v. absorbing depsipeptides which are fairly insoluble. Nevertheless they do dissolve in chloroform, and in that solvent they appear before and after sporidesmin.

THE APPLICATION OF ION-PAIR HIGH PRESSURE LIQUID CHROMATOGRAPHY TO THE RAPID ANALYSIS AND ISOLATION OF UNDERIVATISED AMINO ACIDS, PEPTIDES AND PROTEINS.

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and

Milton T.W. Hearn*, M.R.C., Immunopathology Research Unit, University of Otago Medical School, Dunedin.

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Abstract

This article describes liquid chromatographic strategies for the rapid analysis of amino acids, peptides and proteins. The chromatographic systems consist of reversed-phase columns (C18— or alkylphenyl-silicas) and mobile phases which contain ion-pairing or column modifying reagents. In solution ion-pairing reagents associate with oppositely charged ionised groups on the sample molecule and, thereby, increase or decrease the polarity of the sample depending pon the nature of the ion-pairing reagents. For example, a polar ion-pairing reagent such as dihydrogen phosphate will lead to a more polar complex which exhibits decreased retention on a non-polar reversed-phase column. Alternatively, the use of hydrophobic ion-pairing reagents, e.g. hexanesulphonate, can be used to increase the retention of a sample. Besides influencing the k' values of solute molecules, ion-pairing reagents can also effect the selectivity of a reversed-phase column resulting in different elution orders for a series of peptide samples. Amphiphatic molecules, e.g. tetrabutylammonium salts, sodium dodecylsulphate, etc., may modify the stationary phase by forming dynamic ion-exchangers. At a suitable pH these exchanger systems can also be used to analyse peptides or proteins.

The use of these techniques for the separation of complex mixtures of peptidic components, e.g. peptide maps of proteins, together with a variety of peptide and protein examples will be discussed.

Introduction

A previous article in this Journal introduced the basic concepts of high pressure liquid chromatography (HPLC) [1]. It is the purpose of the present paper to illustrate the potential of this new technique by describing recent advances in one area of importance to the Life Sciences, namely the analysis and isolation of amino acids, peptides and proteins. These natural products are becoming increasingly important in industrial as well as academic and medical areas. Since protein products form a major part of NZ's agri-business the development of new separatory techniques in this field is particularly important.

Early application of HPLC techniques to the analysis of underivatised peptides and proteins with liquid/solid or liquid/liquid reversed-phase systems was not entirely successful. Poor resolution was frequently observed, associated with peak broadening and long retention times [2]. To be of general application in peptide and protein chemistry, HPLC techniques must be able to utilise or manipulate three important properties of these amphoteric molecules.

Firstly, the methods should have the potential to distinguish not only major differences in composition and structure but also minor variations, e.g. an amino acid replacement or deletion. Since more than 20 different amino acids are known to occur in nature and the resultant variety of natural peptides and proteins is very large, this structural element presents formidable chromatographic challenges. Obviously, elution conditions which optimise the electrostatic, hydrogen bonding or hydrophobic interactions of the solute molecules, either intramolecularly or with components in the mobile or stationary phases will determine the ease of a chromatographic separation. Secondly, the methods must be compatible with high sensitivity detection systems. Many natural peptidic molecules of interest are available in only minute quantities, e.g. viral proteins, neuroendocrine hormones. Thirdly, biological activity of proteins is dependent on the maintenance of their correct three-dimensional structure and thus loss of activity could arise from interaction of the protein sample with the chromatographic support or incompatibility with the mobile phase. This latter point is of considerable importance when the HPLC methods are to be used preparatively.

Recent developments in this area have concentrated on the use of microparticulate reversed-phase packings with aqueous-organic solvent mixtures, partly because the chromatographic behaviour of peptidic materials can be significantly improved by the addition of suitable counter-ionic agents to the mobile phase [2-4]. This strategy can, in fact, be applied to any organic molecule which has one or more ionisable groups. In addition, many of these buffer systems are transparent to less than 200nm and thus, a variable wavelength UV spectrophotometer can be used as an universal detector. The use of a micro flow-through cell, (usually less than 10µl) allows high sensitivity detection, for example, 0.1ng of tyrosine can be detected at 190nm [5].

The Principle Of Ion-Pairing And Phase Modification

The most commonly used reversed-phase packings consist of 10µm fully porous, silica particles which have been chemically bonded with alkyl chains. The C18— or C3H7C6H5 (alkylphenyl) bonded phases have proven to be the most useful derivatives to date. In general, the more polar a substance, the weaker is its interaction with a hydrophobic bonded stationary phase and thus the shorter its chromatographic retention on the column. An increase in the organic solvent concentration in the mobile phase decreases the interactive forces between the sample and the stationary phase. Therefore, an organic solvent gradient can be used to elute samples of decreasing polarity which are progressively retained more strongly under isocratic conditions to the hydrocarbonaceous stationary phase.

Ion-pair chromatography has attracted considerable attention in recent years. For reserved-phase systems two theoretical models — the partition model and the ligand adsorption model — have been proposed which accommodate most of the experimental data (for recent reviews see refrences 6, 7). With ionisable molecules like peptides the capacity factors, k's, and the selectivities, α's, have been found to be dependent, inter alia, on the pH and the nature and concentration of both the counter ion and the mobile phase. Since these parameters are easily controlled or varied experimentally considerable flexibility can be achieved in the separation of complex mixtures.

It is now a well documented observation [7-13] that an ion-pair can be formed between a charged or ionisable molecule and a suitable, oppositely charged ion added to the mobile phase. The complex will then segregate between the stationary and mobile phase either by

partition or adsorption mechanisms according to the net polarity of the ion-pair. A very non-polar sample, which would have an inconveniently long retention time on a reversed-phase column, can be complexed with a hydrophilic ion-pairing reagent which will cause a large decrease in the retention time (Fig. 1a).

Conversely, a polar sample which has insufficient retention on a reversed-phase column can be analysed satisfactorily in the presence of a hydrophobic ion-pairing reagent. The use of a variety of ion-pairing reagents allows the analysis of peptides and proteins which exhibit a wide range of polarities with a single set of mobile phase and column conditions.

Since peptides contain both cationic (RNH3+) and anionic RCOO−) groups it is possible to use mixed ion-pairing. An extensive variety of cationic and anionic hydrophilic and hydrophobic ion-pairing reagents are available (see Table 1 and refs. 7, 8). Since the stability of the present generation of reversed-phase columns effectively limits the operating pH to the range 2—7.5, anionic reagents which associate with ammonium groups in the sample have been more widely used [2-4]. At low pH values, the ammonium ion tends to dominate the polarity of peptide and protein molecules and thus interaction with anionic ion-pairing reagents can cause an apparent change in polarity of the sample [9, 14].

W.S. Hancock, B.Sc. (Hons), Ph.D. from Adelaide University (upper right) arrived at Massey University in 1971 as a senior lecturer in the Chemistry, Biochemistry and Biophysics Department. Principle research interest is in the chemical synthesis of peptides and proteins which has involved the synthesis of a 51 residue apolipoprotein (CI) and in smaller peptide hormone releasing factors, e.g. Somatostatin.

C.A. Bishop, B.Sc. (Hons) in Biochemistry from Victoria University (upper left). From 1970 worked at Massey as a research technician and has studied the purification of enzymes from natural sources for commercial and research use. From 1974 he has worked on the application of high pressure liquid chromatography for the analysis of amino acids, peptides and proteins.

Dr Milton Hearn (left) completed his Ph.d. in organic chemistry in 1969 at Adelaide University. After working with Dr Jim Kutney in Vancouver and Prof. Sir Ewart Jones in Oxford, he moved to NZ in 1974, initially as an MRC Senior Research Fellow. He has also been the recipient of a George Murray travelling fellowship, an NRC post-doctoral fellowship at the University of British Columbia and an ICI senior fellowship at Oxford University. He has worked on problems associated with the isolation and structure of natural products and, more recently, in the field of peptide and protein chemistry, particularly related to the bio-medical area.

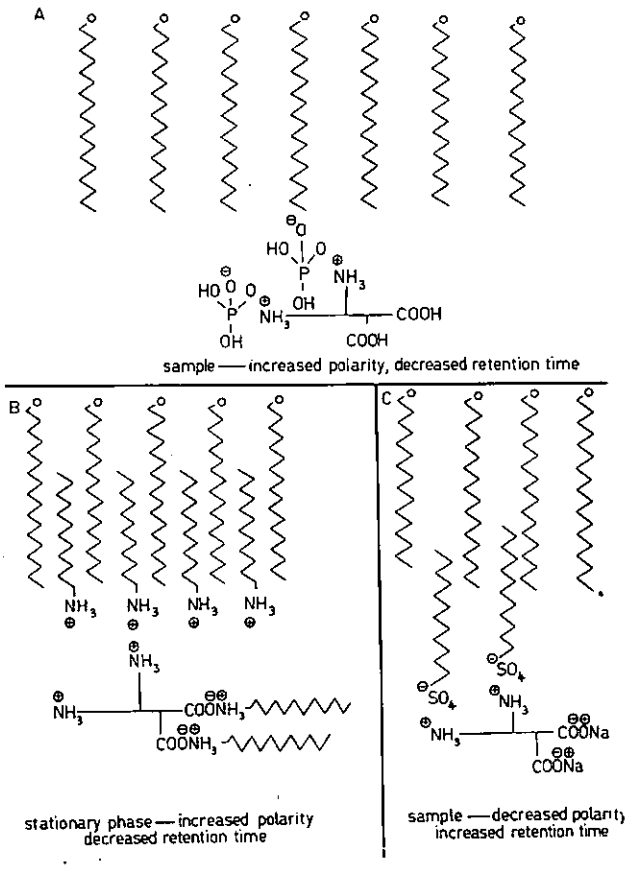

Fig. 1a. The effect of hydrophilic ion-pairing on the interaction of a peptide with a non-polar stationary phase. The C18 stationary phase attached to the silica beads is shown diagrammatically at the top of this figure. At pH2.6 the ionisation of the carboxylate anion is suppressed while the ammonium ions will ion-pair with the dihydrogen phosphate ions.

Fig. 1b. The effect of the dodecylammonium ion on the retention time of a peptide. At pH4 the ionisation of the peptide is as shown in this figure while the dodecylammonium ion coats the stationary phase to form a dynamic cation exchanger.

Fig. 1c. The effect of sodium dodecylsulphate on the retention time of a peptide sample. The effect of this anion is presumably due to mixed ion-pair and phase modification effects, both of which tend to increase the interaction between the peptide and the stationary phase. At pH7 the ionisable groups on the peptide will be as shown in this figure.

With amphiphatic complexing reagents which have significant non-polar regions an additional event can occur, namely modification of the chromatographic support. These reagents have the potential to modify the non-polar stationary phase by the formation of a surface layer of different polarity. If the phase-modifying reagent used has an ionisable group then dramatic changes in retention characteristics are observed. If, for example, the peptide or protein sample is analysed at a low pH in the presence of dodecylamine, then very short retention times are observed (Fig. 1b). In contrast, the addition of sodium dodecylsulphate to the mobile phase will greatly increase the retention time of the sample (Fig. 1c). In these cases, the changes in k' and selectivities presumably reflect the involvement of dynamic ion exchangers. Similarly, polar reagents (e.g. dodecanol) which do not ionise can be used to change k' and α — values. Recent reports [2, 3] have examined the effect of these ion-pairing or phase modifying reagents with a variety of peptides and proteins. As might be expected from the wide diversity encountered with biological samples considerable flexibility in the chromatographic systems is essential to ensure that adequate separation can be achieved with these methods.

Applications

Amino Acids: Recently, there has been considerable progress reported for the separation of PTH— and Dansyl— amino acids by HPLC. These methods, however, require a derivatisation step and are more suited to studies involving the sequential degradation of proteins. Obviously, reliable methods for the separation of underivatised amino acids by HPLC would be most useful in view of the inherent advantages of high resolution and reproducibility, together with a nondestructive method of analysis.

As in shown in Fig. 2a, in the presence of hydrophilic ion-pairing reagents, e.g. dihydrogen phosphate or perchlorate at low pH, the separation of non-polar amino acids can be readily carried out using either isocratic or gradient elution conditions on non-polar reversed-phases [5, 15]. Under these conditions, the elution order of the amino acids approximately follows the relative hydrophobicities of the side chains. This inherent molecular property has been exploited for the separation of the thyroidal iodoaminoacids [16]. In addition, high sensitivity detection is possible at 190-210nm permitting quantitation of nanogram levels of free amino acids [5]. With hydrophilic ion-pairing the polar amino acids Glu,Asp,Ser,Thr,Ala and Gly all elute rapidly from the reversed-phase column (2.7 — 2.9min) so that insufficient resolution is obtained. As is shown in Fig. 2b, these amino acids can, however, be easily resolved using mixed ion-pairing and phase modification conditions. These results illustrate the use of both hydrophilic (decreased the retention of the non-polar amino acids) and hydrophobic (increased the retention of the polar amino acids) ion-pairing. Since the 20 different naturally-occurring amino acids exhibit a wide range of polarities in their side chain groups, the present trend is towards the use of combined ion-pairing and phase modification conditions so that the full range of amino acids can be conveniently analysed in a single chromatogram.

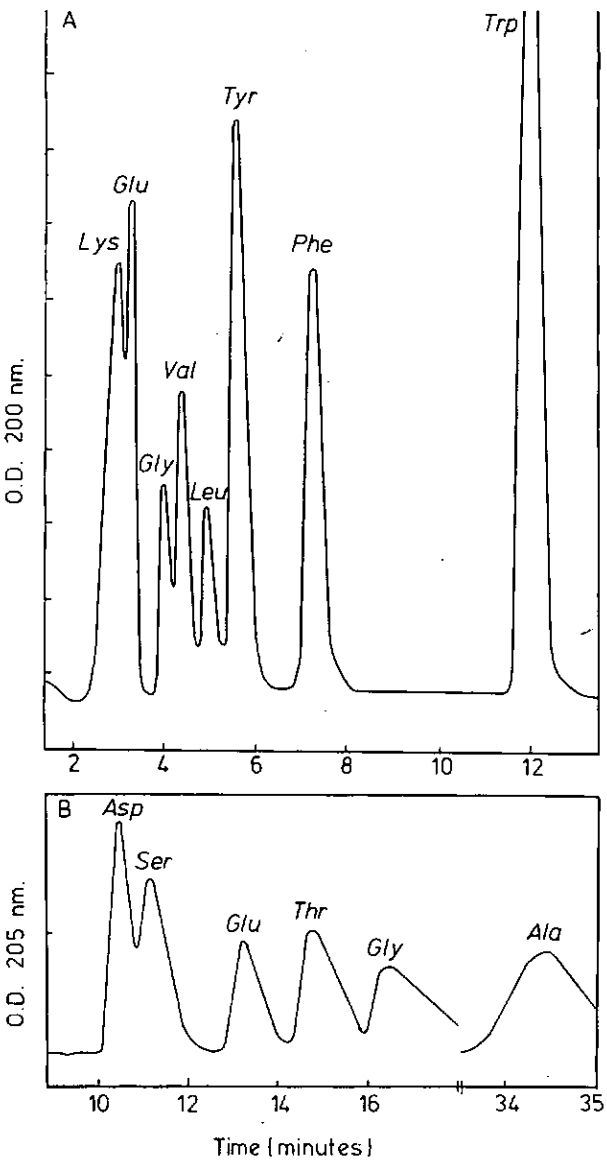

Fig. 2. (a). The separation of an amino acid mixture on a μ—Bondapak-alkylphenyl column which had been equilibrated with 0.1% phosphoric acid, pH2.5, flow rate of 1.5ml/min.
(b). The separation of polar amino acids on a double μ—Bondapak—C18 column at a flow rate of 2ml/min with an aqueous mobile phase containing 10mM NaH2PO4—5mM SDS/0.1%/H3PO4/1% tert-pentanol, pH2.75. (Reproduced by permission of the Journal of Chromatography and Marcel Dekker Inc., from ref.5.)

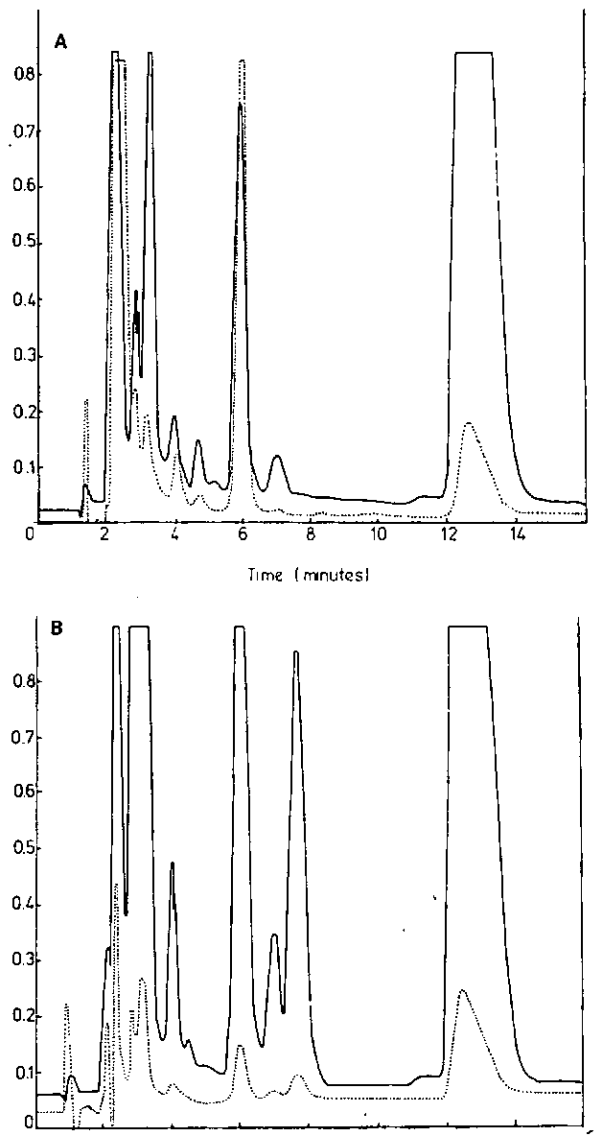

Fig. 3. Comparative elution profiles of solid phase (a) and solution phase (b) synthesis of O-benzyl-Leu-enkephalin amide. The chromatographic conditions were: column μ—Bondapak-alkylphenyl flow rate 2.0ml/min., mobile phase 30% acetonitrile-water-0.1% phosphoric acid. The solid line refers to OD220 and the dotted line refers to OD254nm. (Reproduced by permission of Marcel Dekker Inc., from ref. 24).

The ability of more complex counterions, e.g. metal chelates like C12—dien—Zn(II), to confer further selectivity differences in amino acid separation, has opened the way to the resolution of enantiomeric mixtures with chiral counterions [17].

Peptides: The separation of peptides by partition reversed-phase liquid chromatography was reported in 1976 [18, 19]. Subsequently, we demonstrated that the addition of phosphoric acid or phosphates to a mobile phase consisting of a mixture of water and an UV transparent organic solvent offered very real advantages in the analysis of unprotected peptides and proteins [9]. By comparing

columns 1 and 2 in Table II, it can be seen that a marked decrease in retention time was observed under these conditions together with improved reproducibility [9]. Presumably, the effect of phosphate on retention times is mediated by hydrophilic ion-pairs which forms highly solvated anionic complexes that are less able to partition into the stationary phase. Also, phosphoric acid permits the use of significantly lower concentrations of organic solvents in the mobile phase, thus reducing the possibility of precipitation. These results have been confirmed and extended by other studies [4, 14, 20].

The analysis of peptides by liquid chromatography has been extended by the use of a variety of other ion-pairing reagents [2, 3, 21]. Illustrative of these results is the data summarised in Table II. As can be seen the retention times of a series of peptides can be varied according to the type of counter ion and the mechanism of partition. As anticipated, hydrophilic ion-pairing reagents give shorter retention times than hydrophobic ion-pairing reagents (columns 2 and 3 or 4 and 5).

At pH4, where the ionisation of the carboxylate ions is partially suppressed, the introduction of a cationic stationary phase modification with tetrabutylammonium salts causes the peptide samples to elute very rapidly (see column 7, Table II). At pH4 the peptides are cationic and interact minimally with the dynamic cation ion-exchanger. The addition of the dodecylsulphate anion (SDS) to the mobile phase would be expected to modify the reversed-phase column to a dynamic anion exchanger. If the pH of the mobile phase is chosen so that the peptide is cationic, e.g. pH2.9 for the peptides G-G-Y, G-F and G-L-Y or pH7.15 for the arginine containing peptides L-W-M-R, R-F-A, M-R-F and L-W-M-R-F, the samples are strongly retained on the column (see column 6, Table II). At pH2.0 the arginine containing peptides, with a charge of +2, are indefinitely retained on the reversed-phase column.

Thus it can be seen that the use of ion-pairing and/or phase modification offers a powerful method of varying the retention of peptides on reversed-phase systems. The analysis of a peptide using two different types of mobile phases, e.g. hydrophilic and hydrophobic ion-pairing, can be used as strong evidence for homogeneity similar to thin layer chromatography in several different solvent systems [14].

One important use of HPLC for the analysis and purification of peptides has been the application to synthetic materials. The following example is typical of the approach currently being followed in several research and pharmaceutical institutions. Initially, an analytical separation is established, followed by semi-preparative and finally large scale preparative separations. A pentapeptide, the O-benzyl derivative of Leu-enkephalin amide, was prepared by two different routes using either the solid-phase method in which the carboxyl terminal amino acid leucine was attached via a benzyl ester linkage to a polystyrene resin, or a solution synthesis in which the p—(p—N, N—dimethylamino phenylazo)benzyl ester of leucine was used. After completion of the synthesis, O-benzyl-Leu-enkephalin amide was isolated following deprotection. The analytical HPLC profiles of the crude products prepared by the two methods are shown in Fig. 3a, b. These profiles indicate that a variety of side products, as well as non-peptide materials, are present in the crude product [22]. The use of dual wavelength detection (220 and 254nm) allows one to distinguish between peaks which contain aromatic amino acids from peaks

TABLE I

(a). HYDROPHILIC ION-PAIRING REAGENTS

HPO_4^{2-}, ClO_4^-, CF_3COO^-, ($R\overset{+}{N}H_3$)

$\overset{+}{N}H_4$, Na^+, Mg^{2+}, ($RCOO^-$)

(b). HYDROPHOBIC ION-PAIRING REAGENTS

$C_6H_{13}SO_3^-$, $C_7H_{15}SO_3^-$, $C_{12}H_{25}OSO_3^-$ } ($R\overset{+}{N}H_3$)

$(CH_3CH_2)_4N^+$, Cs^+) ($RCOO^-$)

(c). STATIONARY PHASE MODIFICATION

$C_{12}H_{25}OSO_3^-$	polar, charged.
$C_{12}H_{25}\overset{+}{N}H_3$	polar, charged.
$(C_4H_9)_4N^+$	polar, charged.
$C_{10}H_{21}OH$	polar, neutral.
$CH_3CH_2C(CH_3)_2OH$	polar, neutral.

Selected examples of hydrophilic, hydrophobic and stationary phase modifying reagents. The ionised groups in parentheses refer to ionisable functional groups on the sample molecule.

TABLE II. THE EFFECT OF ION-PAIRING AND PHASE MODIFICATION ON THE RETENTION OF PEPTIDES ON A μ-BONDAPAK-ALKYLPHENYL COLUMN. Eluant: methanol-water (1:1). The values given are the retention times in min.

PEPTIDE[1]	ION-PAIRING REAGENT					PHASE MODIFICATION	
	None	$-\overset{+}{N}H_3$		$-COO^-$			
		Hydrophilic[2] $H_2PO_4^-$	Hydrophobic[3] $CH_3(CH_2)_5SO_3^-$	Hydrophilic[4] Mg^{2+}	Hydrophobic[4] $(C_2H_5)_4N^+$	Anionic $CH_3(CH_2)_{11}SO_4^-$	Cationic[7] $(C_4H_9)_4N^+$
G-G-Y	64.5	1.9	2.4	2.2	2.2	5.5[6]	1.7
R-F-A	> 48	2.05	3.0	2.3	2.6	33.2[5]	1.8
L-W-M-R	112	2.3	4.0	2.8	3.05	16.2[5]	2.7
G-F	2.3	2.4	2.3	2.2	2.6	8.9[6]	2.05
G-L-Y	2.5	2.4	2.4	2.3	2.8	9.3[6]	2.7
M-R-F	32.5	2.5	3.5	2.6	2.8	> 58[5]	1.7
L-W-M-R-F	> 120	5.1	10.2	4.5	6.5	40.5[5]	4.0

1. The code for amino acids is: A=alanine, D=aspartic acid, F=phenylalanine, G=glycine, L=leucine, M=methionine, R=arginine, W=tryptophan, Y=tyrosine.

2. 0.1%, pH2.5; 3. 5mM, pH6.5, Na^+; 4. 2mM, pH4, ^-OAc; 5. 5mM, pH7.15, Na^+; 6. 5mM, pH2.9, Na^+

7. 5mM, pH4.1, ^-OAc.

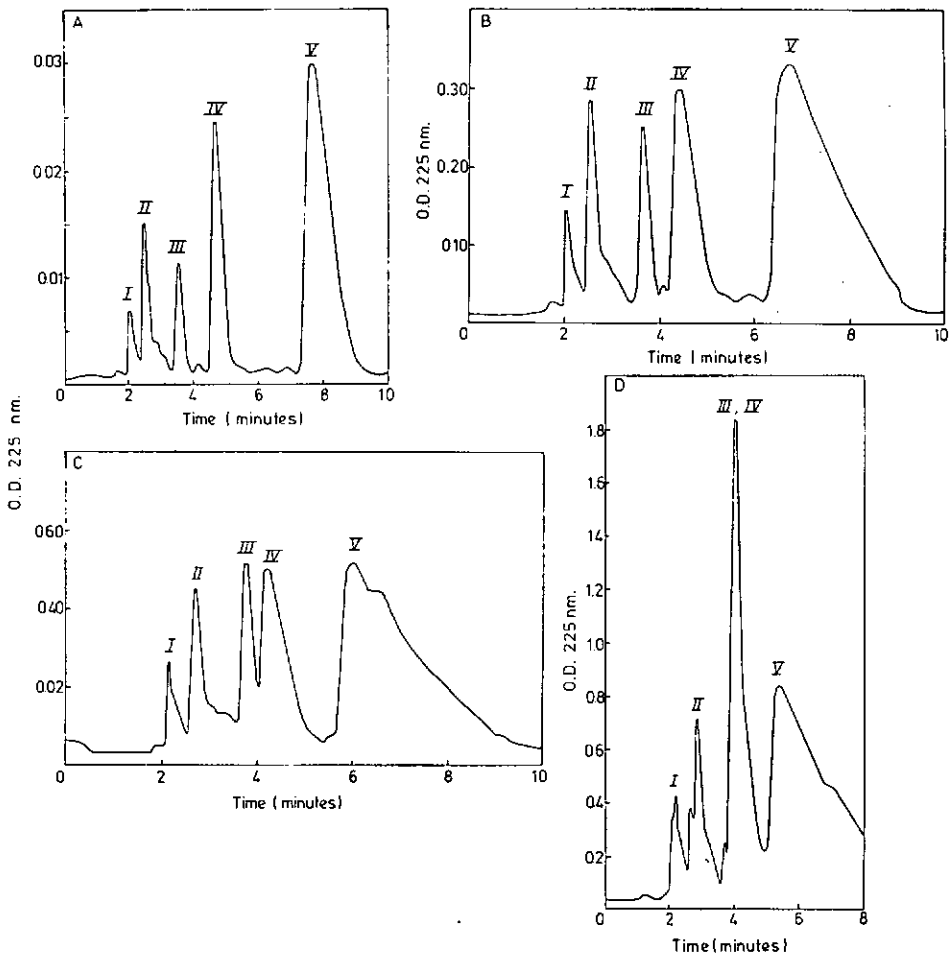

Fig. 4. The elution profile of increasing amounts of O-benzyl-Leuenkephalin amide on a μ-Bonda-pak-alkylphenyl column. The amount of material injected at each analysis was as follows: (a — 2μg, b — 20μg, c — 100μg, d — 200μg). In each analysis, peak V corresponded to the desired peptide. The chromatographic conditions used were — flow rate 2.0ml/min, mobile phase 60% methanol-water-0.1% phosphoric acid. Methanol was used instead of acetonitrile in the separations shown in Figs 4 and 5A because of the high cost of the latter solvent.

without absorption at 254nm. The peak with a retention time of 13min corresponds to O-benzyl-Leu-enkephalin amide and would normally be purified by a combination of gel filtration and ion-exchange chromatography.

Rapid clean up of the sample to be chromatographed preparatively can be carried out using disposable C18 cartridges (C18—Sep—Pak TM) and a judicious choice of eluant [22, 24].

Small amounts of Leu-enkephalin peptide (0.5mg) were then purified on an analytical reversed-phase column (4mm x 30cm with fully porous 10 micron particles), followed by 5mg of material on a semi-preparative reversed-phase column (7mm x 60cm with 37 — 50 micron partially porous particles). The analytical column can be loaded with relatively large amounts of material although, as shown in Fig. 4a to 4d, successively larger injections decrease the resolution of the peptide from the impurities. At a loading of 0.5mg there is still adequate resolution of the peptide peak from the impurity peaks.

In Fig. 5a the elution profile obtained for the chromatography of 5mg of the crude O-benzyl Leu-enkephalin amide mixture on the semi-preparative column is shown. After removal of the organic solvent by rotary evaporation and the ion-pairing reagent by gel chromatography the purified material was re-examined by analytical HPLC. As can be seen in Fig. 5b the peptide was obtained in a state of good purity. The availability of a new generation of preparative instruments has extended the purification to the gram level for many synthetic peptides.

Proteins: Many proteins are strongly absorbed to non-polar stationary phases when they are applied as aqueous solutions. Often irreversible binding and/or denaturation occur under these conditions, presumably due to strong hydrophobic interactions of the non-polar side chains of the protein with the reversed-phase packing. As the organic solvent concentration in the mobile phase increases, these interactions decrease but the low solubility of many proteins in organic solvents places a limit on the workable concentration. We have found, however, that conditions based on ion-pairing principles allow many of these difficulties to be overcome [2]. Since proteins differ widely in their amino acid composition, structure and stability, it is obvious that a variety of conditions are necessary to study different proteisn by HPLC.

In Table III some examples of proteins which can be analysed by ion-pair partition HPLC are listed. In the absence of ion-pairing reagents none of these protein samples eluted from the reversed-phase column, even in the presence of high concentrations of organic solvents. It can be seen, even from this limited range of examples, that different eluant conditions are required for the satisfactory analysis of different proteins. For example, aldehyde dehydrogenase was not eluted from the column using hydrophilic ion-pairing (0.1% H_3PO_4) and required cationic phase modification (dodecylammonium acetate) before a satisfactory retention time was obtained.

Some of the potential of HPLC for the analysis of proteins can be seen from the analysis of different insulin samples. The partial separation of ovine and porcine insulin shown in Fig. 6 illustrates the excellent resolution which can be achieved with hydrophilic ion-pairing. It is noteworthy that these two molecules differ at only three residues in the A-chain (porcine Thr8—Ser—Ile, ovine Ala8—Gly—Val) and that these residues are on the exposed surface of the native molecule.

Proinsulin is the biological precursor of insulin. Polyacrylamide disc gel electrophoresis of bovine proinsulin indicated that the material isolated by ion-exchange chromatography was contaminated with a minor amount of the partial C— peptide cleavage intermediates. Analysis of this protein sample on a μ—Bondapak—C18 column is shown in Fig. 7. Using the same chromatographic conditions and column, bovine insulin elutes with a retention time of 9.2min.

The major current difficulty with HPLC separations of biologically active polypeptides is the preservation of full biological activity. Optimum analytical conditions may not necessarily be directly suitable for preparative work. However, recent studies with volatile alkylammonium salts (acetates, trifluoroacetates) or phosphates in the pH range 5-7 are encouraging and routine preparative separations may shortly be feasible.

Peptide Mapping Of Proteins

Peptide mapping using either enzymatic or chemical cleavage methods has generated invaluable information on the primary structure of proteins. The major disadvantages of conventional mapping methods result from their low level of resolution and insensitive detection methods for progressively smaller quantities of peptides. Recently, we reported [23] rapid and very sensitive ion-pairing HPLC methods for the analysis of proteolytic digests. We have now extended these methods to a wide variety of proteins using improved procedures and the following examples are representative.

Subtle differences in the primary structure of closely related proteins can often be detected by peptide mapping techniques. Normal haemoglobin and its many variants have been extensively studied using conventional methods which usually require micromolar quantities of protein. Application of reversed-phase HPLC in conjunction with an eluant containing hydrophilic ion-pairing reagents allows rapid and reproducible peptide 'finger-printing' of different haemoglobins at the picomole level. The tryptic digest products of a range of haemoglobin mutants each of which differs in sequence by a single amino acid substitution have been compared with normal Haemoglobin A by HPLC (for the comparison of Hb A, C and J see ref. 8, 24). Comparison of the chromatograms in each case reveals at least one difference in the profile obtained. The importance of the gradient shape in a complex separation such as encountered with peptide mapping is shown in Figs. 8a to 8d.

Clearly, careful optimisation of the gradient conditions can greatly enhance the separation achieved. In addition, the ability to analyse the eluted peptides at more than one wavelength aids in the analysis

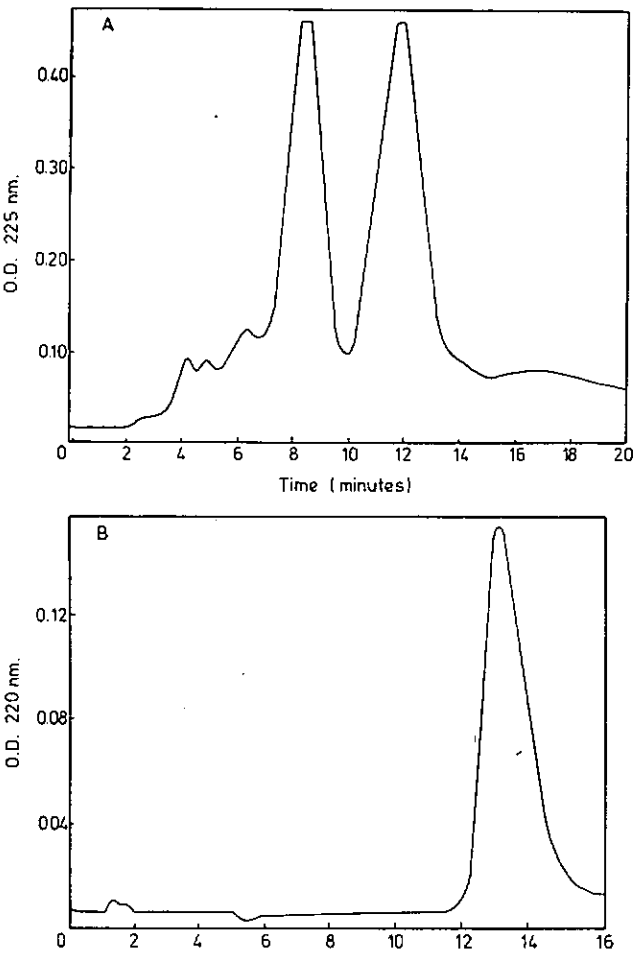

Fig. 5. (A) Semi preparative separation of O-benzyl-Leu-enkephalin amide on a Bondapak phenyl-porasil B column (7mm x 60cm). The mobile phase was 60% methanol-water-0.1% phosphoric acid with a flow rate of 2ml/min and the loading was 4mg.
(B) Analytical elution profile of O-benzyl-Leu-enkephalin amide after HPLC semi-preparative purification on Bondapak phenyl-porasil B column. Chromatographic conditions are as described in Fig. 3. (From ref. 22. Reproduced by permission of the Journal of Liquid Chromatography).

TABLE III. EXAMPLES OF THE ANALYSIS OF PROTEINS ON REVERSED PHASE COLUMNS.

Sample	Eluant[1]	Type of modification	Reagent[2]	Retention time (minutes)
Porcine insulin[3]	60% CH_3OH	Hydrophilic	0.1% H_3PO_4	6.0
ACTH 1-24[4] pentaacetate	40% CH_3OH	Hydrophilic	0.1% H_3PO_4	2.1
Glucagon[4]	40% CH_3OH	Hydrophilic	0.1% H_3PO_4	4.6
Acyl Carrier[3] Protein	5% CH_3CN	Hydrophilic	0.1% H_3PO_4	1.8
	30% CH_3CN	Hydrophobic	5mM sodium hexanesulphonate	4.2
Sheep liver[3] aldehyde dehydrogenase	50% CH_3CN	Hydrophilic	0.1% H_3PO_4	∞
	20% iPrOH	Cationic phase modification	2mM dodecylammonium acetate	1.8
Apolipoprotein C-I[3]	30% iPrOH	Hydrophilic	0.1% H_3PO_4	∞
	5% iPrOH	Cationic phase modification	2mM dodecylammonium acetate	3.2

1. Expressed as a percentage of the organic component, in all cases the other solvent was water.
2. Conditions used as described in Table II.
3. Column used was μ-Bondapak-alkylphenyl.
4. Column used was Bondapak-C_{18}-Corasil.

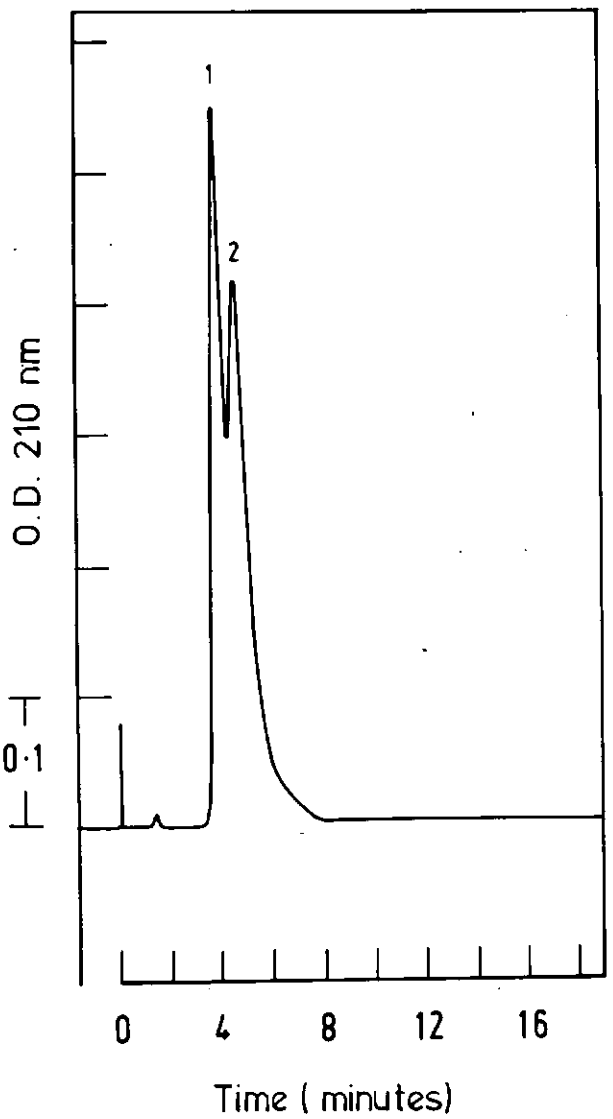

Fig. 6. Chromatographic separation of an equi-molar mixture of purified ovine (1) and porcine (2) insulins. Column: μ-Bondapak-C18, flow rate 4ml/min, 26% acetonitrile-water-0.1% triethylammonium phosphate, pH3.5. (From ref. 24. Reproduced with permission of Marcel Dekker Inc.).

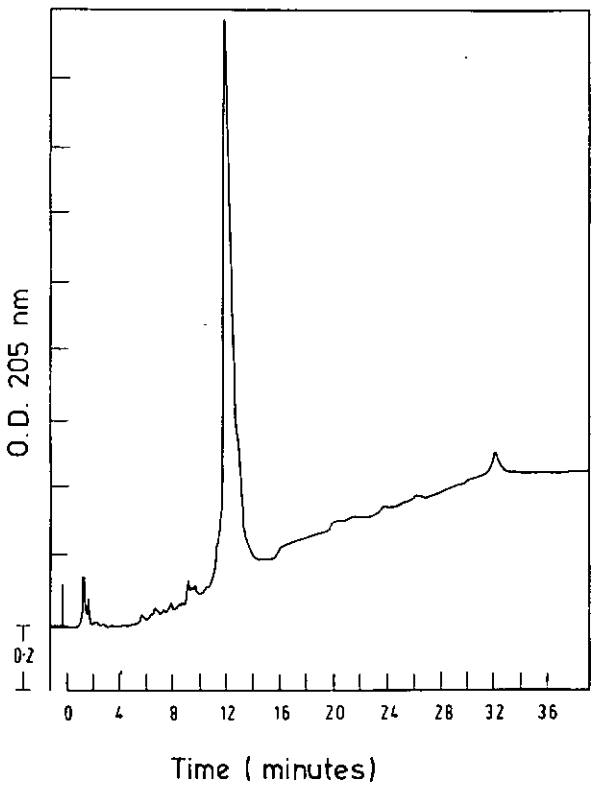

Fig. 7. This chromatogram was obtained with bovine proinsulin using a 30 min linear gradient generated from 10% acetonitrile-water-0.1% phosphoric acid to 75% acetonitrile-water-0.1% phosphoric acid. The column was a μ-Bondapak-C18 and the flow rate was 2ml/min. The gradient was commenced at the time of loading the sample. (From ref. 24. Reproduced with permission of Marcel Dekker Inc.).

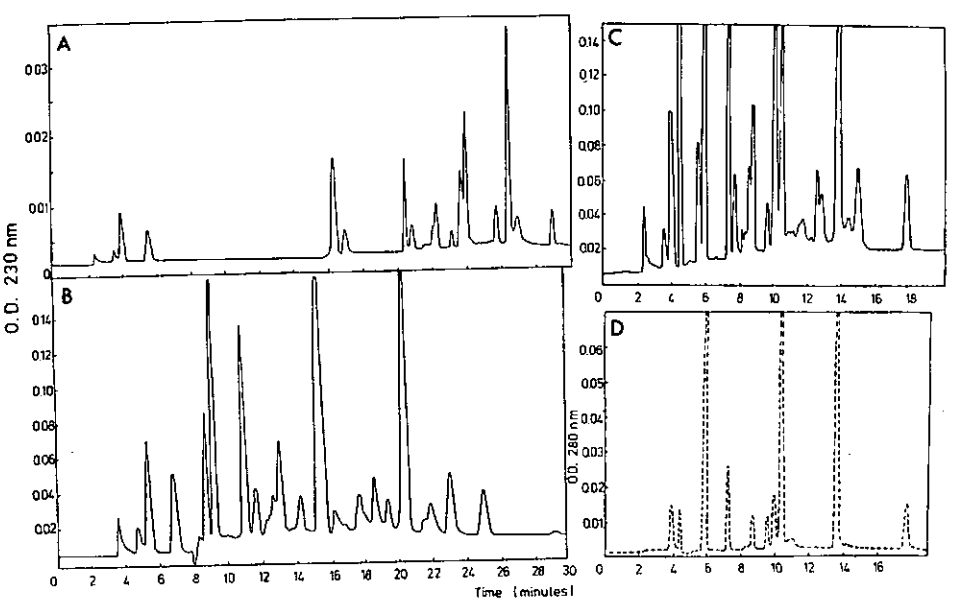

Fig. 8. Analysis of tryptic peptides of haemoglobin A under different chromatographic conditions. Each sample was analysed using a gradient of 0-50% acetonitrile with 0.1% aqueous phosphoric acid on a μ-Bondapak-C18 column. (A) Single column, 3ml/min. (B) 2 columns, 2.7ml/min., 30 minutes concave gradient [No. 7 on the Waters M660 programmer]. (C) Same analysis as in (B) except that a convex gradient (No. 5 on the programmer) was used. (D) Same analysis as in (C) except the eluted peptides were monitored at 254 instead of 220nm.

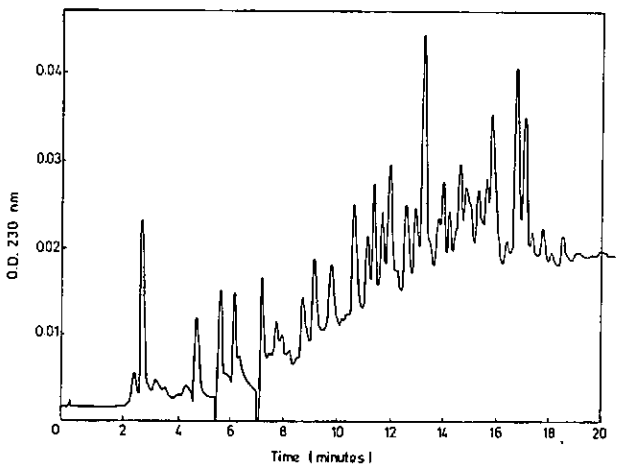

Fig. 9. Gradient elution profile for the tryptic map of sheep heart phosphofructokinase (200μg). The chromatographic conditions were the same as in Fig. 8, part B, and a flow rate of 2.7ml/min were used. (From ref. 24. Reproduced with permission of Marcel Dekker Inc.).

Ion-Pair HPLC (Cont)

of peaks, for example, chromatograph 8d, and allows the identification of peptides which contain Trp or Tyr.

The considerable advantage of using HPLC methods for peptide mapping studies becomes much more apparent when proteins larger than haemoglobin are examined, for example, glycoproteins, antibodies, enzymes. The gradient elution profile for the tryptic map of 200μg of sheep heart phosphofructokinase [24], an enzyme with a molecular weight of 360,000 daltons, is shown in fig. 9. Clearly, the level of resolution which can be obtained with liquid chromatography will be invaluable in the peptide mapping of such proteins.

Summary

The studies reported in this paper show that ion-pair partition reversed-phase HPLC provides a method for the analysis and isolation of underivatised amino acids, peptides and proteins. In addition, a family of closely related proteins can be identified using rapid and sensitive protein mapping techniques, which require relatively small amounts of material. The concept of ion-pairing allows the protein chemist to make at least a partially rational choice of the type of eluant conditions required to elute a given peptide or protein. The conditions developed in these studies are applicable to any organic molecule which contains an ionisable group, so that ion-pairing techniques should also be applicable to steroid conjugates, nucleotides and many carbohydrate derivatives.

Acknowledgements

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HIGH PERFORMANCE LIQUID CHROMATOGRAPHY — ACCELERATING ANALYSES.

W. Roy Day, Waters Associates Pty. Ltd Sydney.

Perusal of current trade and scientific literature concerned with chemical analysis must impress the observer with the rapid emergence of High Performance Liquid Chromatography (HPLC) as a technique for the rapid and quantitive analysis of materials.

The application of pressure to solvents flowing through chromatographic columns to decrease analysis time has been in use for many years. There has been a gradual increase in pressures and linear flow rates to the present point where the practical limits of packed, particulate columns with normal solvents appear to have approached the optimum.

The pressures involved (commonly 100-200 atmospheres) have led to the term "High Pressure Liquid Chromatography" although with the general acceptance of pressures of this order the term "High Performance Liquid Chromatography" is becoming more accepted. Whatever term is used, it is true to say that these devices have attained a level of development which permits analyses which have developed remarkably in resolution, precision and speed.

Another sign of the acceptance of this technique is the publication in 1978 of a journal devoted to this subject: The Journal of Liquid Chromatography.

Undoubtedly the successful use of Gas Chromatography in replacing and enhancing the older "wet chemistry" technique has sped the growth and acceptance of HPLC. Liquid Chromatography will not replace Gas Chromatography of course, but it can extend the range of compounds which can be analysed chromatographically as illustrated by the following diagram:

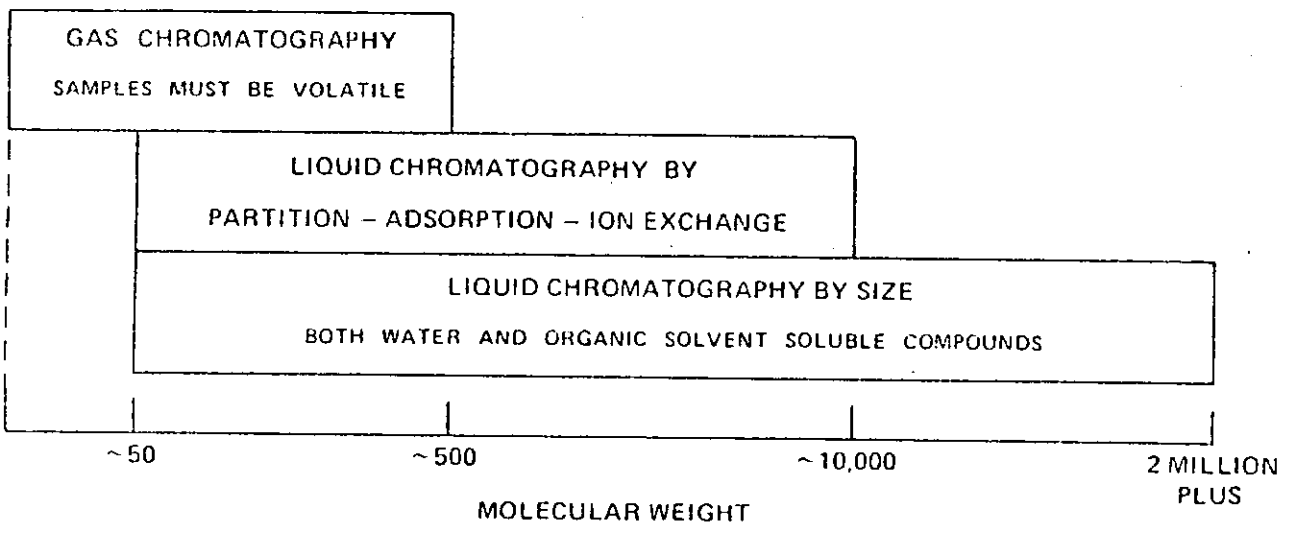
The separation mode covering the greatest molecular weight range is Gel Permeation Chromatography (GPC) which once again is not a new technique. Probably the best known materials used for size separation are polydextran and agarose, gels such as "Sephadex". These materials are restricted to water soluble compounds. A variety of other size separation gels have been developed based on such materials as silica, glass, styrene-divinyl benzene, PVA or polyacrylamide.

The size and distribution of molecular size of plastics can be correlated with the performance of plastics in use. Characteristics such as flexibility, stress cracking elasticity, resistance to wear, weathering etc, are intrinsically tied up with the distribution and sizes of the molecules of the polymer.

Other popular methods for determining these characteristics have relied on osmotic pressure and viscosity measurements but GPC is becoming recognised as the preferred method of examining these materials. The estimation is rapid and produces a "fingerprint" or pattern which can be used to identify polymers suited to particular purposes.

More recently, graded pore-size silicas have been developed which have deactivated surfaces. Deactivation has largely removed the adsorption and affinity effects which have been a problem with such materials. This development has removed the restriction of solvent composition and allows the separation of both water soluble and organic polymers. These materials can also withstand high temperatures. Liquid Chromatographs have been developed to work up to 150°C for the rapid GPC analysis of difficult plastics such as polythene and polypropylene.

COMPARISON OF CHROMATOGRAPHY BY MW



Although it covers a smaller molecular weight range, Affinity Liquid Chromatography (ALC) is capable of greater discretion and resolution than Gel Permeation. The affinity of molecules for the solid stationary phase by processes such as adsorption, partition and ion exchange is particularly versatile in obtaining separation of mixtures.

Untreated silica is still widely used for HPLC column packings. Part of this popularity stems from the familiarity of analysts with separation on silica by Thin Layer Chromatography (TLC). The versatility of affinity column separations has been remarkably increased over those obtained by TLC by the use of "reverse phase" column packings.

These packings are prepared by the chemical bonding of organic radicals to silica. This produces a coating of an organic nature on the silica surface which becomes non-polar and has the opposite character to silica which is polar, hence the term "reverse phase". A variety of radicals have been used, the most popular being alkanes of varying chain length. Two reasons for the popularity of these phases are their chemical stability and predictability in use. Other radicals such as –phenyl, –nitrile and –amine have been used to modify affinity for particular purposes.

Reverse phase packings are often used in a mode called "gradient elution". Using this technique, the polarity of the solvent is gradually changed, commonly from water through to methanol during the analysis. This allows separations of of very complex mixtures and is somewhat analogous to the use of temperature programming in Gas Chromagraphy.

The introduction of reverse phases has led also to a swing away from ion exchange materials which are less stable in operation. Samples of an ionic nature have their ionisation supressed by"Common-ion" additives or by the addition to the mobile phase of a "counter-ion" of opposite charge – the "ion-pairing" technique.

The largely unionised samples are then amenable to separation by reverse phases columns. A group of workers at Massey University have developed these particular techniques into interesting new areas

The detectors used to examine column effluents are commonly small ultraviolet absorbance cells. Other detectors such as refractive index and fluorescence are being used for special purposes. Since these detectors are non-destructive they can be used in series and fractions can be collected for further analysis or identification.

Anaytical columns can be used for prepartion of small amounts of pure compounds. By increasing column size, column capacity can be increased to some degree but with some loss of resolution. Recently a technique called "radical compression" has been used to increase column loadings to the order of grams of sample per run. The extra resolution is obtained by squeezing the silica particles inside a plastic liner thus gaining greater uniformity of the packed bed. These devices are being used for the preparation of pure standards and for highly refined pure chemicals, particularly for testing for physiological activity where spurious results can be induced by small amounts of active contaminants. One of these units is being used in New Zealand for isolating sporidesmin which is a material involved in causing facial eczema,, a source of significant economic losses in parts of New Zealand.

Although HPLC did not really start commercial development until about 1970, its areas of use have increased rapidly both in diverse areas of research and in industry. One of the large areas of increasing use in industry in very recent times has been in the pharmaceutical industry where it has largely taken over routine quality assurance. The technique has such wide applications, the extent of which is still not realised, that its use is bound to increase. Reliable instrument design has, in most cases, begun to stabilize. The main field of growth in the near future will be in development of the wide variety of analyses of which the technique is capable.

Roy Day, A.S.T.C., M.Sc., A.A.I.F.S.T., obtained all his qualifications by part-time study at the Sydney Technical College and the University of New South Wales. Early in his career he worked in industry in the production of veterinary pharmaceuticals and baker's yeast. He transferred to the University of NSW, where he took an interest in analytical techniques in the School of Food Technology. His M.Sc. was done on the aroma of tomatoes, which led to an interest in Chromatographic techniques.

He has taken an active part in the administration of the Australian Institute of Food Service and Technology for many years including being chairman, Food & Beverage Flavour Group, for 2 years and chairman, NSW branch, for 2 years.

In 1978 he joined Waters Associates in Australia and is pursuing developmental work on the use of HPLC in the food industry.

THE USE OF GAS CHROMATOGRAPHY IN AIR POLLUTION STUDIES.

G.C. Brent, C.J. Edmunds, B.W. Graham.
Dept. of Health Environmental Laboratory, Mt. Eden, Auckland.

The responsibilities of the Department of Healths's Environmental Laboratory include the monitoring of various air pollutants. Gas chromatography is used in part of this work, and this paper describes some aspects of this.

Sulphur Gas Analysis

Sulphur containing gases are produced in most combustion processes, in some industrial processes (e.g. Kraft mills), and are also present in geothermal emissions.

To monitor these gases we have acquired a Tracor 270HA automatic sulphur analyser. This employs a flame photometric detector coupled to a multiple column system operated through two 10-port valves to monitor either H_2S, SO_2, and total sulphur in one configuration or H_2S, SO_2, COS, methyl mercaptan, dimethyl sulphide and dimethyl disulphide in another.

A silica gel column is used for the separation of H_2S and SO_2, polyphenyl ether on teflon beads is used for the higher sulphur compounds, and total sulphur is monitored by passing samples directly to the detector without separation. Teflon tubing and stainless steel connections are used throughout and the valves are fitted with two 10ml sample loops. Operation of the instrument can be fully automated, and this can include regular calibration tests from an external calibration source.

Our operating experience with this instrument has been quite satisfactory, although deterioration of columns has been a problem, and it had been used over the last 3 years for extensive monitoring of Kraft mill emissions in the central North Island, and H_2S levels in Rotorua.

QUANTITATIVE ANALYSIS BY GLC OF RETAINED SOLVENTS AFTER PRINTING.

Frank R. Povel, Coates Bros. N.Z. Ltd. Auckland.

The incomplete evaporation of solvents from printing inks can cause problems in the printing industry. To measure the amount of retained solvents, a sample of the printed material can be heated in a closed jar and the vapour analysed by G.C.

In the linear dynamic range of the detector

$$R = p_j(dR/dp_j)$$

where R is the response and pj the partial pressure of component j.

Since the solvent can be partially absorbed in the substrate, pj is not necessarily representative of the total amount of solvent present and the partition between substrate and vapour phase must be taken into consideration. According to Henry's Law and Langmuir's equation, there is a linear relationship netween partial pressure and the total amount (Aj)

$$P_j = \frac{dp_j}{dA_j} \cdot A_j$$

and substitution into the previous equation gives

$$R = \frac{dR}{dA_j} \cdot A_j$$

and the system can be calibrated by determining dR/dAj.

It is, however, often very difficult to obtain a substrate in exactly the same condition as the test sample e.g. the removal of 6% water from a paper sample doubles the partition coefficient for isopropanol.

The problem is easily solved if it is recognised that the calibration in fact determines dR/dAj. If two equal sized samples are placed in separate containers and a known amount of solvent j (dAj) added to one of them, the difference in response is dR.

To test the procedure, "unknowns" were prepared by equilibrating 0.20µl of solvent with 500ml of substrate in a tightly closed container (maximum equilibration time 1½hrs). Two unknown systems, isopropanol and bleached Kraft paper, and toluene and polyethylene were used, and 4 internal standards, isopropanol, isopropylacetate MIBK, toluene (although obviously the solvent and internal standard cannot be the same). Three methods were tested:

1. the solvent addition method described above
2. calibration with preheated substrate and a known amount of solvent
3. calibration with a known amount of solvent but no substrate.

Method 1 gave consistently good results with a standard deviation of about 5%. The choice of internal standard had no measurable effect.

Method 2 gave acceptable results only when pre-heating did not change the properties of the substrate e.g. with but this property is not always predictable.

Method 3 gave results ranging from 0.04 - 0.12µl per 500ml.

Air Pollution Studies (Cont)

Instrument Calibration

In air pollution studies we are usually measuring pollutants present in concentrations in the ppb-ppm range. Furthermore some of these compounds are highly reactive and decompose readily on surfaces and/or in the presence of moisture. Hence the calibration of monitoring instruments presents special problems. A number of different procedures have been used.

Static calibration mixtures can be prepared by injecting a small volume of pure gas into a clean glass jar full of zero air. If very low concentrations are required the resultant mixture is diluted further by repeating the procedure. We have used this method successfully for hydrocarbons, H_2S, MeSH, dimethyl sulphide and dimethyl disulphide, all at ppb levels.

The second method involves an exponential dilution technique. A static mixture is prepared in a container as described above, or using a cylinder of calibration gas, or a permeation tube. The concentrated mixture is then stirred and diluted with zero air at a known constant flow rate. The concentration of the gas mixture leaving the container then decreases exponentially down to the lower levels required. A further advantage of this technique is that the one sample can be used to check the linearity of a detector response over a wide range of concentrations. We have used this technique successfully for H_2S calibration.

Finally permeation tubes are a useful and convenient source of low concentrations of calibration gases. They are, however, expensive ($2-$300 each) and have a limited lifetime (ca. 12 months). Older tubes can still be used but we have found that a 3-year-old SO_2 tube gave marked fluctuations in permeation rate. Calibration of the tubes can be checked by a very careful weighing procedure.

One of the most important requirements in instrument calibration is of course zero air, and with sensitive instruments it can be a major problem, particularly with hydrocarbon analysers. Activated charcoal is an effective trap for sulphur compounds, but we are now aware of an effective trap for methane - which is usually present in ambient air and in all gas cylinders.

The production of low concentrations (ppb) of hydrocarbon mixtures free from much greater proportions of methane (ca 1 ppm) is a problem that we have yet to overcome.

SOME PRACTICAL ASPECTS OF HIGH RESOLUTION GAS CHROMATOGRAPHY.

James R Jeffs, Chromalytic Technology P/L, Glen Waverley, Victoria, Australia

High resolution glass capillary GC is largely determined by the extremely high column efficiencies, and in contrast to the conventional packed columns, liquid phase selectivity is of secondary importance.

High efficiency is essential for the analysis of complex mixtures where the use of packed columns appears in hindsight to be quite impractical compared to the dramatic results obtained on glass capillary columns.

A proper understanding of column efficiency parameters (N-EFF) and Separation Number (T_Z) is of practical significance for optimizing capillary column preformance. The separating ability of a capillary column is best characterised by N_{EFF} as it is directly related to peak resolution and is relatively independent of sample type and column termperature. N_{EFF} (and separating ability) does depend on the capacity ratio K of the peaks concerned which is determined by the liquid phase loading (B). Thus Glass Support Coated Open Tubular (SCOT) columns are preferred for the analysis of volatiles rather than Glass Wall Coated Open Tubular Columns (WCOT).

Separation number is of limited use for column comparison purposes as it is dramatically temperature sensitive and depends on liquid phase polarity.

T_Z can be calculated from N_{EFF} of one knows the relative retentions of adjacent n-alkanes at a particular termperature and for a given liquid phase. A comparison of different column types in terms of N_{EFF} and T_Z helps to put the state of the art in perspective.

For the analysis of complex wide boiling range mixtures where T_Z needs to be maximised, it is readily apparent that for best results-

A POLAROGRAPHIC INDICATOR FOR LIQUID CHROMATOGRAPHY.

B.J. Selman, ANAC Ltd., Auckland

Both for quantitative analysis, polarography is the most widely used of a variety of electrochemical techniques, with usually a Dropping Mercury Electrode (DME) as the working electrode. Essentially the DME is a fine capillary attached to a reservoir: mercury flows through the capillary and exists in the form of droplets. Each droplet presents a completely new electrode surface to the solution, which means there is no past history or poisoning effects.

A disadvantage of the DME is current oscillations caused by charging of the mercury drop and the surface area with time. A refinement of the DME is the lower mercury drop electrode in which the mercury drop is extruded and held at the capillary end before being dislodged. The current is therefore sampled with a stable drop. It is this principle which forms the basis of the PAR 310LC Detector.

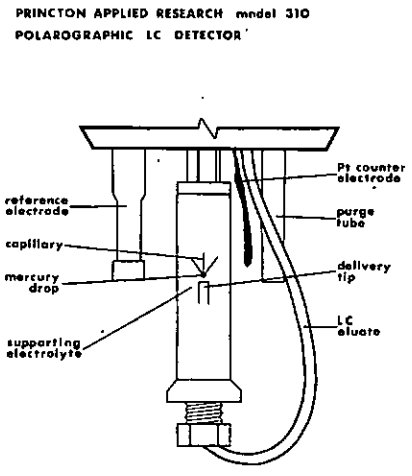
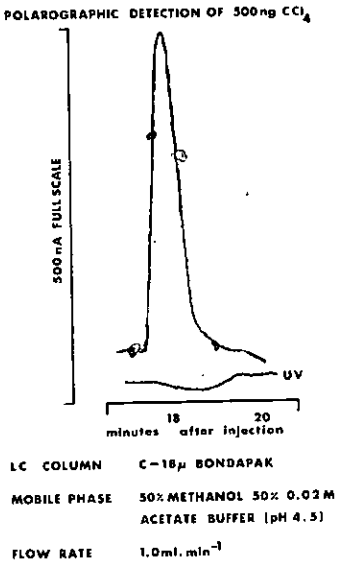


Fig 1. A schematic of the PAR model 310 LC

In operation the 310 is connected to the LC by tubing adaptor and the eluate directed through the delivery tip to the supporting electrolyte and the mercury drop. Here the substance of interest reacts electrochemically with the electrode and the resulting current is recorded as a function of time. As with other detectors, the peak height and area are proportional to concentration. The dead space of the detector is less than 1 microlite.

Polarography complements LC because of its ability to selectively respond to the various functional groups of organic compounds. There are numerous references in the literature to the precise sensitivities and the methods but detection of nanogram qualities is not unusual.

Fig 2.



An illustration of the capabilities of polarography is given in Fig 2 which shows the detection of 500mg of carbon tetrachloride. The UV detector had little response.

Also, the detector will respond well to dissolved metals, a feature not easily available with UV or RI detectors. Information on the oxidation state of the metal can also be elicited.

To configure a practical system, the 310 requires an analyser such as the PAR 364 or 174A. This provides the cell with a stable but variable potential, measures the output current and provides a signal to the recorder. A bonus of such a system is that it can be converted to a standard polarography set-up capable of quantative analyses in the sub ppb range.

Due to the wide range of substances to which the detector will respond, it will find applications in many areas of analytical chemistry, for example food analysis, cosmetics, pesticides, pharmaceutical, petrochemical and pulp and paper industries to name a few.

In conclusion, Polarography provides the analytical chemist with a detector to complement and extend the capabilities of LC. It is straight-forward to use and will interface to most existing LC's.

Practical Aspects of HRGC (Cont)

1) Non-polar phases are preferred;
2) For temperature programmed operation, low heating rates and flow rates slightly above the optimum are required;
3) Columns with low liquid phase loading are advantageous.

For this reason WCOT columns are preferred over SCOT.

In practice the choice of the optimum type of capillary column for a particular separation depends on a number of other factors. The design is a compromise between column efficiency, sample capacity, ease of operation and cost. The relative merits of G-WCOT, G-SCOT and the recently introduced G-Micro SCOT columns are discussed in terms of their suitability for high temperature use, high speed analysis, and their suitability for the analysis of polar samples.

G-SCOT columns are probably the best compromise for many applications and they are recommended as a good starting point for high resolution GC. If the ultimate in efficiency is essential then the next step is to G-WCOT or to G-MicroSCOT columns if high temperature is required.

INDENTIFICATION OF INSECT SEX ATTRACTANTS USING SELECTIVE ION MONITORING.

R. Galbreath, M. Benn & H. Young, Plant Diseases Division, DSIR, Auckland.

A sex attractant, a monounsaturated C_{14} acetate, for the Tortricid moths, *Ctenopseustis obliquana* and *Planotortrix excessana* had been identified in extracts from the female moths. The identification is based on g.c. retention time, mass spectral data, and by trapping and bioassaying the effluent from the outlet of the membrane separator when the monitors ions are detected. The high selectivity and sensitivity of S.I.M. and the high resolving power of SCOT g.c. columns permitted direct injection of crude gland extracts.

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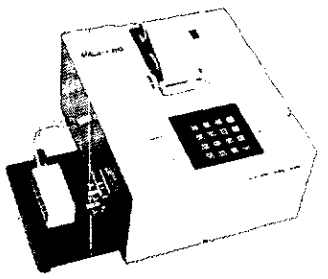

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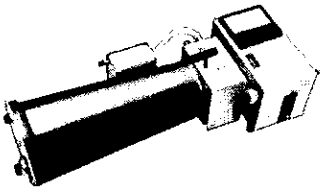

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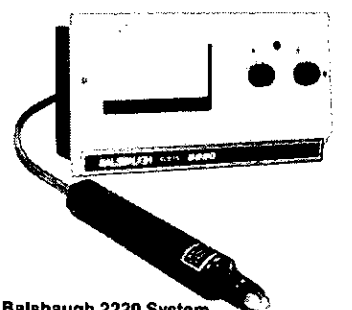

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Outstanding Service Culminates In Institute Leadership For Ted Harvey

Our photo shows the President in the chair at the November Council meeting; on his right is Gavin Fletcher, General Secretary, while on his left is Dr Eric Parker, Registrar, Royal Institute of Chemistry, London, who was a visitor at the meeting.

Dr W.E. (Ted) Harvey has an outstanding record of service to the Institute, having been the Hon. General Secretary for 20 years (1956-76) as well as being Acting - General Secretary on occasions when his predecessor, Mr W.G. Hughson, was overseas. Until the appointment of Mr Denis Hogan as Regitrar, he was also Treasurer. During his years of office, he saw the membership of the Institute grow from 500 to 1300. He has also held various offices in the Wellington branch, including Treasurer and Chairman (1957). For his services as Feneral Secretary, he was presented with a silver salver by the Council in 1977.

Dr Harvey was born and educated in Auckland, where he graduated M.Sc. under the late Professor L.H. Briggs; from this it is not surprising that one of his main reasearch interests has been in the field of natural products. After a short period as a junior lecturer at Auckland, he went overseas: in England he was involved in some pioneer work on nucleic acids with Sir (later Lord) Todd, and he did research on wood chemistry with Prof H. Erdtman in Stockholm before going to Victoria University in 1953. He reached the rank of Associate Professor in the Chemistry Department in 1969, and now holds the post of Registrar – possibly on the basis of his administrative skill shown with the NZIC!

Dr Harvey has also been the Institute's representative on the Technicians' Certification Authority for many years, and has made a significant contribution.

Dr Harvey's wife, Helen, is also a chemist, being on the staff of the DSIR at Gracefield. His outside interests include tramping and hockey umpiring, in which he has reached international status.

UK CHEMISTS' PLIGHT CAUSES CONCERN

The economic plight of chemists in Britain, where 1% were unemployed and many others under-employed was mentioned by Dr Eric Parker, Secretary and Registrar, Royal Institute of Chemistry, London, when he visited NZ in November.

He attended the Council meeting and also spoke at some branch meetings.

He noted that graduates were doing the work of technicians - in some cases at salaries less then those of factory workers in the same plant. Output of graduates from tertiary institutions had fallen from a peak of 3000 in 1970 to less than half that number now, with staffs of universities and tertiary teaching institutes being reduced correspondingly.

The RIC, said Dr Parker (photographed here with the Editor), was not a trade union but there was a body in Britain, originally called the Association of Professional Scientists, but now known as the Association of Management and Professional Staff; it did have trade union status and had gained sole negotiating rights for the scientific staff of ICI.

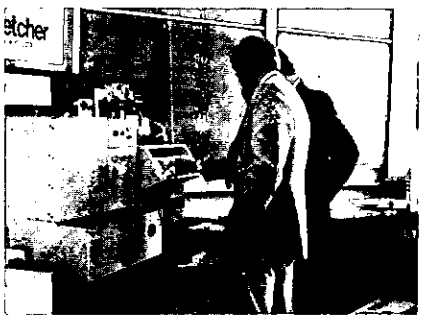
One aspect of the RIC's activities which surprised many who heard Dr Parker was its active political lobbying, both at Westminster, and in the local electorates. It had two advisors in the House of Commons, and in the electorates, members of the RIC are paired with MP's. This system is going well, one result being the issue of four special stamps by the Post Office to mark the Institute's centenary. The RIC is looking at the licensing of chemists in Britain, already a fact in Malaysia and Quebec.

Chromatography Symposium Attracted Interest

A very successful 2-day symposium was organised by the Auckland branch last October. A very good trade display was a major feature of the event and the organising committee appreciated this support.

Several speakers contributed as follows:-

Prof A.L. Odell, Auckland University - Radio GLC of Tritium labelled compounds.



The Perkin-Elmer display aroused visitors' interest

P. Robinson, Waikato Technical Institute - An automated on-line gas chromatography system for analysis of natural gas.

Prof M. Meerkin, School of Medicine, Auckland University - Selection criteria for an HPLC.

S. George, Hewlett- Packard, USA - A first microprocessor based liquid chromogram.

D. Scott, Waters Associates, Auckland - Vitamin analysis in foods by HPLC

B. Walker, Waters Associates, Sydney - Calibration of gel permeation systems.

Prof Meerkin's paper, which is of general interest, since the criteria can be applied to the purchase of most instruments, will be published in a later issue, where we may include some of the other contributions also.

Chemists' Plight (Cont)

Dr Parker spent some time discussing the proposed union of the RIC with the Chemical Society (CS), to form the Royal Society of Chemistry, to take effect in July 1980. While all members of the RIC would become members of the CS, and would use appropriate letters after their names, e.g. FRSC instead of FRIC, members of the CS would be classified as Associates of the RIC. The total membership of the combined body would be 40,000. We have since learned that at consecutive extraordinary general meetings of both bodies on November 21, the proposal was carried by a large majority :- 11,251 to 2,157 for the CS, and 9,677 to 1,145 for the RIC. The percentage of members who voted was 34 and 43 respectively.

SPECIAL SERVICE TO NZIC MEMBERS

Institute members who are without employment are invited to use the Journal's newly-introduced service to assist them in finding posts.

At no cost, they may place a 2.5 centimetre single-column advertisement announcing their availability.

Replies should be directed to the Journal (to ensure confidentiality), which will then pass them on to the person concerned. Insertion will be at the publishers' discretion.

This service is not available to currently-employed members who may be seeking a change. In such circumstances, the normal classified rates apply i.e. $4 per single column centimetre (minimum space 2.5 col.cm) plus a flat $1 per insertion charge if replies are directed to the Journal for forwarding to the person concerned.

1979 BRANCH CHAIRMEN

AUCKLAND

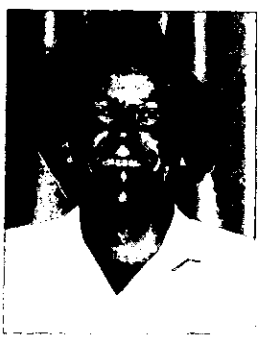
Dr R.A. Dormer

Dr R.A. Dormer has been re-elected to the chair for 1979.

WAIKATO

Dr P. Judd

The new Waikato branch chairman is Dr Paul Judd, who was born in Auckland in 1938. He graduated B.Sc. at Auckland in 1960, and M.Sc. in the following year, after which he became a junior lecturer in the same University. In 1968 he completed his Ph.D., and went to the New Mexico State University, at Las Cruces, USA, on a post-doctoral fellowship. His research interests were in organic rearrangements. In 1969 he became a Tutor at the Waikato Technical Institute and was promoted to his present position as Head of the Science Department in 1971.



Paul Judd

While at University he took a keen interest in soccer being secretary of the University club for several years, during which period the senior team nearly reached first grade status, but it has fallen away since. Dr Judd is married with two children and has a keen interest in gardening.

MANAWATU

Dr A. Brodie

Andrew Brodie was educated at Cashmere High School and Canterbury University from which he graduated B.Sc. (Hons) in 1966 and Ph.D in 1968. His Ph.D research, under the supervision of Prof. C.J. Wilkins and Dr G.A. Rodley, was in the area of co-ordination chemistry. In 1968 he was awarded a British Science Research Council Fellowship, to work at University College, London, with Prof. J. Lewis on organo-metallic chemistry. He returned to NZ in 1970 to take up a lectureship in the Department of Chemistry, Biochemistry and Biophysics at Massey University, where he is now a Senior Lecturer. He spent 1976 as guest lecturer in the Department of Chemistry, Faculty of Engineering Science, Osaka University, Japan.

Dr Brodie is an inorganic chemist with a particular interest in the transition elements, especially the role they play in biological systems. One area of current research is the iron-binding protein, lactoferrin, which is found in high concentration in human milk.

Andrew Brodie

He is also interested in the teaching of chemistry and the school-university interface. Since 1971 he has been a member of the Institute, serving on the Manawatu branch and Conference Committees, and on the Publications Committee, being its chairman in 1978.

WELLINGTON

Dr J.D.B. Featherstone

John Featherstone has spent the past 18 months as a Tutor in Pharmaceutical Chemistry at the Central Institute of Technology, Heretaunga. During this time he was also Director of a Medical Research Council project for research in dental caries. His work in this area has led to the award of an MRC Senior Fellowship which he will take up in early February. He will be based jointly with the Dental Research Unit of the MRC and the Chemistry Department at Victoria University.

John Featherstone

John graduated B.Sc. from Victoria University in 1964 and then spent 10 years in the pharmaceutical and cosmetic industries in NZ and UK, being involved in quality control, product development, process development and production. This was followed by an M.Sc. in the Pharmacy Department of Manchester University with research into intermolecular bonding. He returned to NZ and completed a Ph.D. at Victoria University on 'Chemical Aspects of Dental Caries', in conjunction with the MRC Dental Research Unit in Wellington. He then took up his post at C.I.T.

Dr Featherstone held a Unilever Study Award during B.Sc., a Sandoz Pharmaceutical Research Award during M.Sc., an MRC Postgraduate Scholarship and subsequently an MRC Training Fellowship during Ph.D. His research into dental caries has won the NZ IADR (International Association for Dental Research) Colgate Travel Award (1976), the Australian IADR Colgate Research Award (1976) and the coveted Edward Hutton Award at the World IADR in Copenhagen in 1977.

He is a member of the Institute, a senior member of the European Caries Research Organisation and a member of IADR. John was a contributor to the NZIC working party on Possible Contributions of the Institute to Society.

His research interests are predominantly the chemistry of dental decay but also include intermolecular bonding and its application to drug action. The dental work is aimed at a deeper understanding of dental enamel demineralisation processes with a view to possible preventive measures.

In his spare time Dr Featherstone is active in boating, tramping, mountaineering, and theatre (in conjunction with his wife and 2 children). John is immediate past president of the Hutt Valley Tramping Club, and was a member of the inaugural NZ Outdoor Training Advisory Board. He is a member of the Search and Rescue Organisation and part of the Wellington Face Rescue Team.

CANTERBURY

Mr J. Butchard

Mr Joe Butchard is By-Products Production Manager, Canterbury Frozen Meat Co. Ltd, Christchurch. He is concerned with the processing and production of tallow, meal, casings, wool, pelts, and similar by-products of the meat industry.

Joe Butchard

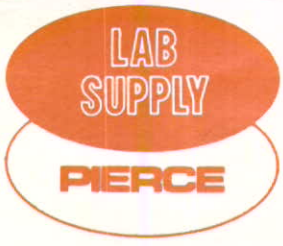
He graduated M.Sc. at Canterbury in 1971, and joined Thomas Borthwick & Sons (A/sia) Ltd as a works chemist. In 1973 he went to a similar position at CFM's Pareora works and was appointed Laboratory Manager at the central laboratory at Belfast in the following year. He took up his present position in 1977.

His principal fields as a chemist have been in effluent treatment and analysis, hygiene, tallow and meal production and pelt processing. His outside interests include the Credit Union movement, church and community affairs, gardening and a young family.

OTAGO

Dr M.G. Shepherd

Maxwell Shepherd graduated B.Sc. (Hons) in Chemistry from Canterbury in 1965. He then moved to Canada and completed his Ph.D. in Biochemistry at Alberta University, Calgary.



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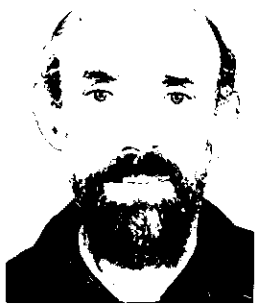
BRANCH NEWS

AUCKLAND

The appointment of Associate-Prof. B.R. Davis of the Chemistry Department to a Personal Chair in Chemistry was announced recently by Auckland University Council.

Prof. Davis has made contributions within the University as a researcher, teacher and administrator. His published work is in the field of organic chemistry where he has made important advances, especially in the studies of the Clemmensen reduction, dienone-phenol rearrangements and organic synthesis. His work has been recognised by the award of the ICI Medal by the NZIC in 1969, and with a D.Sc. from the University in 1972.



Prof. Davis: A personal chair

In his teaching role, Prof. Davis has been an innovator of teaching methods and has played a major part in the organisation and direction of organic chemistry courses at all levels. He has also made considerable contributions to the administrative functions of the University. In addition to a lengthy term as Dean of the Faculty of Science, he has also acted on numerous Senate and Council Committees. These include terms as an Assistant Vice-Chancellor and as Chairman of the Dean's Committee.

* * *

Dr D.J. McLennan, Dept of Chemistry, Auckland University, has been awarded the D.Sc. by the University. He has also been appointed to an Associate Professorship in the Chemistry Dept.

* * *

Professor A.L. Odell attended the RACI Congress at Surfers Paradise in November to give an invited lecture to the Solid State Division. His lecture was entitled "Tritium NMR and Applications in Surface Catalysis". He also lectured at University of NSW on "Metal Ion Catalysis of some Reactions of Coordinated Oxalate".

WAIKATO

Branch meetings: The address to the August meeting was given by Dr C.F. Ramberg from the School of Veterinary Medicine, Pennsylvania University, who is Facial Eczema Research Fellow at Ruakura Agricultural Research Centre. He spoke of "Homeostatic responses to high zinc doses in sheep."

In September Dr R.A. Whitworth, Forest Research Institute, Rotorua, addressed the branch on "Indigenous (renewable) liquid transport fuel supplies", in which he described the FRI developmental work on the production of ethyl alcohol from wood.

At the AGM in November, Dr J. Watkinson gave his chairman's address on "Model systems for selenium in grazing animals." Dr Eric Parker, Registrar of the RIC, was also present.

Forest Research Institute, Rotorua: Dr Terry J. Fullerton has been awarded a Selwyn J. Robinson Scholarship. He will be visiting pulp and paper companies and research establishments in North America in March, and will be presenting papers at the TAPPI conference in New York, and at the ACS Pacific conference in Honolulu, on research relating to the use of anthraquinone as a pulping catalyst.

Waikato University: In October the chemistry department was visited by Dr S.R. Logan of the New University of Ulster. He gave a seminar on photochemical studies of ferrocene and ferrocene derivatives.

Prof. A.T. Wilson is currently in USA on special leave from the University. He is working on the extraction of copper from low grade ores as a director, Duval Corporation, Tucson, Arizona.

The Hamilton Science Centre, Water and Soil Division, Ministry of Works, was visited in October by Dr E. Windle-Taylor, ex-Director of Water Examination, London Metropolitan Water Board. He gave a seminar on "Chemical Pollution of Water in Relation to Health."

MANAWATU

Massey University: Dr Bill S. Hancock (Department of Chemistry, Biochemistry and Biophysics) and Dr Milton T.W. Hearn (Medical Research Council, Dunedin) have been awarded a grant of $50,000 from Waters Associates to further their combined research into protein separation and analysis. The grant will cover the purchase of 3 more liquid chromatographs, two identical to the ones in use at present and the third with a far greater capacity for separation of peptides and proteins. The equipment will be used to further studies on the use of ion pairing reagents for the analysis and purification of peptides and proteins. The grant was made on 8 November, 1978 by Mr Des Scott, general manager, Waters Associates.

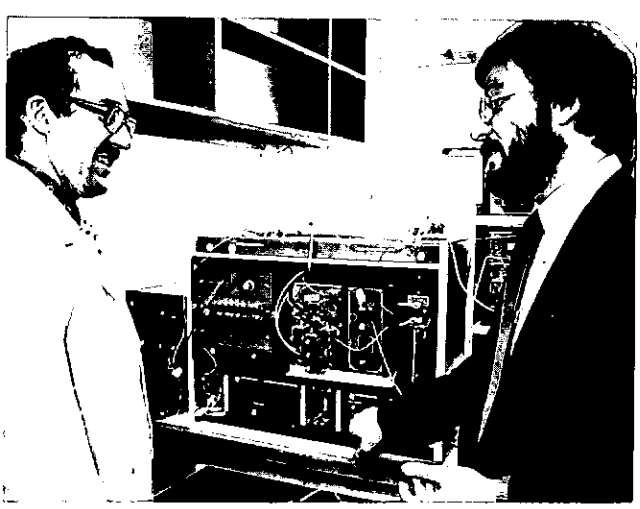
Dr E.N. Baker (Department of Chemistry, Biochemistry and Biophysics) recently received a grant of $16,860 from the University Grants Committee to purchase an Arndt-Wonnacott oscillation camera. This camera is especially designed for collecting X-ray diffraction data for large molecules and it will greatly increase the speed and efficiency of data collection in his crystallographic work on proteins. Together with the automatic microdensitometer and the rotating-anode X-ray generator which is already in the Department, the new camera will result in essentially a complete set-up of equipment for protein crystallography.

Feilding Agricultural High School: Mr Barry E. Hassall, who has been head of the Chemistry Department for 6 years, recently transferred to Taieri High School, Mosgiel as head of the Science Department.

Branch Chairman (Cont)

Maxwell Shepherd

In 1969 he took up his present appointment in the Biochemistry Department at Otago University, where he is now a Senior Lecturer. In 1975 he was awarded a Nuffield Fellowship to work under Prof. Mandelstam at Oxford University on bacterial sporulation. His current research interests are concerned with the control of morphogenesis in the yeast Candida albicans and the mechanism of action of the flavoprotein lactate oxidase.



Dr Milton Hearn (left) discusses with Des Scott, general manager, Waters Associates, the company's $50,000 grant to purchase more chromatographs.

DSIR: Philip D. Pearce (Applied Biochemistry Division) recently returned after 3½ years at Adelaide University where he was studying for a Ph.D. He was working with Prof. W.H. Elliott on an investigation of the transport of proteins across membranes.

The DSIR library expanded at the end of last year to take over the upper floor of the Applied Biochemistry Division —Grasslands Division Building. This will result in an urgently required increase in working area and shelves for books and journals. The library, organised by Miss Cynthia M. Owen, serves these Divisions as well as Plant Physiology Division and the various DSIR substations at Palmerston North. Specialist publications held by the library are used also by staff of the NZ Dairy Research Institute, the NZ Leather and Shoe Research Association and Massey University.

WELLINGTON

The now traditional, more lighthearted, end-of-year supper function was held in the Training Dining Room of CIT. Mrs Pamela Thompson (Hotel and Catering Industry Training Board) addressed the less formal gathering on the development of traditional foods from the pre-Roman period to the present day fully processed diet under the title "Are You Being Processed". An enjoyable time was had by all.

Victoria University: Dr Brian Halton attended the 5th National Organic Division meeting of the RACI in Hobart in mid-January and presented a paper entitled "Skeletal Rearrangements in the Dehydrochlorination of Some Tricyclo-octanes".

DSIR Chemistry Division: Dr W. Passl (Pharmaceuticals Section) has now returned after spending 7 months in Europe studying glass technology as related to the manufacture of pharmaceutical containers. Dr T. Seward was recently invited to lecture on Gold Mineralisation at a joint University/Industry symposium in Perth, Western Australia. Dr W. Giggenbach spent a part of the summer on a further visit to Antarctica.

Dr R.B. Williamson (Water Section) has left Wellington to take up a position with the Ministry of Works at the Waikato Science Centre in Hamilton.

PACRA: Dr Harry J. Percival (Director, NZ Pottery & Ceramics Research Association) returned to NZ in December, after spending 7 weeks visiting research institutes, laboratories, and production units concerned with ceramics in UK, Norway, Germany, Holland and France.

Industrial Activities: One of our younger members (and Wellington branch committee member) Mr Philip Tree, was a candidate for the Western Hutt electorate, standing for the Tory Party (otherwise known as the Imperial British Conservative Party), but was unsuccessful in his bid for entry to the hallowed corridors of power.

On the oil company front, we wonder how many of our members have had to swot up what happens when you put ethanol or methanol in your petrol?

OTAGO

Invermay: Dr D. Forss is presenting papers on "Sensory Characterization" and "Flavours in Dairy Products" at a flavour workshop organised by the American Chemical Society on March 29-30 in Honolulu.

Otago University Chemistry Department: Dr R. Dickson, an inorganic chemist from Monash University, has been working in the department for 4 weeks and Prof. T. Bergman of the Hebrew University, Israel, paid a brief visit to the department.

Biochemistry Department: Prof. Kenneth Burton is presently visiting the Biochemistry Department. He is the William Evans Visiting Professor for 1978 and is from Newcastle University. Prof. Burton has had a long association with the Biochemistry Department. He was the Ph.D. Supervisor for Prof. G.B. Petersen and Associate-Prof. M.G. Smith. Between November 27 — December 1 a Nucleic Acid Sequencing Workshop was held in the Biochemistry Department and there were 17 participants, 8 of whom came from Australia. Dr George Brownlee from the MRC laboratory, Cambridge, also attended. The Workshop was supported by the NZ Biochemical Society, the Medical Research Council of NZ and the medical faculty.

If it's news — tell us about it!
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1979 Branch Officers

(Keep for future reference)

AUCKLAND
Chairman: Dr R.A. Dormer, 18 Maungakiekie Ave, One Tree Hill, Auckland.
Secretary: Mr M.G. Gibson, Auckland Technical Institute, P.B., Auckland.
Treasurer: Mrs F.V. Gifford, Merck Sharp Dohme (NZ) Ltd, Box 23-244, Papatoetoe.
Delegate: Dr L. Eyres, Abels Ltd, Box 9573, Newmarket, Auckland.
Br. Editor: Dr W.A. Denny, Cancer Res. Lab., Box 1724, Auckland.

WAIKATO
Chairman: Dr W.P. Judd, 8 Balmoral St, Hamilton.
Secretary: Dr C.E. Devine, Meat Indus. Res. Institute, Box 17, Hamilton.
Treasurer: Mr O.E. Clinton, Ruakura Agric. Res. Centre, P.B., Hamilton.
Delegate: Dr E. Payne, Ruakura Agric. Res. Centre, P.B., Hamilton.
Br. Editor: Dr P.C. Molan, School of Science, Waikato University, Hamilton.

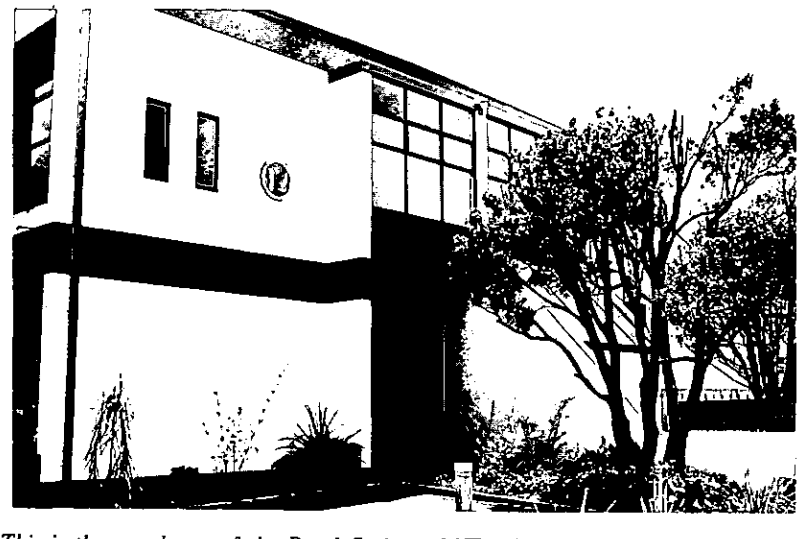
MANAWATU
Chairman: Dr A.M. Brodie, Dept of Chemistry, Massey University, Palm. Nth.
Secretary: Dr D.R. Husbands, Dept of Chemistry, Massey University, Palm. Nth.
Treasurer: Dr K.R. Whittle, P.N. Technical Institute, P.B., Palm. Nth.
Delegate: Dr L.K. Creamer, NZ Dairy Research Institute, P.B., Palm. Nth.
Br. Editor: Dr C.B. Johnson, Applied Biochem. Div., DSIR, P.B. Palm. Nth.

WELLINGTON
Chairman: Dr J.D.B. Featherstone, Pharmacy School, C.I.T., P.B., Trentham Camp P.O.
Secretary: Dr D.M. Bibby, Chemistry Div., DSIR, P.B., Petone.
Treasurer: Dr D.H. Buisson, Industrial Processing Div., DSIR, P.B., Petone.
Delegate & Br. Editor: Dr B. Halton, Chemistry Dept, Victoria University.

CANTERBURY
Chairman: Mr J.A. Butchard, Canterbury Frozen Meat Co., Box 283, Christchurch.
Secretary: Dr H.K.J. Powell, Chemistry Dept, Canterbury University.
Treasurer: Dr J.R. Cretney, Ch.Ch Technical Institute, Box 22095, High St, Christchurch.
Delegate: Dr R.F.C. Claridge, Chemistry Dept, Canterbury University.
Br. Editor: Dr C.G. Freeman, Chemistry Dept, Canterbury University.

OTAGO
Chairman: Dr M.G. Shepherd, Biochemistry Dept, Otago University, Box 56, Dunedin.
Secretary: Dr J.M. McKenzie, Dept of Nutrition, Otago University, Box 56, Dunedin.
Treasurer: Dr J.F. Cutfield, Biochemistry Dept, Otago University, Box 56, Dunedin.
Delegate: Dr P.K. Grant, Chemistry Dept, Otago University, Box 56, Dunedin.
Br. Editor: Mr S.G. Gray, Fletcher Industries Ltd, Box 973, Dunedin.

Royal Society's Wellington HQ



This is the new home of the Royal Society of NZ, where the NZIC Council meets. Located in Halswell Street, the 2-storey building has a total floor area of some 558 sq.m. Administration offices, reception and council room are on the ground floor, while the upper floor includes a fully equipped lecture room seating 100, kitchen facilities and 4 smaller rooms.

RETIREMENT

ARTHUR PLEASANT OLIVER
M.Sc. [Hons] FNZIC

The retirement has been announced of Mr A.P. Oliver, chief chemist, Lion Breweries Ltd.

Arthur Oliver joined the Agriculture Dept as a clerical cadet in the mid 1930's and 2 years later transferred to the Chemistry Section of that Department. Prior to World War II he gained a B.Sc. degree from Victoria University and during the war he served in the Navy where he was involved in the development of radar. He spent time in various shore installations around NZ and served as a radar operator on "Achilles".

After the war Arthur gained an Honours degree from Victoria University and spent some years working as a toxicologist in the Chemistry Division of the Dominion Laboratory. He also worked in the Fats Research Laboratory and as chemist in the vitamin preparation plant of R. Greenwell Ltd, Auckland.

In 1954 he joined the then NZ Breweries Ltd as chief chemist under technical manager Mr J.A. Rhind, and in this capacity was charged with the responsibility of setting up a head office laboratory in Wellington and developing control laboratories in each of the company's branches. As well as his administrative duties he has taken particular interest in a wide range of brewing fields including the agronomy and chemistry of hops, the vitamin requirements of brewing yeasts and the non-biological stability of packaged beers. Along with the current general manager, Mr J.R. Beck (recently elected to the Fellowship of the Institute), he was intimately involved in the development of continuous fermentation as a commercial venture. Arthur's conscientious reading of the brewing and scientific literature, allied to an extremely retentive memory, has led to an international respect for his knowledge of brewing science and technology.

His outside activities have included a keen interest in Institute affairs and for a period during Ted Harvey's absence, he undertook the duties of Hon. General Secretary. He has always shown an interest in NZ flora and over many years has acquired an extensive collection of NZ ferns. For many years Arthur has had a passion for geology in general and the location of gold in particular. He has represented the Manufacturer's Federation on the Executive Committee for Science of the Technicians Certification Authority for a number of years and recently chaired the Geology Syllabus Revision Committee for that body.

Sporting interests have included tramping, badminton and tennis and he is still active both as a participant and an administrator in the latter two.

Interest in both his hobby and his profession will be retained following his retirement from the brewing industry, by part time work in the field of pottery clays in Nelson, a district he has grown to know well and love over many years.

The Register

Changes approved by NZIC Council

The Register: The following changes were made at the November meeting of the Council:-

Fellows

Arcus, Alexander Colvin, M.Sc., Ph.D. (NZ) Medical Unit, Princess Margaret Hospital, Christchurch.

Baird, Walter Norman, B.Sc., Dip. Ind. Chem., Shirley Boys' High School, Christchurch.

Beck, John Richard, B.Sc., Lion Breweries Ltd, Wellington.

Campbell, Alistair Shand, M.Sc., (NZ), Ph.D. (Cantuar) Soil Science Dept, Lincoln College.

Futter, John Herbert, M.Sc., (NZ) Chemical Service Laboratories Ltd, Wellington.

Garside, John Herbert, B.Sc., Ph.D. (Lond) NZ Portland Cement Assn, Wellington.

Griffin, Donald John, M.Sc. (NZ) Central Institute of Technology, Heretaunga.

Halliburton, Gavin Jack, M.Sc., (NZ) ANZIM Waitaki-NZR, Islington, Canterbury.

Happer, Duncan Alan Robert, B.Sc. (Hons) Ph.D. (Cantuar) Chemistry Dept, Canterbury University.

Hughes, John Theodore, B.Sc., A.R.I.C., Chemistry Division, DSIR.

Keeley, Graeme Montague, B.Sc., Canterbury Frozen Meat Co. Ltd, Christchurch.

McCurdy, Alan Underwood, B.A., B.Sc., Christchurch Teachers' College.

McEwan, Murray James, M.Sc., Ph.D. (Cantuar), Chemistry Dept, Canterbury University.

Metcalf, Mary Glen, M.Sc., (NZ), Ph.D. (Otago). Medical Unit, Princess Margaret Hospital, Christchurch.

Metcalfe, Alan, B.Sc., (Hons), Ph.D. (Bristol). Chemistry Dept, Canterbury University.

Mills, John Rutherford, B.Sc., (Hons, Notts), Christ's College, Christchurch.

Mitchell, Thomas Alexander, M.Sc., (NZ) Wheat Research Institute, Christchurch.

O'Kane, John McDonald, M.Sc., (NZ), MIChemE, C.Eng., ICI Tasman Ltd, Upper Hutt.

Peet, Nelson John, B.Sc., (Hons. Edin.), Ph.D. (Cantuar) Dept of Chemical Engineering, Canterbury University.

Powell, Harry Kipton James, M.Sc., Ph.D. (Well.) Chemistry Dept, Canterbury University.

Richards, Peter Russell, M.Sc., (NZ), Riccarton High School, Christchurch.

Rodley, Gordon Allen, M.Sc., (NZ), Ph.D. (Lond.), Chemistry Dept, Canterbury University.

Smedley, Stuart Ivor, M.Sc. (Well.), Ph.D. (S'hamptn), Chemistry Dept, Victoria University.

Stedman, Brian Maurice, M.Sc., (NZ), Waitaki-NZR Ltd, Christchurch.

Stewart, Rex George, M.Sc. (NZ), MRIC. Wool Research Org. of NZ, Lincoln.

Stonyer, Cyril Lester Haste, B.Sc., ICI Tasman Ltd, Upper Hutt.

Stott, James Boothroyd, B.Sc. (Hons), Ph.D. (Leeds). Dept of Chemical Engineering, Canterbury University.

Tennant, William Craighead, M.Sc. (NZ), Ph.D. (NSW), Chemistry Division, DSIR Gracefield.

MEMBERS

Greenway, Anthony Michael, B.Sc. (Hons), Ph.D. (Cantuar), Chemistry Dept, Arizona University.

Keen, Roger Douglas, M.Sc. (Cantuar), NZ Industrial Gases Ltd, Lower Hutt.

Kirk, Christopher Matthew, M.Sc., Ph.D. (Cantuar), Christchurch Technical Institute.

Leong, Richard Choo-Keng, M.Sc. (Auck) NZ Co-op Dairy Co. Ltd, Hamilton.

McConnell, Stephen Robert, M.Sc. (Well), ARACI, NZFMRA, Auckland.

Melling, Peter James, B.Sc. (Hons Cantuar), Chemistry Dept, Victoria University.

Newth, Ronald Penrose, Ruakura Agric. Research Centre, Hamilton.

Novelle, Andrew John Scott, M.Sc. (Auck). Hellaby Shortland Ltd, Auckland.

Rodgers, Mrs Beryl Edith Winifred, B.Sc. (Hons. Shmptn), Central Institute of Technology, Upper Hutt.

Sissons, Christopher Hal, M.Sc. (Well), Ph.D. (Auck), Ruakura Agric. Research Centre, Hamilton.

GRADUATE MEMBERS

Miller, Christopher John, NZCS, Gamlen Chemical Co. NZ Ltd., Auckland.

Pamich, Ivan, NZCS, Formica NZ Ltd., Auckland.

Budhia, Dahya Makan, B.Sc. Chemistry Dept, Canterbury University.

Chung, Arthur, M.Sc. (Auck) Fruit Research Division, DSIR, Auckland.

Gibson, Jeffrey Raymond, B.Sc. (Hons. Cantuar), Chemistry Dept, Canterbury University.

Gray, Denys Andrew, B.Sc., Ministry of Works & Development, Dunedin.

Jones, Evan John, B.Sc., McWilliams Wines Ltd, Napier.

Lake, Robin John, B.Sc. (Hons. Cantuar), Chemistry Dept, Canterbury University.

Limmer, Alan Wayne, M.Sc. (Waik), Chemistry Dept, Waikato University.

Moroney, Lee Moala (Ms), M.Sc., D. Phil, Waikato University.

Thomson, Mrs Barbara Mary, B.Sc. (Hons. Cantuar). Chemistry Division, DSIR, Christchurch.

Weatherall, David Alex, M.Sc. (Auck), Potter Brown Ltd.

Yeo, Kheng Hong James, B.Sc., Chemistry Dept, Canterbury University.

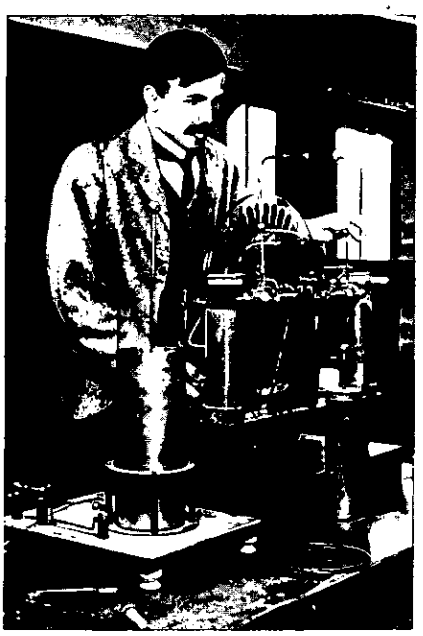
TECHNICIAN MEMBERS

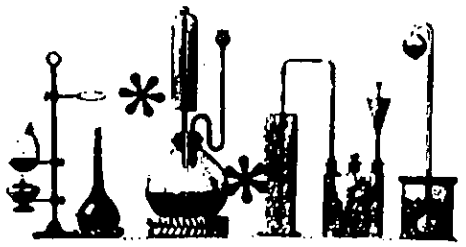
Hawke, David John, NZCS, Dept. of Chemistry, Biochemistry, Massey University.

Stratton, Malcolm Kenneth, NZCS, DB Central Brewery, Pahiatua.

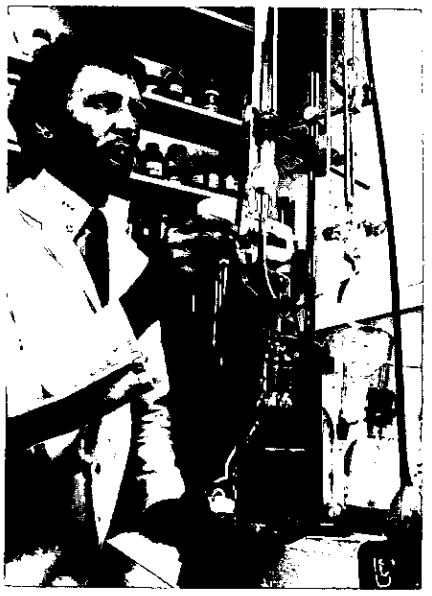
THANKS!

Appreciation of the NZIC's "very generous assistance" towards completion of "Rutherford's Den" (illustrated) has been acknowledged by Mr Brett Riley, general manager, Christchurch Arts Centre, where the exhibit is located. A plaque recording the Institute's role has been placed in the Den, which is proving a popular attraction.





New Products, Services



CONSTANT PRESSURE PUMPS

Constant pressure pumps for use with column packing-type HPLC separation are available from a Manurewa company.

The method by which columns are packed is critical as the packed column is used as a fine high-pressure strainer to break down the substance being analysed by the chromatograph.

Two basic methods are used: dry packing techniques use gravity to feed the packing into the column as a dry powder; wet (or slurry) packing requires a pump to feed the packing into the column as a liquid suspension.

The solvent drives the slurry down against a porous disc (frit) at the bottom of the column where the resistance builds up to about 7500-10,000 psi (although lower pressures can be specified). As the slurry 'packs' harder inside the column the solvent drips out of the end.

Haskel pumps, available through Air Driven Pumps (NZ) Ltd, are designed to pack columns and operate with most solvents used. A full range is offered, operating from as low as 3-4psi, together with fittings and accessories to provide a completed installation; kitset units are also available.

Pumps are said to pay for themselves quickly and it is noted that users have obtained better results by making columns and slurries to suit their own particular needs.

Replacement of an inefficient column with fresh packing is described as a minor expense once the initial investment has been made and gives the user who does his own greater control over performance.

C002 For further details, use Reader Service Card.

ANALYSIS AND FORMULATION SERVICE

Chemical product analyses, manufacturing formulations and costings are undertaken by a new firm of industrial chemical consultants and development chemists, W. Grayson & Associates Ltd, Auckland.

The company was established to provide the chemical industry with services not usually offered by purely analytical laboratories. These include: development of quality control standards; quality control services; problem solving in production chemistry; advice on supply of raw materials and plant; development of new products; redevelopment of existing products; reformulation of imported products for manufacture with locally available chemicals; management of contract manufacturing programmes.

Projects undertaken to date have involved such diverse products as aerosols, printing inks, agricultural chemicals, animal remedies, cleaning products, toys, adhesives and liquid fertilisers.

A recent typical report to a company proposing to make a substitute for an imported plastics adhesive included: analysis of the imported product contents; new product formulation and costings; production method; advice on production equipment and containers; a production sample; and corrosion test results.

Immediate turnround of urgent work is assured and the company's modern laboratory (illustrated) is conveniently located in Te Papapa.

C001 For further details, use Reader Service Card.

SWARF REMOVAL

Turno-Klean filters, manufactured by AMF Cuno, provide positive removal of all particles down to the rating on the particular filter. They are normally cleaned continuously by a geared motor drive when clearing heavy swarf loads. The swarf is drained from the filter sump. Turno-Klean filters are available from Niven Industries Ltd, Water and Waste Treatment Division.

C005 For further details, use Reader Service Card.

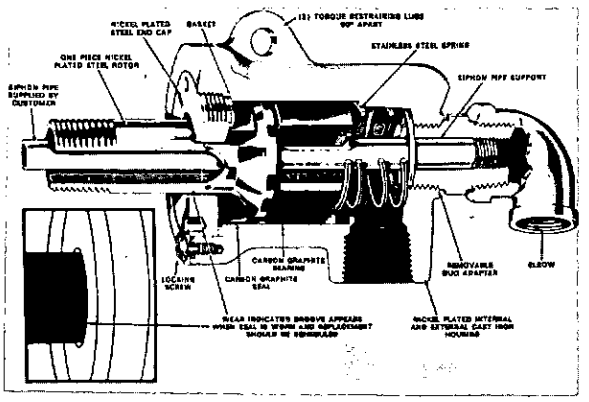
ROTATING UNION

A new high temperature, high pressure, non-lubricated, self supported model has recently been added to the range of American-manufactured Deublin rotating unions marketed in NZ.

This follows a recent visit by Mr Gary Deubler, vice-president, Deublin Co., Illinois, to meet executives of the local distributors, Kidd Garrett Ltd.

Rotating unions manufactured by Deublin, specialists in this equipment, are designed specially to handle fluids such as hot water and hot oil, as well as steam and air.

(A rotating union (illustrated) is a plumbing connection which unites a non-moving component with a rotating pipe carrying fluids or steam at high pressure. The union connects the two in a way that no leakages occur.)



The latest model of rotating union in the Deublin range is intended particularly for use in the paper converting and corrugating process industry, as well as for the pulp, paper and paperboard industries. The union is also particularly suited to the rubber, plastics, textile, chemical, marine and steel industries.

The union is nickel plated both internally and externally to resist corrosion and to increase service life. Rotors and threaded cap are also nickel plated; the coil spring is stainless steel.

A seal wear indicator shows when seal is worn and replacement can therefore be anticipated. That and other preventative maintenance keeps the union operating at a maximum.

Mr Deubler said he saw much potential in equipping plant for the food processing industry with rotating unions.

C003 For further details, use Reader Service Card.

For Your Diary

The 4th Energy Conference is to be held at Auckland University, May 17-19. It is being sponsored by the University, NZ Institution of Engineers, Ministry of Energy and the NZ Committee of the World Energy Conference. Further details are available from the University's Centre for Continuing Education, which is administering the meet.

"Setting The Standard" is the theme of the 5th Australian Symposium on Analytical Chemistry, to be held in Perth, WA., August 20-24.

Recent advance in all aspects of analytical chemistry will be covered. Scientists of international repute will deliver plenary lectures on Atomic Spectroscopy, Computers, Forensic Science, HPLC, ICP and Water Quality. There will also be 16 invited review lectures on a variety of instrumental techniques, safety, international and environmental standards.

The Australasian Corrosion Association's NZ branch is to hold a one-day symposium on "Corrosion Prevention in Industry" at Auckland University's School of Engineering on May 18, 1979.

The Auckland region contains NZ's heaviest concentration of industry and the symposium has been designed to look at problems facing industrial personnel as regards corrosion, and will possibly provide some solutions, or help people faced with a growing corrosion bill in their particular industry, and will outline the nature of the problems and modern preventative measures against corrosion. Ample time will be allowed for questions and discussion.

This symposium will be of interest to chemists, engineers, maintenance personnel and managers of firms and industries throughout NZ and, in fact, anyone who has an interest in modern techniques of prevention.

Further information is available from the Association, Box 5961, Auckland.

The 27th IUPAC congress is to be held at Finlandia Hall, Helsinki, Finland, August 27-31, 1979.

The scientific programme includes the following main sections and subsections: Trace Element Analysis; Modern Methods in Clinical Chemistry; Chemistry and Biology of Cell Membrane Carbohydrates; Chemistry and Technology of Natural Polymers and their Degradation Products; Biotechnology and Bioengineering; Mineral Resources in Northern Europe.

The Irrelevant Computer

The trade marks section of the British patent office is committed to a long term policy which involves computerising all its data as a first step towards mechanised searching for conflicting names. But it is to be hoped the system will work a little more efficiently than the one now in operation covering the Benelux countries. If the experience of the Woking firm Tyrell Ltd is anything to go by, the computer has a lot to learn from good old-fashioned human beings.

Tyrell licenses firms throughout the world to manufacture miniature models of racing cars — it therefore requires both design and trade mark registrations. An application to register the name Tyrrell was made in the Benelux countries and duly fed into the computer for the obligatory search. The computer obliged by disgorging a list of 190 previously registered marks which it regarded as possible anticipations of the word "Tyrrell". Among the phrases thrown up by the computer were such total irrelevancies as: Laurel & Hardy, Israel Tours, Barrel of Monkeys, Time to Retire and Mercedes-Benz Diesel. Human searching, as still practised in UK, would produce a search report without the irrelevancies [the computer obviously reported every name that included "tyr", "rell" etc] and would probably save much time and money for all concerned.

— "New Scientist"

New Products (Cont)

FOXBORO ANALYTICAL

W. Arthur Fisher Ltd has been named NZ representative of Foxboro Analytical, a division of the Foxboro company, manufacturers of quality process control instrumentation, which the company also represents.

To date, instrumentation in the Foxboro Analytical Division includes Wilks, a leading manufacturer of selective wavelength infrared analysers. Foxboro/Wilks can supply a complete analysis and control system — gaseous, liquid, or solid — enabling second-by-second information and, because abnormal operating conditions can be spotted and corrected under dynamic conditions, plant shutdown for loss of control is essentially eliminated.

Other companies coming under Foxboro Analytical control are Ferrograph, for analysis of wear in lubricated machinery, Balsbaugh, pH and conductivity monitoring, Analab and Arcas, for gas chromatography.

Foxboro/Arcas manufacture the Arcas model 505 process gas chromatograph now used worldwide in chlorine productions, natural gas plants, refineries and many other operating units of the chemical and petrochemical industries. Typical applications include the determination of hydrocarbons in stack gas, pollution monitoring and product purity analysis.

C004 For further details, use Reader Service Card.

LEVEL MEASURING INSTRUMENTS

A new range of level control instrumentation called 'Series 80' has recently been introduced by Foster Cambridge Ltd and available through Kent Instruments (NZ) Ltd, Auckland.

Many industries, at one time or another, need to know or control the level of a liquid, slurry, powder or granular solid in a variety of situations. The instruments are therefore of importance to public services such as water supply and sewage treatment and all industrial and process industries, from petrochemicals to food processing, cement manufacturing and storage, brewing and many others.

Over 25 years' experience in level measurement is built into the new Foster Cambridge designs. The new range uses three different measuring techniques — "Tektor" and "Telstor" for capacitance; "Noflote" for conductivity; and submersible instruments operated by pressure.

The result has been standardisation and simplification wherever possible, e.g. the number of different housings from which the user has to choose has been reduced from 20 to 4, yet the variables which can be obtained remain unchanged, leaving the freedom to build up a configuration suited to particular applications.

Because the measurements are made electrically, the systems do not have moving parts; as a result, the instrumentation is said to be robust, easy and inexpensive to install, needs no maintenance and is not subject to wear.

C006 For further details, use Reader Service Card.

CROSSWORD SOLUTION

Solution to Chemical Crossword, November, 1978, edition. (Mike will have another puzzle in the April issue).

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