



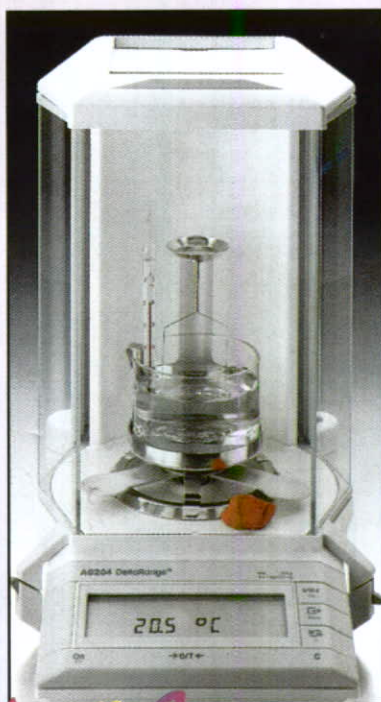
# Chemistry

IN NEW ZEALAND

ISSN 0010-5566

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For further details see the cover story on page 2



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## COMING UP ...

May 1995 - Focus on the Petroleum and  
Oil Industry

July 1995 - Focus on Nutrition, Pharmaceuticals  
and Cosmetics

#### Deadline for material:

5th of the month of publication

#### Contributions and enquiries to:

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### Calibration:

With the push of a button, the AG self calibrates using its built-in weight, ensuring accurate and reliable measuring results. The AG range also has "Varical", allowing calibration to a partial, but frequently used, weighing range. This allows a more accurate and reliable calibration for specific user requirements than just the usual single point calibration.

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# LETTER TO THE EDITOR

13.2.95

11 Wyn Street  
Christchurch 2

Dear Sir,

Alan Mackney misrepresents me on two matters in his letter in your January issue. The matter of past and present performance is not nearly as simple as it is made out to be and cannot be analysed properly in a letter. This is what Bill Denny's committee will do.

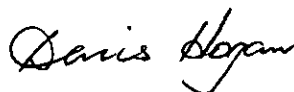
On the question of professionalism I did not, as Alan "deplores", suggest that NZIC might cease its endeavour to be fully professional. If he reads my letter carefully he will see that I suggested abandoning entry qualifications as a "topic for discussion". I did not express an opinion. In fact my personal view is strongly in favour of a fully professional entry qualification to corporate membership.

May I remind Alan that it was he who in the 1980s led the Institute into adopting rule

changes that I argued lowered this standard. He will remember how hard a group of us fought in opposing these changes. However we lost this battle and we live with the consequences. I find it supremely ironic, after the sometimes very bitter battle of the 1980s, to be cast as the one suggesting lowering the standard of the Institute's professionalism. Having taken the first step down that road with the 1987 rule changes it will be very difficult to go back. However, if Alan is concerned to return the Institute to a fully professional level of entry I offer my full support.

In the mean time while the Institute is under the presidential microscope I see nothing to be deplored in discussing other options.

Yours sincerely



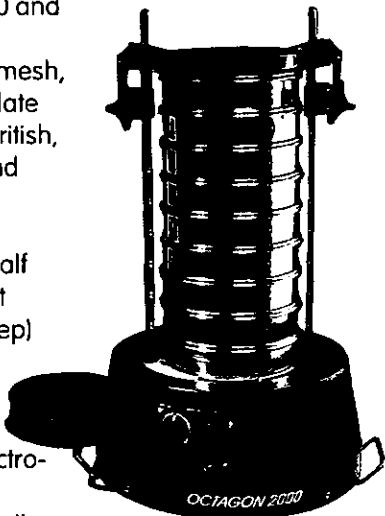
Denis Hogan - Hon. Fellow

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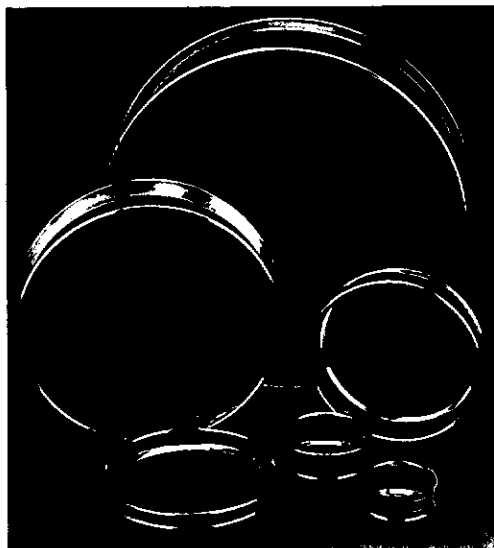
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# LOCAL NEWS

## NZ Women Scientist Wins Important American Dairy Science Award

Dr Audrey Jarvis of the Biological Science Section, New Zealand Dairy Research Institute, has been selected as the 1995 recipient of the Marschall Rhône-Poulenc International Dairy Science Award. This award is presented annually by the American Dairy Science Association and is the most competitive of the American Dairy Science awards. Audrey's selection is in recognition of her research on bacteriophage. Bacteriophage are viruses that attack the starter cultures used in cheese-making and are of major importance to the dairy industry.

Audrey will travel to the ADSA meeting in Cornell, New York State USA in June 1995 to receive the award and present an invited lecture. The award is given to a scientist outside the USA for the contribution to dairy science.



*Audrey Jarvis speaking at the recent International Dairy Lactic Acid Bacteria Conference.*

Audrey is the first woman to have won this award and it is fitting that she heard the news during the International Dairy Lactic Acid Bacteria Conference held in Palmerston North recently, particularly as she was secretary of the Organizing Committee. The 190 delegates from 25 countries were delighted to have the opportunity to congratulate Audrey personally.

Two other NZDRI staff, Dr Bob Lawrence and Dr Lawrie Creamer, were previous recipients of this award.

### Overseas Scientists Sponsored by European Commission

Eleven young scientists, one from each of the European Countries, recently spent five days at the International Dairy Lactic Acid Bacteria Conference in Palmerston North. These scientists were sponsored by the Biotech Committee of the European Commission.



*Front row (L to R): John Smart (New Zealand), Audrey Jarvis (New Zealand), Silvano Gregoli (Counsellor, Science and Technology European Union).*

*Back Row (L to R): Monica Herrero (Spain), Maria Luisa Callegari (Italy), Sonja Lick (Germany), Dionyssia Stefanitsi (Greece), Mads Johnsen (Denmark), Bruno Pot (Belgium), Tracey Eaton (United Kingdom), Denis Twomey (Ireland), Patrick Duwat (France), Dag Lillehaug (Norway, absent).*

The scale of the European sponsorship was made possible thanks to the support of the new Commissioner for Science, Research and Development, former French Prime Minister Edith Cresson. It also reflects the EC trend to boost Science and Technology collaboration in third world countries wherever there is mutual benefit and the research is aimed at an improvement in the quality of life.

### A.i. Scientific - New Zealand Office

A.i. Scientific is pleased to announce the opening of a New Zealand office to support existing customers and users of A.i. Scientific products. Key products include the AIM500 automated block digestion systems, the AIM1250 autosamplers, CEM microwave systems, Alitea pumps and tubing, Kipp & Zonen recorders and solar products plus Claind gas generators. For the petrochemical industries, A.i. Scientific also supply AC Controls, Phase Technology and Grabner Instruments products.

A.i. Scientific has appointed Kevin Moloney as New Zealand Sales Manager. Kevin has an extensive background in laboratory instrumentation. In addition to expanding A.i. Scientific's customer base, Kevin will also be responsible for establishing a support and service network throughout New Zealand.

For further details contact:  
Kevin Moloney  
New Zealand Sales Manager  
A.i. Scientific  
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## BAYER PROVIDES SPONSORSHIP WORTH \$33,500 TO AID SCHOOL SCIENCE

For the third year running, Bayer New Zealand Ltd has shown its commitment to supporting science education by providing sponsorship awards worth a total of \$33,500 to ten schools drawn from around the country.

Each of the schools will receive \$2,500 worth of scientific equipment of its choice, as well as Agfa photographic products worth \$200 and other educational aids valued at \$650.

The schools have also won the right to nominate a student for one of five Bayer scholarships worth \$4,500 each over three years for students pursuing University science studies.

The ten winning schools were chosen during December 1994 by a panel of senior lecturers from Colleges of Education throughout New Zealand.

*The winning schools are:*

Northland College	-	Kaikohe
Penrose High School	-	Penrose
Wairoa College	-	Wairoa
Lincoln High School	-	Canterbury
Nayland College	-	Stoke, Nelson
Papakura College	-	Papakura
Fairfield College	-	Hamilton
Heretaunga College	-	Upper Hutt
Linwood High School	-	Christchurch
Cromwell College	-	Cromwell

Bayer New Zealand's Managing Director, David Hope-Cross, says that each of the winning schools has demonstrated dedication and enthusiasm for the future of science education in this country. "We are proud to be associated with science education and New Zealand's young scientists," he says.

## UNIVERSITY FUNDING CUTS UNJUSTIFIED

The annual report of the University Vice-Chancellors' Committee says the Government is unjustified in cutting University funding further after the improvements in efficiencies Universities have made in recent years.

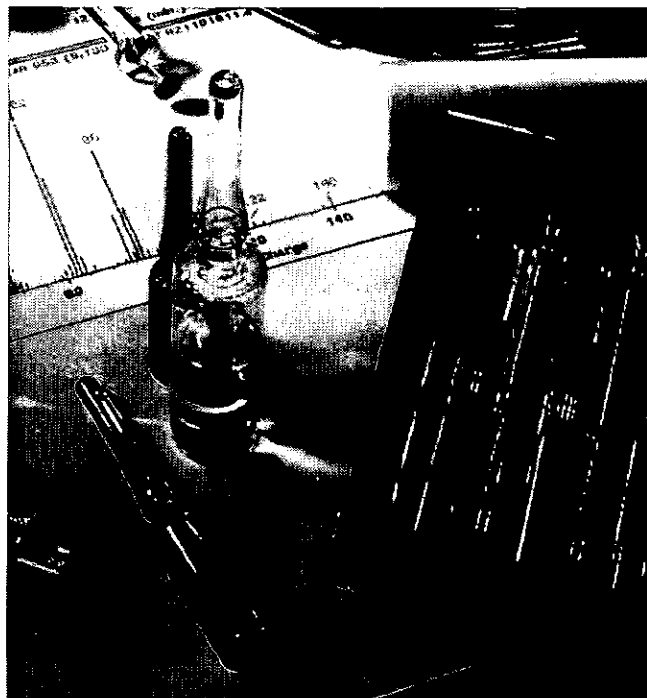
Last August, the Minister of Education cut the amount of funding per student and earlier this year announced further progressive cuts through to 1999.

Significant funding cuts have been made to law and dentistry course funding, and costs such as earthquake cover, ACC and superannuation levies and upgrading of buildings to comply with the Health and Safety Act have put added financial burden on Universities. A further levy of around \$15 million will fall on Universities probably during the next twelve months when a new accounting system which will charge tertiary institutions based on their assets comes into effect.

The planned funding cuts are intended to create more student places and ease the pressure on polytechnics but less money will increase the pressure on current university staff, equipment and resources.

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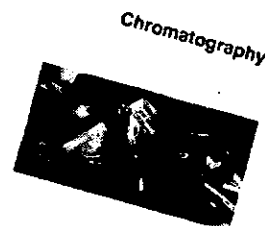
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# DETERIORATION OF FRYING FAT IN A SIMULATED FAST FOOD OPERATION

by Neil Fraser, Richard Beyer and John Birch  
Food Science Applied Research Unit, University of Otago, P O Box 56, Dunedin

## Abstract

Soft beef fat is the most commonly used frying medium in New Zealand fast food and "take-away" food operations. Currently, the quality of the oil is regulated by its peroxide value. We have confirmed the observations of previous workers that levels rise in the early stages of simulated repeat frying but fall at later stages. Therefore oil/fat with apparently satisfactory peroxide levels (less than 10 meq of peroxide oxygen kg<sup>-1</sup> fat) may have undergone substantial damage. None of the commonly used antioxidants offer very much protection for the fat during repeat frying but butylated hydroxy toluene (BHT) reduced peroxide formation to a greater extent than the other antioxidants tested - butylated hydroxy anisole (BHA) and propyl gallate (PG). Predictably, there was a greater loss of unsaturated fatty acids than saturated fatty acids during the cyclical heating programme.

Although there was a substantial loss (30-fold) of linolenic acid during repeat frying, initial values were low (~1%). Linolenic acid losses do not therefore contribute significantly to the change in P:S ratio after repeat frying.

## Introduction

Deep-fat frying of food is very common in fast food and "take-away" food operations. The cost of the frying medium contributes significantly to the total production cost of the finished products which means that operators commonly reuse oil or fat. Furthermore, the oil is frequently kept hot for long periods of time usually in the presence of oxygen and moisture. As a result, hydrolysis and oxidation occurs (Carlin *et al.*, 1954; Beuerlen *et al.*, 1986; Roth and Rock, 1972;) causing not only the development of "off flavours" (Clark and Serbia, 1991) but also the development of potentially hazardous oxidation products (Alexander, 1978; Nolen, 1973; Clark and Serbia, 1991).

Deterioration can be delayed by good manufacturing practices and by the addition of antioxidants. For instance, products can be dewatered before frying, oils and fats can be filtered after each heating cycle. Adding fresh fat to ageing fat can act as a diluent but deterioration is autocatalytic so that the fresh fat may deteriorate unnecessarily rapidly. Furthermore a "topping up" regimen may mean the oil/fat is rarely completely replaced and fryers may be completely cleaned only rarely.

Antioxidants may be added to oil/fat which serve to protect it during storage as reviewed by Frankel (1991). Antioxidants are commonly phenolic compounds which act as proton donors thereby suppressing initial free radical propagation and subsequent peroxide formation. Antioxidants will delay autoxidation, they will not prevent it (Warner *et al.*, 1970; De Fouw *et al.*, 1981). Peroxide values however, continue to be used in New Zealand as a measure of the extent to which oxidation has occurred (New Zealand Food Regulations, 1984). In fast food operations it is possible that damaged oils and fats

many still conform to this standard in which cases the regulatory authorities are powerless to act.

Unfortunately most commonly used antioxidants are destroyed or lost by steam distillation in repeat frying operations. The present study is a comparison of the rate of peroxide formation in soft beef fat with the antioxidants butylated hydroxy anisole (BHA), butylated hydroxytoluene (BHT) and propyl gallate (PG) added under simulated commercial frying conditions. During the course of repeat frying, samples were collected and methyl esters of fatty acids were separated by gas chromatography to assess relative survival of fatty acids.

## MATERIALS AND METHODS

### The Samples

Soft beef fat was chosen as the frying medium since this is most commonly used in New Zealand. Although fats with very low peroxide values are available, they are expensive and would not be used in commercial practise. All experiments were conducted on fat samples obtained directly from the fat supplier. The maximum permitted quantity of each antioxidant (100 ppm: New Zealand Food Regulations, 1984) was added to the melted fat to ensure even distribution. The temperature did not exceed 100°C during addition. The control was treated in an identical manner but no antioxidant was added.

### Deep Fat Frying

The deep fat frying procedure was a modification of the regimen described by DeFouw *et al.* (1981). A Zip deep fat fryer equipped with a wire basket was used for frying frozen commercial french fried potatoes (stored ~ -15°C). The fat (1400 g) was heated to 180°C ± 5°C and a 10% (w/w) sample of french fried potatoes was added and fried for 4.5 minutes. Each fat was subjected to 20 consecutive fryings. No attempt was made to replenish lost oil but the proportion of the added french fries was maintained at 10% (w/w). For each repeat frying the fat was allowed to reheat to 180°C (± 5°C) after addition of the frozen chips. One frying cycle thus lasted ~ 2 h. This was repeated four times per day. Both fat and fryers were stored at ambient temperature when not in use. Fat was filtered through muslin after 8 h and 24 h.

### Peroxide Analysis

The method of Egan *et al.* (1981) was chosen as the most appropriate method of peroxide determination since this is the method used by the regulatory authorities. Samples were analysed after 0, 0.5, 4.5, 12, 18, 24, 30, 36 and 40 h. Each sample was assayed in triplicate since titration end-points were difficult to standardise.

### Fat Analyses

Fat samples (0.5 g) were heated at 65°C for 10 minutes with methanolic potassium hydroxide (4 mL, 6% w/v) in a Kimax screw-cap culture tube. After cooling, non acids material was

removed by partitioning with diethyl ether. The remaining acid salts were acidified and the resulting free acids were removed by partitioning with further aliquots of diethyl ether. Methyl esters were prepared using boron trifluoride/methanol (4 mL) by heating for a further 10 minutes. The ether soluble fraction was transferred to a sample vial. The esters were separated using a Perkin-Elmer Model 8410 flame ionisation gas chromatograph fitted with an FFAP capillary column (30 m x 0.54 mm I.D.) containing 3% (film thickness 1.2  $\mu$ ) carbowax on gaschrom Q 100-120 mesh. Injection was made using a split mode (ratio 100:1) with nitrogen make up gas at 30 mL  $\text{min}^{-1}$ . Fatty acid methyl esters were identified by chromatography with known standards.

## RESULTS AND DISCUSSIONS

### Peroxide Values

The peroxide values obtained from the fat samples are shown in Table 1 and illustrated in Figure 1.

The results show that peroxide values rise rapidly during the early stages of use and subsequently drop as autoxidation proceeds. With continued heating the peroxide value for all the samples except that containing propyl gallate fell below 10 meq peroxide oxygen  $\text{kg}^{-1}$  fat. Our result questions the value of the maximum permitted value of 10 meq peroxide oxygen  $\text{kg}^{-1}$ . This specification is very likely to be exceeded by only mild heat abuse. Further more, we confirmed the results of previous workers (Perkins, 1967, and Roth and Rock, 1972) that peroxide levels drop after prolonged heating as peroxides undergo further reactions in the degradation process. Therefore, fats that have suffered extensive heat damage may continue to conform to this specification.

At 0.5 and 4.5 h levels of peroxide in the fat protected by BHT were significantly lower than the others. In the remaining fats (unprotected fat, BHA-fat and PG-fat), there was no significant difference in the maximum peroxide values reached nor rate at which they formed.

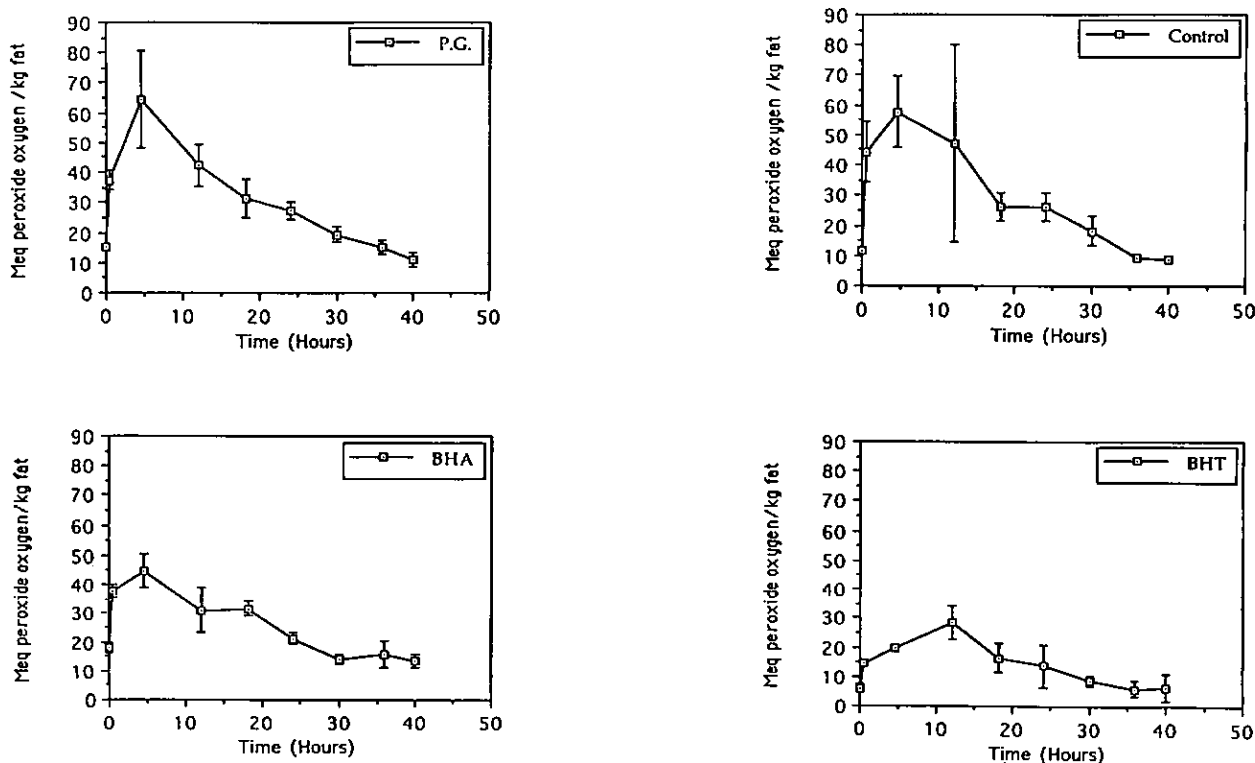
Table 1: Peroxide determinations from fat samples<sup>1</sup>

Time (Hours)	Fat Treatment*			
	Control	BHA	BHT	Propyl Gallate
0	11.49 $\pm$ 1.03	17.96 $\pm$ 1.74	5.96 $\pm$ 1.42	15.06 $\pm$ 0.52
0.5	44.23 $\pm$ 10.07	37.68 $\pm$ 2.27	14.70 $\pm$ 0.32	37.22 $\pm$ 3.24
4.5	57.77 $\pm$ 11.75	44.66 $\pm$ 6.01	19.67 $\pm$ 1.06	64.44 $\pm$ 16.13
12	47.14 $\pm$ 32.87	31.02 $\pm$ 7.89	28.57 $\pm$ 5.96	42.37 $\pm$ 6.77
18	26.14 $\pm$ 4.42	31.64 $\pm$ 2.62	16.50 $\pm$ 4.99	31.43 $\pm$ 6.51
24	25.91 $\pm$ 4.71	20.95 $\pm$ 2.04	13.80 $\pm$ 7.18	27.34 $\pm$ 2.67
30	18.10 $\pm$ 4.90	13.90 $\pm$ 1.98	8.96 $\pm$ 1.67	19.45 $\pm$ 2.58
36	9.26 $\pm$ 1.08	15.80 $\pm$ 4.71	5.97 $\pm$ 2.60	15.16 $\pm$ 2.31
40	8.74 $\pm$ 1.19	13.37 $\pm$ 2.32	6.52 $\pm$ 4.74	11.09 $\pm$ 2.09

<sup>1</sup> Average of three determinations

\* meq peroxide oxygen per kg fat

Figure 1: Peroxide Values from Fat Samples



## Fatty Acid Profiles

Since BHT seemed to perform better than the other antioxidants, we limited our investigation of changes to fatty acid composition during heating to BHT-protected fat and used unprotected fat as a control.

Fatty acid profiles determined by GLC are shown in Table 2. The ratio of saturated fatty acids C16:0 to C18:0 and C14:0 to C18:0 and C16:0 to C14:0 remain constant during heating (Figure 2). There was an apparent increase in the saturated fatty acid content of both the BHT protected fat and the control but this is misleading and occurs because of losses of more labile unsaturated fatty acids. Relatively, C18:1 levels fell slightly during repeat frying. Relative linoleic acid levels fell by 64% after 40 h, and no significant difference was observed between the BHT-protected and the control samples as shown in Figure 2.

## Visual Assessment

As frying progressed, foaming increased and the oil darkened in colour (Figures 3 and 4). These criteria are commonly used by fast food outlets to assess oil quality.

## Conclusions

Peroxide values rise in the early stages of heat abuse but fall as heating continues. They are not, therefore, a good index of oil quality. Yoon *et al.*, (1985) have suggested that dielectric constant, refractive index or polymer content may be more useful physical indices.

The saturated fatty acids withstand continuous heating slightly better than monounsaturated and significantly better than polyunsaturated fatty acids. There was no significant difference in heating losses between the saturated fatty acids.

Repeat frying operations is commonly practiced amongst fast food outlets. Under such circumstances, BHT apparently offers the most protection for soft beef fat but only in the early stages.

**Table 2: Fatty Acid Concentrations in BHT - Protected Fat and Unprotected Fat (Control)**

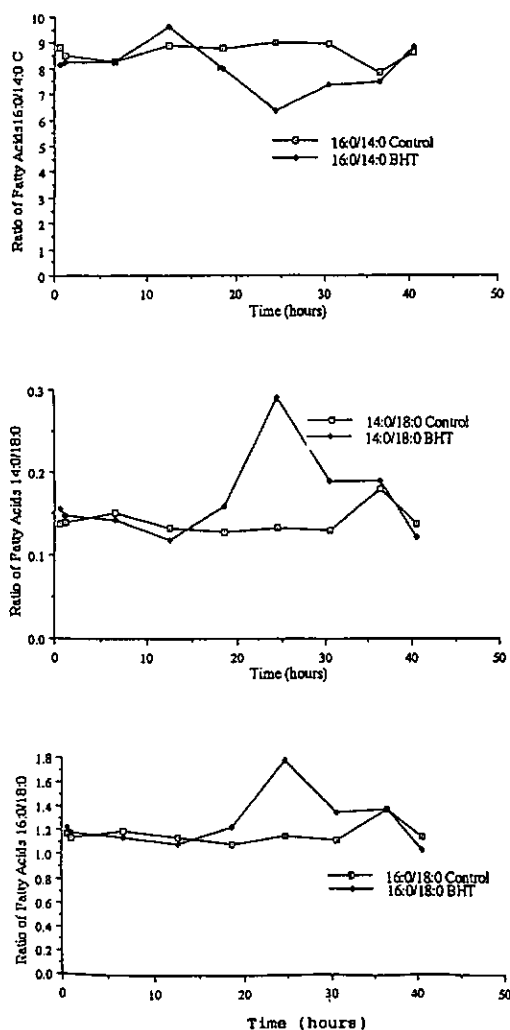
Fatty Acid*	Sampling Time (hours) - BHT								
	0	0.5	6	12	18	24	30	36	40
12:0	0.0842	0.0723	0.0994	0.0599	0.0734	0.1327	0.1502	0.0970	0.1174
14:0	3.1125	2.9320	2.8808	2.6915	3.0836	4.4576	3.9420	3.9837	3.0833
15:0	0.6282	0.6083	0.6072	0.6158	0.6522	0.8421	0.7969	0.1847	0.6794
16:0	24.649	23.626	23.235	25.198	24.044	27.286	28.286	28.941	26.500
16:1	2.2594	2.3268	2.2803	1.9940	2.3261	2.8342	2.2944	2.3841	1.8572
17:0	1.1237	1.1178	1.1343	1.2962	1.1401	1.0827	1.2875	1.3117	1.3710
17:1	0.6158	0.6152	0.6224	0.5778	0.6537	0.6782	0.5973	0.6008	0.5226
18:0	20.941	20.725	21.277	24.196	20.378	15.692	21.685	21.825	26.734
18:1	36.129	36.117	36.812	34.934	38.234	32.794	33.269	31.797	32.200
18:2	5.3266	5.4404	5.3771	3.823	3.7531	2.9115	2.1245	2.3385	1.9005
18:3	0.9981	1.1211	0.8546	0.3728	0.3347	0.2082	0.1138	0.0843	0.0522

\* % composition as the methyl ester

Fatty Acid*	Sampling Time (hours) - Control								
	0	0.5	6	12	18	24	30	36	40
12:0	0.283	0.0365	0.0725	0.0470	0.0913	0.0865	0.0648	0.1050	0.0982
14:0	2.8487	2.8611	3.0998	3.0207	2.9738	3.1351	3.0497	4.0472	3.3870
15:0	0.6073	0.6011	0.6465	0.6451	0.6402	0.6887	0.6665	0.8386	0.7355
16:0	24.473	23.690	24.977	26.083	25.404	27.443	26.689	30.950	28.533
16:1	2.1060	2.1657	2.2525	2.0130	2.0426	1.9344	1.9263	2.1590	1.8538
17:0	1.142	1.1247	1.1784	1.2735	1.2667	1.3311	1.3046	1.3853	1.3749
17:1	0.5719	0.5834	0.5995	0.5570	0.5667	0.5373	0.5368	0.5616	0.5166
18:0	21.783	21.610	21.713	23.897	24.524	24.939	24.957	23.428	25.860
18:1	35.962	35.844	35.205	33.784	34.636	32.476	32.813	30.160	30.950
18:2	5.2016	5.4889	5.0591	3.5142	3.0104	2.5747	2.4630	1.9067	1.7299
18:3	0.8832	0.9859	0.7279	0.3889	0.2704	0.1960	0.0913	0.0551	0.0352

\* %composition as the methyl ester

**Figure 2: Saturated Fatty Acid Ratios BHT - Protected Fat and Unprotected Fat (Control)**



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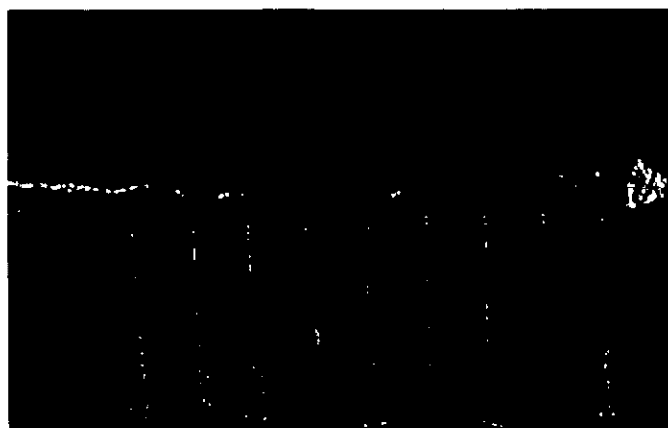
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**Figure 3: Progressive Darkening of Fat Samples**



**Figure 4: Increased Foaming as Usage Progresses**



**References**

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# ENVIRONMENTAL ISSUES



## LETTER FROM AUSTRALIA

I spent some time at the end of 1994 in public consultations over waste materials which contain polychlorinated biphenyls (PCBs). These substances were used for a few decades in the electricity industry in Australia where they found use as insulating dielectric fluids in transformers and capacitors. Up to 20,000 tonne was imported to Australia - we are not exactly sure how much - but manufacture (never undertaken in Australia) and importation were banned a few years ago. Because of their stability, particularly towards oxidation at high temperatures, PCBs were widely used in electrical apparatus and most of the remaining quantities we know about are held by companies and electricity generating authorities.

There remains the task of destroying any PCBs which remain, and a plan to manage this process is being drawn up by the Scheduled Wastes Management Group (SWMG) which is made up of state and territory EPA managers working under the auspices of the Council of Australia and New Zealand Environment and Conservation Ministers (ANZECC). Both the managers and ANZECC receive advice from a National Advisory Body on which there are representatives of industry, conservation groups and others with interests in this area. I chair both of the SWMG and the NAB which are, unlike the council of ministers, devoid of New Zealand representation.

The reason for our concern over PCBs is the effect they have been shown to have on human health and on the environment, most evidently in reproduction in marine animals. PCBs are persistent and - because of their fat solubility - accumulative. Detailed figures are not available for Australia, but it is possible that most of us carry a small body burden without showing any overt health effects.

In other countries PCBs are among the substances which are destroyed in high temperature incinerators, but we are unlikely to see one of these in Australia because of the outrage resulting from earlier proposals to build one. Environment Minister Ros Kelly resigned from parliament in January this year, but she banned export of PCBs in 1991 and as a result there are some interesting examples of other treatment processes which have been developed in Australia. The use of plasmas to 'burn' the PCBs and other chloro-organics has attracted some attention as has the use of cement kilns for oils which contain low-levels of these substances. A plasma system developed by Australia's government research organisation CSIRO, and a private company is used to destroy halons and other species under laboratory conditions. A larger model has been installed at a chemical plant where it chews up the leftovers from a phenol chlorination plant, mostly chlorophenols with some of the inevitable dibenzodioxins and dibenzofurans. Treatment with lime in a ball-mill also seems to destroy PCBs, and there are possibilities for molten salt baths and for bioremediation.

Another class of processes relies on reductive dechlorination by catalytic hydrogenation or by hydrogen transfer from paraffin

oil in the presence of strong alkali. The Canadian process EcoLogic is operating in Perth, with hydrogen gas at high temperatures (over 800 °C) breaking up complex chloro-organics and forming methane and HCl. CSIRO is also involved in the development of a catalytic hydrogenation process which operates at lower temperatures and restores the oil to fitness for use in electrical applications. The base catalysed dechlorination process, invented by the USEPA, is operated under licence by companies in Brisbane and Melbourne, and has already resulted in the hydro-dechlorination of several hundred tonnes of PCB materials.

Nearly two hundred people accepted the invitation of the National Advisory Body to join in the public consultation process in state capitals and other centres, and we received some forthright advice about our draft management plan. Some of this concerned the difficulty of regulating action at local level even though the states and territories may have reached agreement among themselves and with the Commonwealth. An interesting side effect of the consultations we held was that people were anxious to tell us about stocks of PCBs that had not been listed on national and state inventories. No doubt there is more of this sort to be learned. Clearly the most contentious item on our agenda was the proposed threshold concentration above which treatment of the PCB waste would be mandated. A well-prepared submission from the electricity industry argued for 50 mg/kg; but environment groups pressed for something more like 2 mg/kg or even less. A possible compromise would be a three-tier system consisting of non-PCB (content below, say, 2 mg/kg), PCB wastes to be handled according to state and territory laws (2 to, say, 50 mg/kg), and higher concentrations being designated as scheduled wastes and attracting stringent regulation.

The draft management plan for PCBs will now be revised and there will be a further round of consultation with a view to completing it by mid 1995. Any one who would like to have a copy of the draft plan may do so by writing to me at the address below, or using electronic mail to [ian=rae@vut.deu.au](mailto:ian=rae@vut.deu.au). Further tasks ahead of our committees include management plans for hexachlorobenzene - mainly a by-product of ICI Australia's chlorinated solvent production in the past - and for organo-chlorine pesticides. There is plenty to do, but it's a good cause.

Wish us luck!

Professor Ian D Rae  
Deputy Vice-Chancellor  
Victoria University of Technology  
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# CONFERENCES & SEMINARS

3-5 May, 1995 **3rd Australasian Symposium on Applied ICP - Mass Spectrometry**

Venue: Wallaceville House, Upper Hutt, New Zealand  
Contact: Terry Manning or Pam Coulson  
ESR  
P O Box 12-444  
Wellington  
New Zealand  
Ph: +64-4-4990540  
Fax: +64-4-4990541

22-26 May, 1995 **6th Asian Chemical Congress**

Venue: Metro, Manila, Phillipines  
Contact: Dr Ishmael Ordoñez  
Chairman  
Media and Promotions Committee  
6th Asian Chemical Congress  
Fax +63-2-996868  
or Alan Turner  
General Secretary/Executive Officer NZIC  
P O Box 12-347, Wellington  
Ph (04) 473-9444  
Fax (04) 473-2324

22-26 May, 1995 **11th Philippine Chemistry Congress**

Venue: Manila, Philippines  
Contact: 6ACC'95 Secretariat  
c/- Philippine Federation of Chemistry Societies  
U.P. NSRI Building, Diliman, Quezon City  
1101 Philippines  
Ph +63-2-945736  
Fax +63-2-996868

9-14 July, 1995 **13AC/4EC**

Venue: Darwin, Australia  
Contact: David Parry  
NTU  
P O Box 40146, Casuarina  
NT 0811, Australia  
Ph +61-89-466 666  
Fax +61-89-466 712

10-13 July, 1995 **5th COMTOX Symposium on Toxicology and Clinical Chemistry of Metals**

Venue: Vancouver, Canada  
Contact: F. William Sunderman Jr.  
Dept Laboratory Medicine and Pharmacology  
University of Connecticut Medical School  
P O Box 1292, Farmington  
Connecticut 06034-1292, USA

16-21 July, 1995 **ISMAR 95 "The next 50 years"**

Venue: Sydney, NSW, Australia  
Contact: Dr Les Field  
Chairman ISMAR - 95  
Department of Organic Chemistry  
The University of Sydney  
NSW 2006, Australia  
Ph +61-2-692 2060  
Fax +61-2-692 3329  
e-mail: ismar-95@biochem.su.oz.au

24-27 July, 1995 **14th International Chemistry Symposium**

Venue: Cambridge, United Kingdom  
Contact: Dr John F Gibson  
Secretary (Scientific)  
The Royal Society of Chemistry  
Burlington House, London W1V 0BN  
Ph +44-71-437 8656  
Fax +44-71-437 8883

11 August, 1995 **"Advances in Polymers II - Polymers in Communications and Electronics"**

Venue: CSIRO Division of Chemicals and Polymers  
Bayview Ave, Clayton, Victoria, Australia  
Contact: Bronwyn Fox  
Ph: +61-3-5438160  
Fax: +61-3-5438160  
or Marcus Zipper  
Ph: 61-3-9054935  
Fax: 61-3-9054940

14-19 August, 1995 **35th IUPAC Congress**

Venue: Istanbul, Turkey  
Contact: Professor A R Berkem  
35th IUPAC Congress  
Halaskargazi Cad.No:53, D.8  
80230 Harbiye, Istanbul, Turkey  
Ph +90-212-2407331  
Fax +90-212-2317037

27 August-1 September, 1995 **10th International Conference on Fourier Transform Spectroscopy**

Venue: Budapest, Hungary  
Contact: Mrs Klára Láng *or*  
Mr Attila Varga  
Conference Office  
Roland Eötvös Physical Society  
H-1371 Budapest, P O Box 433  
Hungary  
Ph/Fax +36-1-2018682

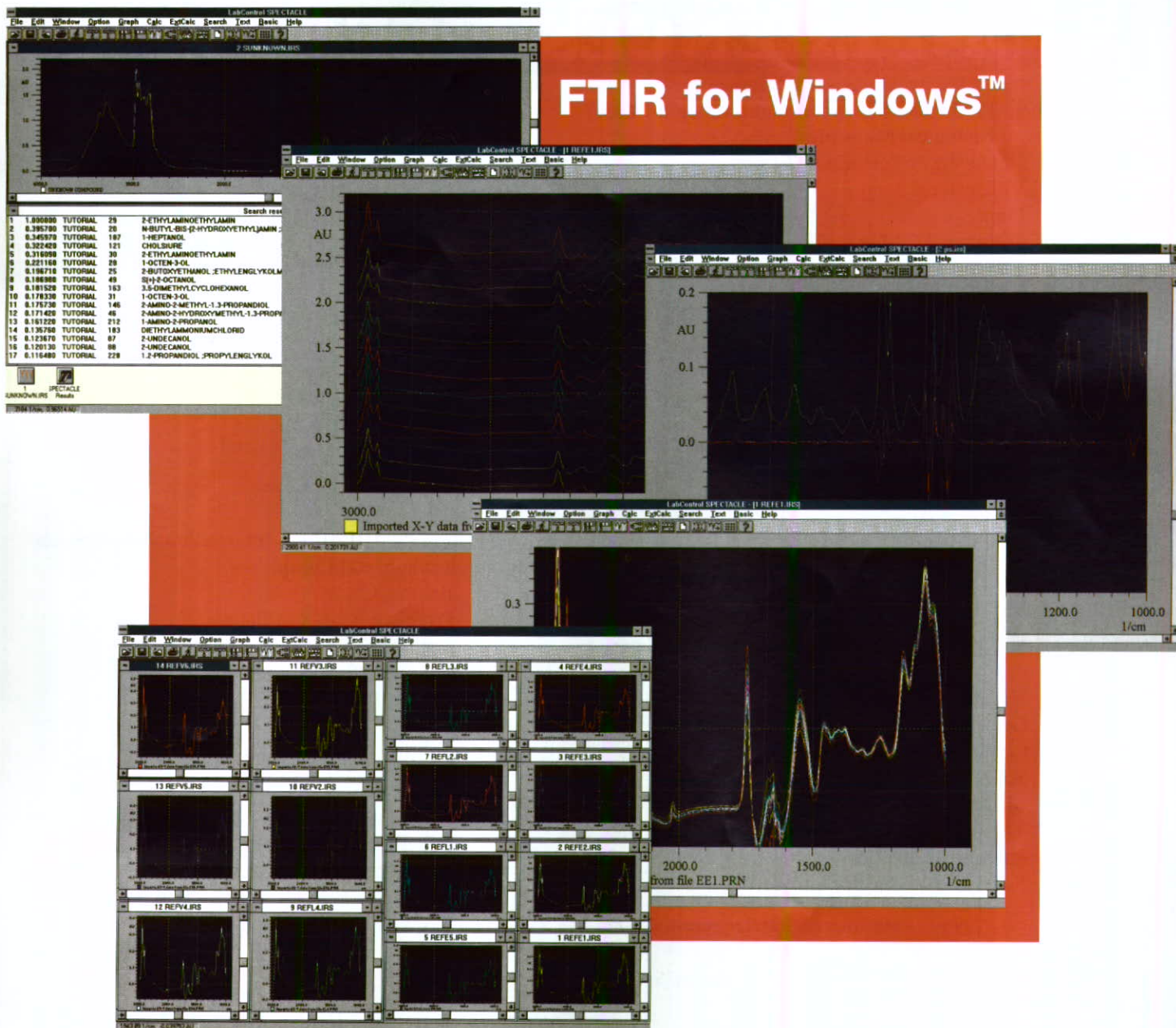
September, 1995 **Best-Best Engineering Science Technology Exposition**

Venue: Melbourne, Victoria, Australia  
Contact: Best Secretariat  
Oliver Scofield/Michael Clohesy  
P O Box 191, Hawksburn  
VIC 3142, Australia  
Ph +61-3-804 3844  
Fax +61-3-804 3855

3-5 September, 1995 **NSW Southern Highlands Conference on Heterocyclic Chemistry**

Venue: Milton Park, Bowral, NSW, Australia  
Contact: Professor David St. C. Black  
School of Chemistry, University of NSW  
Sydney, NSW 2052, Australia  
Fax +61-2-6622835  
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## 3-8 September, 1995 6th European Conference on the Spectroscopy of Biological Molecules

Venue: Universite de Sciences et Technologies de Lille  
Villeneuve d'Ascq  
France

Contact: Professor J C Martin (Chairman)  
ECSBM '95, LASIR, UST Lille  
Bât. C5  
59655 Villeneuve d'Ascq, Cedex, France  
Ph +33-204-36988  
Fax +33-204-36755  
Email: ECSBM95@univ-lille1.fr +33-20434920

## 5-8 September, 1995 WATER SOLUTIONS - AT WHAT COST?: The 1995 Annual Conference and Trade Display of the New Zealand Water & Wastes Association

Venue: Auckland, New Zealand  
Contact: David Ogilvie  
P O Box 2009, Auckland, New Zealand  
Fax +64-9-3761980

## 10-15 September, 1995 45th RACI Cereal Chemistry Conference

Venue: Adelaide, South Australia  
Contact: Geoffrey Palmer  
Conference Chairman  
S.A. Research and Development Institute  
21 Divett Place, Adelaide  
SA 5000, Australia  
Ph +61-8-226 7730  
Fax +61-8-226 7722

## 10-15 September, 1995 8th International Symposium on Marine Natural Products

Venue: Santa Cruz de Tenerife, Canary Islands, Spain  
Contact: Professor J D Martin  
Instituto Universitario de Bio-Organica  
38206 La Laguna, Tenerife, Canary Islands  
Spain

## 27 September-1 October, 1995 12th Medicinal and Agricultural Chemical Division Conference

Venue: Adelaide, South Australia  
Contact: Dr Michael L West  
Centre for Drug Design and Development  
University of Queensland  
Brisbane, QLD 4072, Australia  
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Fax +61-7-365 1990

## 27 September-2 October, 1995 10 NC

Venue: Adelaide, South Australia  
Contact: Des Williams  
10NC Organising Committee  
GPO Box 1906, Adelaide  
SA 5001, Australia

## 14-16 October, 1995 International Society of Magnetic Resonance Conference

Venue: University of Sydney  
Sydney, New South Wales, Australia  
Contact: Dr L Field

Department of Organic Chemistry  
University of Sydney  
Sydney, NSW 2006, Australia  
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## 16-18 October, 1995 6th New Zealand Coal Conference "Clean Coal Technology"

Venue: Park Royal Hotel, Wellington, New Zealand  
Contact: The Conference Secretary  
Sixth New Zealand Coal Conference  
Coal Research Association of NZ  
P O Box 31-244  
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## 3-6 November, 1995 IUPAC 6th International Symposium on Macromolecule-Metal Complexes

Venue: Beijing, China  
Contact: Professor Ying-Yan Jiang  
Institute of Chemistry  
Academia Sinica, Zhongguancun  
Beijing 100080  
China

## 12-16 December, 1995 4th Pacific Polymer Conference

Venue: Kauai, Hawaii, USA  
Contact: Professor Ray Otterbrite  
Department of Polymer Chemistry  
Virginia Commonwealth University  
Richmond, Virginia 23204, USA  
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## 17-22 December, 1995 Pacifichem '95

Venue: Honolulu, Hawaii, USA  
Contact: Professor B Halton  
Chemistry Department  
Victoria University  
P O Box 600, Wellington  
Ph (04) 472 1000

### Pacifichem '95 - Update

Have you been thinking of going to Pacifichem next December? Then you ought to book your flights as soon as possible. The Auckland - Honolulu route only has direct flights with Air New Zealand. If you leave Honolulu on Friday December 22 after the congress you will arrive in Auckland on the morning of December 24. You can travel with Qantas, but the route is via an Australian port.

Abstract forms for invited and submitted papers are to be in the Pacifichem office in Washington by March 31, 1995.

## 7-12 July, 1996 Organometallic Chemistry XVII

Venue: Brisbane, Australia  
Contact: Eva Comino  
Secretariat, International Conference on Organometallic Chemistry  
Faculty of Science and Technology  
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# CONFERENCES & SEMINARS

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**14-19 July, 1996 RACI/SETAC/ASE International Conference on Environmental Chemistry and Toxicology**

Venue: Sydney, NSW, Australia  
Contact: Dr Graeme Batley  
CSIRO Centre for Advanced Analytical Chemistry  
PMB 7, Menai  
NSW 2234, Australia  
Ph +61-2-710 6830  
Fax +61-2-710 6837

**14-19 July, 1996 14th International Conference on Chemical Education (14ICCE)**

Venue: Brisbane, Australia  
Contact: Sally Brown  
Conference Secretariat

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**December, 1996 NZIC Conference**

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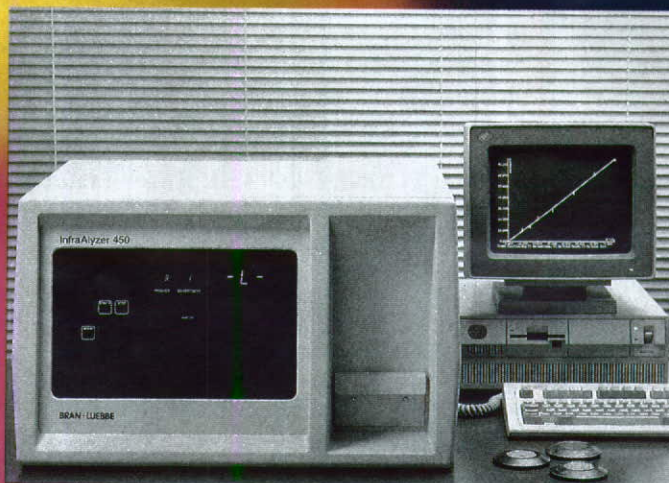
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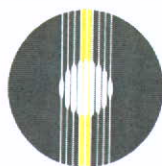
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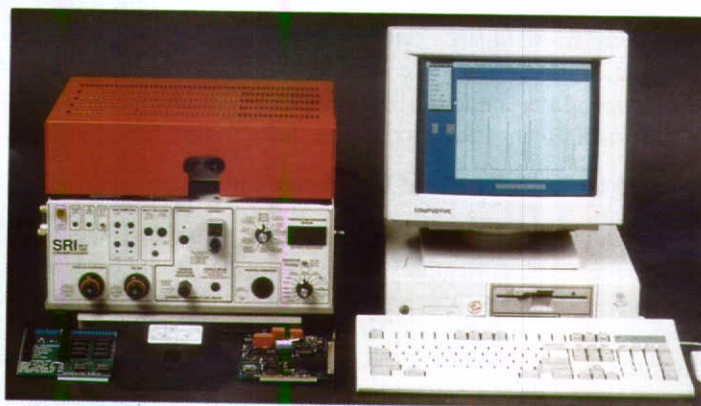
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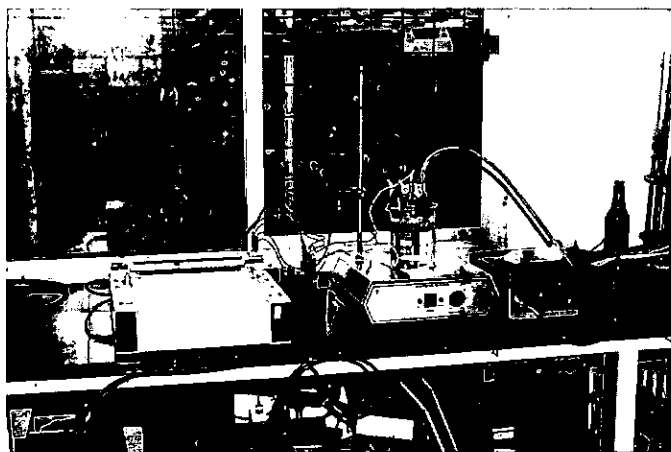
R. H. Molony, MOLAB Limited  
14 Goldie Street, St Heliers, Auckland

Ozone is being increasingly used for water treatment and sterilisation in the food and other industries. As a substitute for chlorine, ozone has the advantage that it does not normally produce halo-alkanes. Ozone does not appear to produce objectionable compounds during normal water treatment.

Aqueous solutions of ozone are not stable and the behaviour of ozone in waters, which may contain a variety of compounds, is extremely variable. Important factors affecting the effectiveness of the ozonation process are the concentration of the ozone and the lifetime of the ozone solution. These factors must be taken into account when ozonation as a treatment process is being considered.

Due to the reactivity and instability of ozone solutions, standard chemical methods are not very suitable for determining the behaviour of ozone in waters. Electrochemical methods appear to be more suitable.

Ozone is theoretically able to react at an inert electrode producing a voltage or a current. The behaviour of ozone at platinum electrodes was examined. The measuring equipment consisted of a platinum micro-electrode, a recorder with an input impedance of 1 megohm, and a silver/saturated silver chloride potassium chloride electrode with a porous junction. Ozonation of water samples was carried out in a beaker on a magnetic stirrer. The ozone generation equipment consisted of an aquarium air pump, a silica gel drying column, and an ozone generator. The ozone generator itself was of the traditional silent electrical discharge type. A pulsed voltage of approximately 10 kV was supplied to the tube by an HT transformer and a relaxation oscillator. A potentiometer bridge was used to null out the baseline voltage due to the initial potential difference between the platinum and silver electrodes.



Equipment used for determining the behaviour of ozone in water. Left to right: recorder, potentiometer box, stirrer, reaction vessel, ozone generator with silica gel drier and air pump.

The behaviour of the experimental setup was examined. A beaker of distilled water was put on the magnetic stirrer, the

sensing electrodes were put in and ozonated air bubbled into the water with stirring. A stirring speed of an estimated 300 rpm was used. Ozonation was stopped by removing the bubbler from the beaker at the end of the impregnation time. The ozone decay section of the curve was a normal first order decay curve (Figure 1). This indicates that the electrode response is amperometric not potentiometric. Note that a first order decay curve on a potentiometric system would be a straight line not a curve. The electrode response is affected by stirring speed. On turning the stirrer off, the electrode response immediately dropped very substantially. Over the range of stirrer speed examined the effect of stirrer speed on electrode response tended to decrease with increasing stirrer speed. The effect of stirrer speed again indicated that the electrode response is amperometric. In an amperometric response the current flow is limited by the diffusion of the active species across the electrode boundary layer. Boundary layer thickness is influenced by the stirring speed. The electrode system is behaving as a fuel cell with ozone being consumed at the platinum electrode and silver being consumed at the silver electrode. Calculation of the system current draw showed that the ozone consumed by the electrode was not significant when compared to ozone decay rate in this equipment.

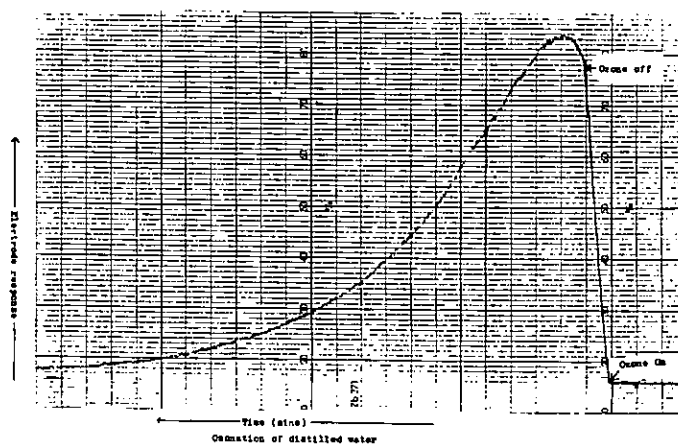


Figure 1: Ozonation of Distilled Water

Examination of the ozonation response curve shows a number of features. During the initial ozone impregnation the curve shows an expected increase in electrode response. At the end of the ozone impregnation time, impregnation is stopped by removing the bubbler from the water sample. After removal of the bubbler the electrode response continues to increase for a time before starting to decrease. Shortly after the curve starts to decrease it assumes the form characteristic of a first order reaction rate. The platinum electrode obviously is not responding to dissolved ozone but to the product of a reaction between ozone and water.

The series of reactions which occur during the ozonation of pure water are worth considering. There appear to be three main reactions;

Reaction 1: Ozone gas passes through the gas liquid interface and dissolves in the water.

Reaction 2: Dissolved ozone reacts with water producing the compound which causes the electrode response. This compound is also probably responsible for the oxidising effects of ozone in aqueous solution. This reaction probably would be either first or second order. One of the reactants is dissolved ozone the other would be water (the concentration of water is constant). This reaction is therefore almost certainly first order with respect to dissolved ozone.

Reaction 3: The product of the ozone-water reaction decays. The electrode response curve of this reaction is first order as mentioned previously.

The literature suggests that in an aqueous solution the ozonide ( $O_3^-$ ) ion is the active component. The formation of the ozonide ion involves non-ionic reactions. This suggests that the formation of the water-ozone compound might be slow as is confirmed by the recorder plot. After the ozone impregnation has stopped the gradient of the recorder plot at any point is dependent on two reactions, the reaction of dissolved ozone with water and the decay of the product of that reaction. By measuring the gradient at several points on the electrode response curve the rates of the two reactions can be calculated. A rate constant for the reaction of dissolved ozone with water has been found to be approximately  $1 \text{ sec}^{-1}$ . This corresponds to a "dissolved ozone" half-life of 0.7 seconds at room temperature.

The reaction of critical importance to the water processor (and food processor where applicable) is not the reaction of dissolved ozone with water but the decay of the product of this reaction. The rate of this decay is conveniently represented by the half life. This is the time taken for half of the active agent to decay. The half life can be determined from simple measurements on the decay section of the response curve. Examples of half lives found for the decay of "ozone" in distilled water are 6.9 minutes at  $15^\circ\text{C}$  and 2.3 minutes at  $25^\circ\text{C}$ . Comparison of the rate of the first reaction with these figures shows that after the electrode response peak the rate of the first reaction makes little contribution to the response curve. In calculating the decay rate the contribution of the first reaction to the response curve can be ignored shortly after the peak. Two types of measurements can be used to determine the half life of "ozone" in solution. First is the height of the response curve above the baseline at two different times. The other procedure is to measure the height of the response curve above the baseline at a single time and also the curve gradient at the same time.

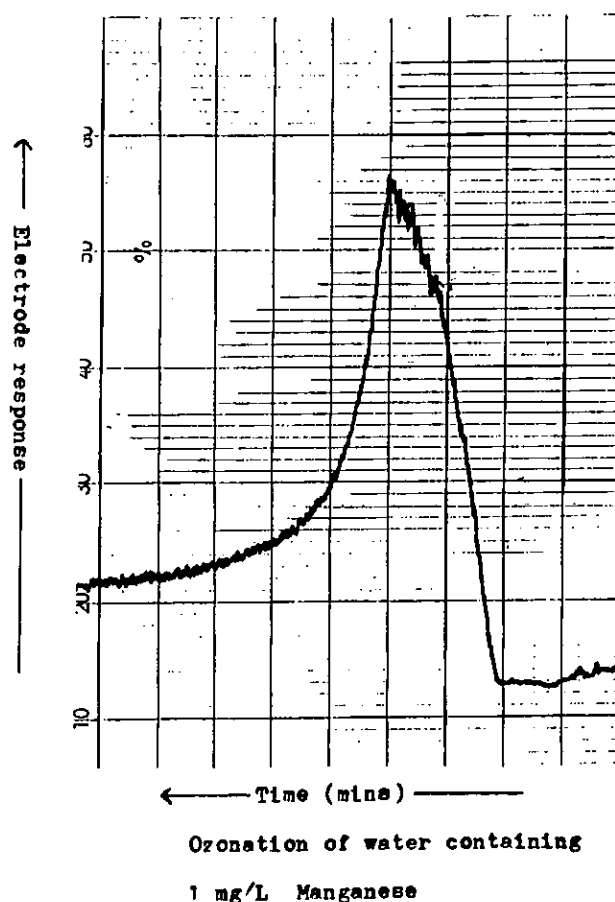
The usefulness of ozone in water treatment depends on its ability to oxidize a variety of materials present in the water. The amount of the oxidation which takes place depends both on the "ozone" concentration and the contact time with the "ozone". There is a suggestion in the literature that some trace metals may catalyse the decomposition of "ozone" solutions. The author on one occasion saw a water sample which contained no permanganate reducing matter and in which a detectable free residual ozone could not be attained.

**Table I:**  
Effect of some metals on the half-life of "ozone" solution.  
Ozone addition approximately 5 mg/L.

METAL	mg/L	ANION	HALF-LIFE (mins)
Cobalt(II)	0.8	Sulphate	3.7
Cobalt(II)	8	Sulphate	3.4
Manganese(II)	1	Sulphate	0.5
Iron(III)	0.5	Chloride	3.1
Iron(III)	2.5	Chloride	1.7
Sodium	13	Sulphate	3.9

Note: all the solutions contained sodium sulphate at the concentration given for sodium.

The effects of a few transition metals on "ozone" solution half life were examined. (See Table I). Cobalt, iron and manganese all showed an ability to cause the decomposition of "ozone" in solution. Manganese in addition to being a good catalyst showed another effect. The electrode response did not decay to the preozonation level but to a level higher than the initial electrode response (See Figure 2). Not only was there a change in the baseline level of response but the solution also showed a distinct colour change to an orange-brown. Manganese(II) Sulfate which was originally present in solution appears to have been oxidised to a higher oxidation state, probably either manganese(III) or manganese(IV). This higher oxidation state catalyses the break down of "ozone" solution. The decreasing gradient of the response curve with increasing ozone impregnation indicates that the higher oxidation state of manganese causes a more rapid break down of "ozone" in solution than does the initial oxidation of the manganese(II).



**Figure 2:** Ozonation of water containing 1mg/L manganese

Cobalt(II) causes a break down of "ozone" solution with a shift in the electrode baseline response. Cobalt(III) is almost certainly the product of the oxidation however the catalytic break down of "ozone" in solution in this case seems dubious. It should however be noted that cobalt(III) is an oxidant.

The naked platinum electrode does not exclusively respond to the "ozone" in solution. In fact the electrode will respond to a variety of oxidizing and reducing agents in solution. In the above cases the electrode responds to the higher oxidation states of manganese and cobalt in addition to "ozone".

Iron(III) causes a rapid break down of "ozone" in solution with the electrode response returning to the preozonation response level. Iron(III) appears to be a good catalyst for the break down of "ozone" solution. Iron(II) is extremely rapidly oxidised to iron(III).

Ozone impregnation is not always an effective treatment process being markedly affected by materials present in water. In addition to the effect water components may have on "ozone" half life, surfactants retard the passage of ozone through the gas-liquid interface. Adequate experimental work should be carried out on the water to be treated before committing finance to an expensive ozonation plant. The process described is a useful tool for the investigation of the ozonation of waters.

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Degremont; Water Treatment Handbook 1979.

Lingane; Electroanalytical Chemistry 1966.

\* \* \* \* \*

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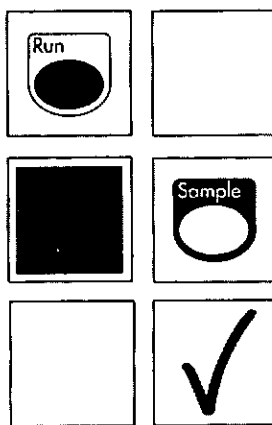
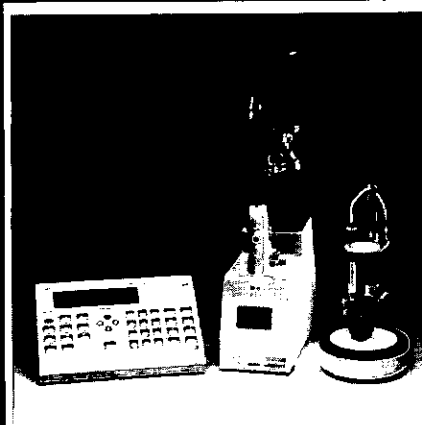
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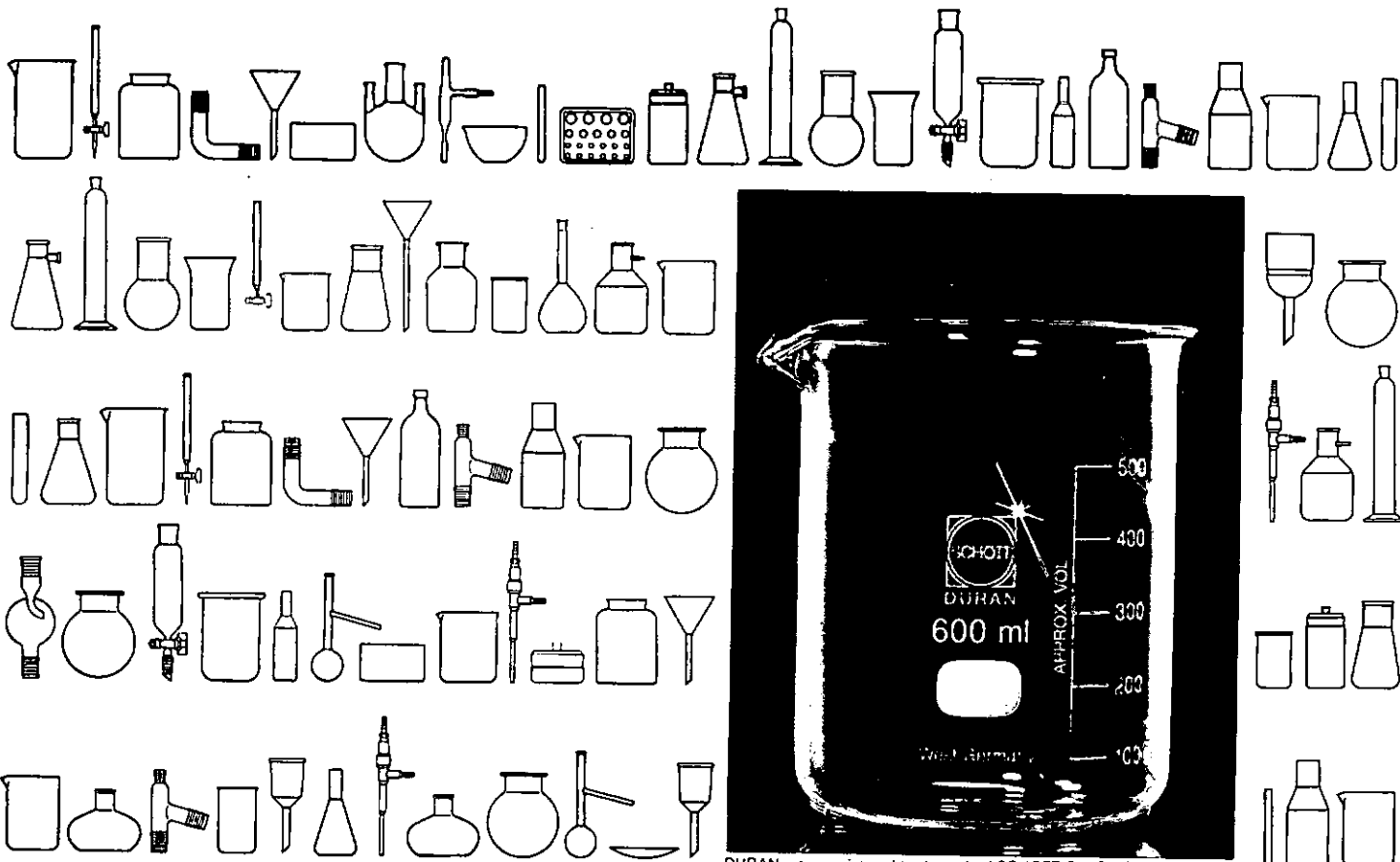
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# SOLID PHASE MICROEXTRACTION SIMPLIFIES PREPARATION OF FORENSIC, PHARMACEUTICAL, AND FOOD AND BEVERAGE SAMPLES

By R. F. Mindrup

Supelco Incorporated, Bellefonte, Pennsylvania, USA

Solid phase microextraction (SPME) is a fast, solvent-less alternative to conventional sample extraction techniques. In SPME, analytes establish an equilibrium between the sample matrix and a stationary phase coated on a fused silica fibre, then are thermally desorbed from the fibre to a capillary GC column. Because no solvent is injected, and the analytes are rapidly desorbed onto the column, minimum detection limits are improved and resolution is maintained. SPME is proving to be useful in many diverse analyses, including forensics, quality evaluations of pharmaceuticals, and characterization of foods and beverages.

Solid phase microextraction (SPME) is a simple, solvent-less extraction procedure in which a phase-coated fused silica fibre is immersed in a liquid sample or exposed to the headspace above a liquid or solid sample. Analytes adsorb to the phase, and then are thermally desorbed in the injection port of a gas chromatograph and transferred to a capillary column. Selectivity can be altered by changing the phase type or thickness according to the characteristics of the analytes. For example, the small distribution constants and low polarity of chlorinated and aromatic volatile organic compounds dictate the use of a thick, nonpolar phase for efficient extraction. The analyst can improve analyte recovery, or alter selectivity in favour of more volatile or less volatile compounds, by adding salt to the sample, changing the pH, sampling the headspace rather than the sample (or vice versa), or making other changes in conditions.

In the months immediately after Supelco introduced products for solid phase microextraction (*The Supelco Reporter XII*, No. 2, 1993), most of the applications reported for this revolutionary new sample preparation technology involved environmental samples. Recently, however, analysts in other fields have begun to describe a wide variety of uses for the technique. In particular, SPME is proving very effective for headspace sampling applications. The new applications summarized here were developed by groups of independent investigators in three diverse fields: forensics, pharmaceuticals manufacture, and the foods and beverages industry.

## Forensics: Gasoline

Investigators in the Crime Laboratory Bureau of the Metro-Dade Police Department (Miami, Florida, USA) and the Department of Chemistry at Florida International University (Miami) developed what they described as a simple, inexpensive, rapid, and sensitive method for analysing gasoline in fire debris, using SPME for headspace sampling (1). According to the investigators, current methods for sampling flammable or combustible liquid residues from fire debris include static headspace sampling (capable of detecting ~10  $\mu$ L of petroleum product residue) and concentration methods

including solvent extraction, dynamic headspace concentration, and passive headspace concentration (capable of detecting ~1  $\mu$ L of petroleum product residue). All of the concentration methods are cumbersome and time-consuming, and require the analyst to use carbon disulfide, a toxic and highly flammable solvent. In a direct comparison of headspace SPME and passive headspace concentration on activated charcoal strips, the SPME method was faster, simpler, and more economical, and offered greater sensitivity. The SPME method also eliminated the need to expose the technician to carbon disulfide.

Figure A. Headspace Sampling of 0.1  $\mu$ L Gasoline

SPME: 100 $\mu$ m polydimethylsiloxane phase fiber  
headspace sampling (20 min)  
10 sec desorption (splitless mode)  
Cat No.: 5-7300  
Column: polydimethylsiloxane phase,  
30m x 0.25mm ID, 0.25 $\mu$ m film  
Oven: 35°C (2 min) to 220°C, at 10°C/min, hold 2 min,  
to 300°C at 30°C/min, hold 5 min  
Carrier: helium, 1mL/min (split 50:1)  
Det.: FID, 300°C  
Inj.: splitless (closed 3 min), 220°C  
(2mm ID injector liner)

1. Toluene
2. Ethylbenzene
3. m-Xylene, p-Xylene
4. o-Xylene
5. 1,2,4-Trimethylbenzene
6. 1,2,3-Trimethylbenzene
7. n-Butylbenzene
8. Naphthalene
9. 2-Methylnaphthalene
10. 1-Methylnaphthalene

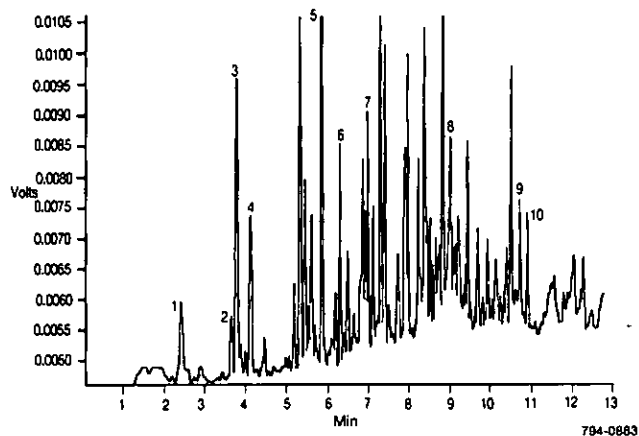


Figure courtesy of José Almirall, Crime Laboratory Bureau, Metro-Dade Police Department, Miami, Florida, USA, and Kenneth Furton and Juan Bruna, Department of Chemistry, Florida International University, Miami.

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After heating various fixed volumes of gasoline at 40°C for 30 minutes, the investigators exposed a 100  $\mu$ m polydimethylsiloxane-coated SPME fibre to the headspace above the sample for 20 minutes, then desorbed and analysed the analytes under conditions listed for Figure A. They used a Hamilton® heated syringe cleaner (120 VAC; Supelco Cat. No. 2-0770) to clean the fibre assembly between samples. Although relatively small amounts of analytes were recovered under these conditions, the SPME technique eliminated sample transfer/sample

handling losses and yielded high sensitivity because the entire extract was introduced onto the GC column. The investigators calculated that headspace SPME offered, on average, a 1.0-, 3.8-, and 11.2-fold increase in response, relative to passive headspace concentration, for three measured components of the gasoline samples (Table 1). SPME produced acceptable, identifiable chromatograms from as little 0.04  $\mu\text{L}$ . The forensics experts also were impressed with the savings in time (20 minutes or less, versus 16 hours) and cost per sample (less than half the cost of passive sampling) offered by SPME. These analysts are now applying their headspace SPME technique to analysis of other samples, including alcohols and diesel fuel.

**Table 1:** Detector Responses for Selected Hydrocarbons in Gasoline

Analyte	Gasoline Qty. ( $\mu\text{L}$ )	FID Response SPME	(Volts) Passive	SPME Response/Passive Response
Ethylbenzene	5	0.07	0.12	0.6
	1	0.015	0.010	1.5
	0.1	0.0057	0.0060	1.0 $\bar{x}$ = 1.0
n-Butylbenzene	5	0.26	0.078	3.3
	1	0.042	0.0065	6.5
	0.1	0.0090	0.0062	1.5 $\bar{x}$ = 3.8
2-Methylnaphthalene	5	0.28	0.02	14
	1	0.042	0.0023	18.3
	0.1	0.0076	0.0055	1.4 $\bar{x}$ = 11.2

Data from reference 1.

### Residual Solvents in Pharmaceuticals

Chemists at Hoffmann-La Roche Inc. (Nutley, New Jersey, USA) compared headspace SPME and immersion SPME for determining organic volatile impurities (OVIs) and residual solvents in several water-soluble drug substances (2). The United States Pharmacopoeia (USP) chapter <467> describes several methods of analysis for OVIs (benzene, chloroform, 1, 4-dioxane, methylene chloride, trichloroethylene) in pharmaceutical drug substances and raw materials. In addition, pharmaceutical manufacturers must monitor residual organic solvents from the manufacturing process of a bulk drug substance.

Immersion and headspace SPME were essentially equal with respect to precision (Table 2), sensitivity (Table 2), and accuracy. The Hoffmann La Roche chemists preferred the headspace method because it prolonged the lifetime of the SPME fibre. A 100  $\mu\text{m}$  polydimethylsiloxane-coated fibre provided higher sensitivity toward the more nonpolar analytes (i.e., the OVIs). A polyacrylate-coated fibre offered higher sensitivity toward the polar analytes (alcohols). Detection limits, using the polydimethylsiloxane-coated fibre, ranged from 0.06  $\mu\text{g}/\text{mL}$  and 0.3  $\mu\text{g}/\text{mL}$  for 1,4-dioxane (by headspace and immersion, respectively) to 0.002  $\mu\text{g}/\text{mL}$  for benzene (both techniques). Figure B shows typical SPME/GC chromatograms of an OVI/residual solvent standard and a pharmaceutical drug substance. Methanol, added to obtain reproducibility for the OVIs, is present at 1.0% v/v in the water diluent. The SPME sample introduction technique has proved to be useful for screening OVIs in pharmaceutical drug substances.

**Table 2:** SPME for Organic Volatile Impurities and Final Recrystallization Solvents

Solvent	Precision (%RSD)		Detection Limit ( $\mu\text{g}/\text{mL}$ )	
	Headspace SPME	Immersion SPME	Headspace SPME	Immersion SPME
Acetone	1.1	0.5	0.2	0.4
Ethanol	7.0	5.8	5.0	2.0
Isopropanol	1.4	1.9	0.6	1.6
Benzene	2.7	2.8	0.002	0.002
Chloroform	3.2	2.2	0.03	0.04
1,4-Dioxane	1.9	2.2	0.06	0.3
Methylene chloride	2.6	2.2	0.06	0.08
Trichloroethylene	3.4	3.2	0.02	0.01

Data from reference 2.

**Figure B.** Residual Organic Solvents in a Pharmaceutical Preparation

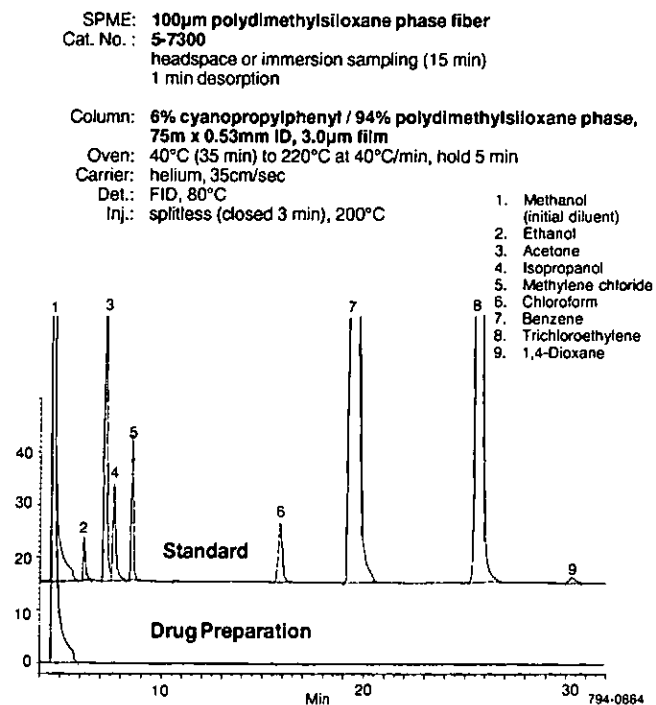


Figure courtesy of Stephen Scypinski, Ann-Marie Smith, Linda Clark Nelson, and Sandra Rosen Shaw, Hoffmann-La Roche, Nutley, New Jersey, USA.

### Flavour Components: Solid, Water-Based, and Vegetable Oil-Based Samples

Flavour chemists at Givaudan-Roure Corporation (Clifton, New Jersey, USA) used headspace sampling SPME, immersion SPME, and conventional extraction methods to monitor flavour components in espresso-roast ground coffee, a fruit juice beverage, and butter-flavoured vegetable oil (3). In their analyses of ground coffee, conventional headspace sampling was more sensitive for highly volatile compounds, but headspace SPME sampling was superior for less volatile compounds. The SPME method also eliminated the introduction of water into the chromatograph.

The sensitivity of immersion SPME was comparable to or higher than that of solvent (dichloromethane) extraction for most esters (e.g., ethyl isovalerate, cis- and trans-methyl cinnamate),

terpenoids (e.g., linalool,  $\alpha$ - and  $\beta$ -terpineol) and  $\gamma$ -decalactone in fruit juice (Figure C). The 100  $\mu$ m polydimethylsiloxane SPME fibre exhibited weak affinity for fatty acids in the beverage (Figure C), but the investigators considered this an advantage because it reduced the potential for interference with the flavour compounds.

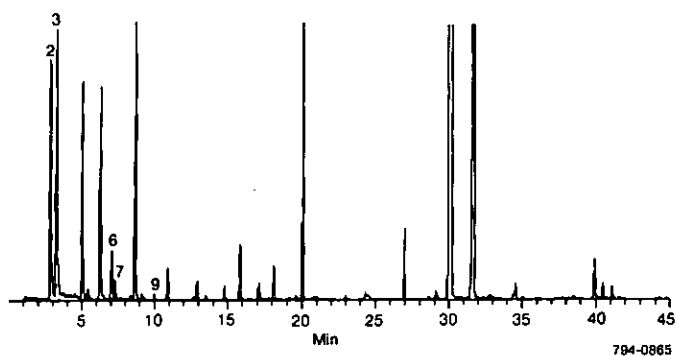
**Figure C. Flavour Compounds in Fruit Juice Beverage**

SPME: 100 $\mu$ m polydimethylsiloxane phase fiber  
immersion sampling (10 min); 3mL sample, 0.6g NaCl  
added 3 min desorption  
Cat. No.: 5-7300

Column: Carbowax 20M-type phase,  
30m x 0.25mm ID, 1 $\mu$ m film  
Oven: 50°C (2 min) to 220°C at 4°C/min  
Carrier: helium, 20cm/sec  
Det.: MS (m/z = 35-400)  
Inj.: splitless (closed 2 min), 200°C  
(1mm ID injector liner)

- |                            |                                   |
|----------------------------|-----------------------------------|
| 1. Dichloromethane         | 14. Linalool                      |
| 2. Ethyl butyrate          | 15. $\beta$ -Terpineol            |
| 3. Ethyl isovalerate       | 16. Butyric acid                  |
| 4. Limonene                | 17. 2-Methylbutyric acid          |
| 5. Ethyl hexanoate         | 18. $\alpha$ -Terpineol           |
| 6. Isoamyl butyrate        | 19. Hexanoic acid                 |
| 7. Hexanyl acetate         | 20. cis-Methyl cinnamate          |
| 8. cis-3-Hexenyl acetate   | 21. 1-(2-Furyl)-2-hydroxyethanone |
| 9. Hexanol                 | 22. Furanol                       |
| 10. cis-3-Hexenol          | 23. trans-Methyl cinnamate        |
| 11. cis-3-Hexenyl butyrate | 24. $\gamma$ -Decalactone         |
| 12. Furfural               | 25. Dodecanoic acid               |
| 13. Benzaldehyde           | 26. (Hydroxymethyl)furfural       |

**SPME (100 $\mu$ m polydimethylsiloxane)**



**Solvent Extraction (dichloromethane)**

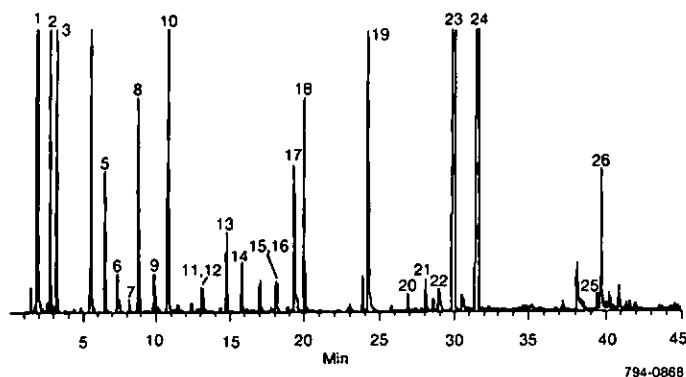


Figure courtesy of Xiaogen Yang and Terry Perrard, Givaudan-Roure Corporation, Clifton, New Jersey, USA.

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Headspace SPME at an elevated temperature (160°C) proved effective for detecting the characteristic components of butter flavour in vegetable oil (diacetal,  $\delta$ -decalactone,  $\delta$ -dodecalactone). The investigators considered alternative methods, including static headspace sampling insensitive and/or considerably more time-consuming. Like the forensics group and the pharmaceutical chemists, the flavour chemists concluded that SPME is a useful tool, complementary to other sample extraction methods. In analyses of flavour compounds in solid, liquid, or gaseous samples, it is simple, fast, and requires no solvent.

Because liquid and headspace sampling methods differ in kinetics, the flavour chemists considered the methods complementary (3). For a given sampling time, immersion SPME was more sensitive than headspace SPME for analytes predominantly present in the liquid. The reverse was true for analytes that were primarily in the headspace. These generalisations can be used to advantage to selectively adsorb more volatile or less volatile flavour compounds, as a situation warrants.

Several additional factors can affect SPME and influence the choice between immersion and headspace sampling. Addition of an electrolyte (e.g., a salt) to a solution generally increases the adsorption of analytes by both immersion SPME and headspace SPME. Similarly, increasing the sample volume from 200  $\mu$ L to 3 mL, while keeping the ratio of liquid to headspace constant (1:1, increased analyte adsorption by both immersion and headspace SPME (3). For higher sensitivity from headspace SPME, the sample headspace should be as small as is practical. A detailed theoretical discussion of headspace SPME is presented in reference 4.

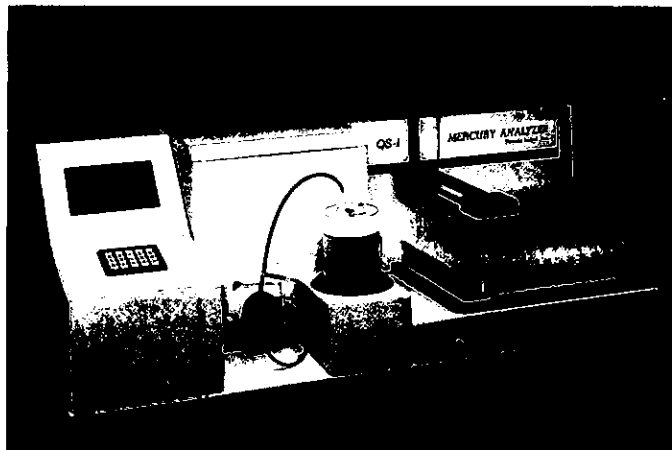
The diverse results summarized here made several points in common. SPME is fast, easy, and economical, and eliminates the costs and hazards associated with using organic solvents. Under consistent sampling conditions, analytes can be extracted with good precision over wide ranges of concentrations. SPME can be used for screening samples prior to a detailed analysis. Good precision also makes the technique effective in quantitative analyses. If you are interested in reducing the time and expense of sample concentration in your analyses, SPME might be the answer to your needs.

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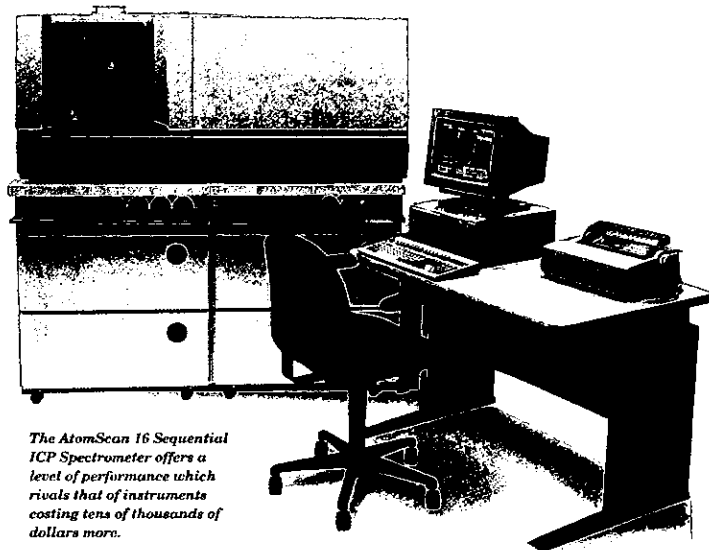
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Phone: (03) 379-8467  
Phone: (03) 477-7860



or circle number 13 on the reader reply card.

CHEMISTRY IN NEW ZEALAND/MARCH 1995

# NEW PRODUCTS

## THE AMAZING NEW ASE 200 ACCELERATED SOLVENT EXTRACTOR FROM DIONEX - EXTRACTION THE WAY YOU ALWAYS WANTED IT TO BE

ASE is a new technology for extraction of solid matrices that is fast, easy and economical. ASE uses conventional extraction solvents, but in significantly smaller quantities than other solvent based methods, dramatically reducing operating costs.

Extraction is accelerated by using solvents at elevated temperature and pressure. Increased temperature accelerates the extraction kinetics, while high pressure keeps the solvent below its boiling point resulting in safe and rapid extractions. Both time for extraction and solvent consumption are significantly reduced compared to Soxhlet and sonication techniques.

Operation of the ASE 200 is so straight forward and easy you can plan on extracting samples minutes after your unit is installed. Start by using the same solvent specified by your existing method - it will most likely provide good recoveries on the first attempt.



## FLEXIBLE NEW Biochemistry Analyser for Bioprocessing

The new YSI 2700 SELECT Biochemistry Analyser offers speed and flexibility for testing in food and pharmaceutical production and biotechnology.

Little or no sample preparation for measurements of:

- ▼ Dextrose
- ▼ L-Lactate
- ▼ Galactose
- ▼ Sucrose
- ▼ Lactose
- ▼ Starch
- ▼ Hydrogen Peroxide

John Morris Scientific Ltd

John  
Morris

Scientific

Auckland  
P O Box 6348  
Wellesely Street  
Ph: (09) 366-3999  
Fax: (09) 366-3060  
Toll Free: 0800 651700



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# NEW PRODUCTS

ASE meets all of the requirements for extraction under US EPA SW-846 method 3545 (proposed) for accelerated solvent extraction of base/neutrals and acids (BNAs), chlorinated pesticides and herbicides, polychlorinated biphenyls (PCBs) and organophosphorus pesticides.

Find out how easy it is to bring more efficient, cost-effective extraction to your laboratory.

Contact: SciTech,  
P O Box 663, Dunedin  
Ph: (03) 477-7860 Fax: (03) 477-7870  
circle number 21 on the reader reply card.

## CHECK WEIGHING WITH A DIFFERENCE

The Denver Instrument DI Balance with its detachable display brings new precision to check weighing, and parts counting. Because it employs force restorative mechanics (not the less accurate strain gauge type) results are more accurate even in the most difficult conditions. But operation is simplified with OVER, UNDER and OK indicators for daily operation by unskilled operators. Computer connections are standard, but 255 values are stored and analysed right on the spot for statistical variation. The demountable display feature is especially useful with glove boxes, fume hoods, and sterile manufacturing facilities.



For easy to use, high precision check weighing in challenging conditions call:

Alphatech Systems  
P O Box 37583 Parnell, Auckland  
Ph: (09) 3770392 Fax: (09) 3098514  
circle number 22 on the reader reply card.

## IEC MEDISPIN CENTRIFUGE ECONOMY AND SAFETY

IEC's new MediSpin is an economical benchtop centrifuge ideal for routine, low volume blood and urine separations, as well as a host of other low speed applications. This compact, reliable unit spins most common tubes - up to six 15 mL or twelve 10 mL tubes at 3,100 rpm for fast, positive separations.

The MediSpin features an integral 30 minute timer with hold mode, fail-safe cover interlock, brushless motor for unbeatable reliability, and comes complete with either a six-place or a twelve-place rotor and standard tube shields. Sealed Aeroshields™ are available to accommodate tubes in either rotor and provide aerosol containment.

Contact: SciTech  
P O Box 663, Dunedin  
Ph: (03) 4777860 Fax: (03) 4777870  
circle number 23 on the reader reply card.

## AUDIO VISUALS FOR ANALYSIS AND TRAINING

SAVANT (Sloane Audio Visuals for Analysis and Training) offer a wide range of audio visual training aids covering laboratory safety, electrochemistry, nuclear instrumentation, gas chromatography, ion chromatography, thin layer chromatography, HPLC, RIA, EIA, and AA, ICP/OES, ICP/MS, NMR, IR, UV/Visible spectroscopy and many other techniques.

Formats include video, text and PC-based programmes.

Demonstration disks are available from SciTech the New Zealand distributors.

Contact: SciTech  
P O Box 663, Dunedin  
Ph: (03) 4777860 Fax: (03) 4777870  
circle number 24 on the reader reply card.

## SYNTHEWAVE MICROWAVE ASSISTED ORGANIC SYNTHESIS

Discover new chemistry with the new PROLABO focused microwave reactor. A new dimension in organic synthesis.

### ADVANTAGES

- Faster organic synthesis
- Solvent free reactions
- Minimum wastes
- Higher yields (>80%)
- Higher purity
- Higher reproducibility

### FEATURES OF THE PROLABO SYSTEM

- Infrared and continuous feed back temperature control
- Constant visual control
- 250 mL quartz reactor

### APPLICATIONS

- Pharmaceutical research
- Polymers industry
- Cosmetics

Contact: SciTech  
P O Box 663, Dunedin  
Ph: (03) 4777860 Fax: (03) 4777870  
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# NEW PRODUCTS

## FINNIGAN MAT INTRODUCES THE GCQ AND GCQ TANDEM UNIVERSAL BENCHTOP GC/MS AND MS/MS DETECTORS

Whether the compounds you analyse are low or high molecular weight, derivatized or underivatized, the GCQ can provide you with both accurate qualitative and precise quantitative data. From positive and negative ion detection to the power of MS/MS, the GCQ has all the tools, you'll need to confidently detect, identify, and quantitate virtually all types of compounds.

The affordable GCQ combines the ion source and detection system from Finnigan MAT's high-performance quadrupole mass spectrometers with the sensitive quadrupole ion trap mass analyser. With this unique combination of proven technology, you now can generate classical spectra at ultra-low detection limits.

With a mass range of 1000 amu, you can analyse compounds previously out of range for benchtop GC/MS systems.

Choose from the most popular GCs, or even optional probe inlets, to introduce your samples into the mass spec detector. The GCQ interfaces to and fully controls Finnigan, Hewlett Packard, and Varian GCs. For compounds not amenable to GC analyses, the system also accommodates either a Direct Exposure Probe or a Membrane Introduction Mass Spectrometry (MIMS) probe for direct analysis of aqueous samples.

Switchable Electron Ionization (EI) and Chemical Ionization (CI), with Finnigan MAT's patented exchangeable ion volumes, is a standard feature of the GCQ and gives the user flexibility to choose the amount of fragmentation they desire. What's more the analyst can choose to use CI to generate adduct ions, for additional information to confirm molecular weight.

With high pressure chemical ionization and the GCQ's high performance detection system you can also create and detect negative ions. Negative ion spectra have inherently lower background, and produce very high signal-to-noise analytical responses for detection and quantitation. This feature, known as ECD-MS, replicates the ionization process of an Electron Capture Detector, a highly sensitive GC detector for electrophilic materials such as halogenated compounds.

For target compound analysis, and the ultimate in detection, the GCQ features Ultra-SIM, a selected ion monitoring (SIM) mode of scanning. When a complete sample spectrum is required, the full scan mode of analysis provides you with library searchable spectra.

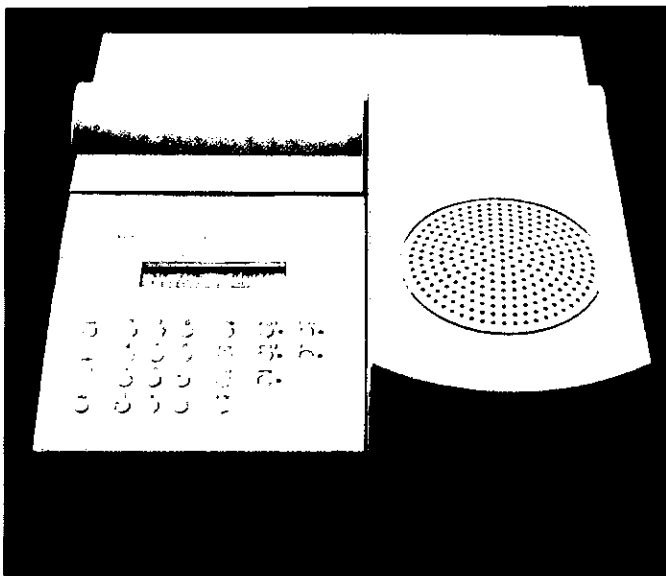
In addition to all the standard features of the GCQ, the GCQ *Tandem* delivers to your laboratory the power of MS/MS. With MS/MS, your analyses can be even more selective, and the ions of interest can be totally isolated from background contamination or coeluting interferences. On the GCQ, analytical parameters are calculated automatically to make MS/MS a tool for high productivity. Patented ion isolation techniques allow the analyst to choose either one or two ions (e.g., the analyte and internal standard) per analytical scan.

For data processing and instrument control, the GCQ is operated by a personal computer and software operating under the powerful, full-featured Microsoft® Windows™ NT environment. Windows NT includes familiar Windows' features such as pull down menus, dialog boxes, scroll bars, etc., plus many added benefits. Among these, the new environment allows you to work on multiple tasks simultaneously, share information and files with integrated mail and networking services, maintain controlled security access, and still run all of your favourite Windows applications.

Classical spectra, superior sensitivity, extended mass range, and a host of other standard features make the GCQ the new standard for benchtop MS detectors.

Contact: SciTech  
P O Box 663, Dunedin  
Ph: (03) 4777860 Fax: (03) 4777870  
circle number 26 on the reader reply card

## MOISTURE BY DRYING ON THE SHOP FLOOR



The IR-500 is the newest Infrared Moisture Balance from Denver Instruments. Designed for use in the production area the IR-500 gives accurate answers more quickly than an oven, without sacrificing accuracy. Multiple methods composed of several drying steps and rates are user programmable so your real samples are all accommodated, with results displayed in the units you want to see, whether you are drying to a constant weight or a preset time, in % moisture or grams. No calculations, no redrying check steps, no trips from the oven to the balance juggling a desiccator. Ruggedly built the IR-500 belongs on the production floor.

Contact: Peter, Alphatech Systems  
P O Box 37583 Parnell, Auckland  
Ph: (09) 3770392 Fax: (09) 3098514  
circle number 27 on the reader reply card.

# NEW PRODUCTS

## THE NEW BIORAD BioLogic System FOR HIGH RESOLUTION PROTEIN PURIFICATION



Bio-Rad announce the introduction of a new liquid chromatography system for high resolution protein purification. The new modular BioLogic System is completely biocompatible and can be used in the coldroom. A core system comprises a workstation, controller and rack.

The BioLogic Workstation is the heart of the new system. It houses the gradient pump system consisting of two 1000 psi dual piston pumps, a pressure transducer, and a high pressure dynamic mixer to ensure highly accurate and reproducible gradients. Made entirely of biocompatible PEEK and ceramic materials to preserve protein integrity, the advanced design pump head facilitates easy removal for inspection and routine maintenance. Also contained within the workstation is the control circuitry for the UV detector, and conductivity detector which are standard with the BioLogic system, and the valves controller. This integrated design eliminates the complex electrical connections of individual components, simplifying system set up and saving valuable bench space.

The brain of the new system is the BioLogic Controller. This provides single point control of all system components and peripheral instruments. The BioLogic Software accelerates the process of developing and optimizing biomolecule separation protocols. The graphical user interface takes full advantage of the Microsoft Windows operating system, simplifying method programming, set up, on-line monitoring and documentation.

Additional instruments and peripherals that are required to suit each individual users needs are supported and controlled by the BioLogic System. This means the new system is completely compatible with Bio-Rad Model 2110 or 2128 Fraction Collectors or Model 1325/1327 Chart Recorders. The BioLogic Controller can also provide basic control for non Bio-Rad instruments.

Contact: Bio-Rad Laboratories Pty Ltd  
P O Box 100-051, Auckland  
Freephone 0508-805500, Fax (09) 443 3097  
circle number 28 on the reader reply card.

## NEW OMNI HOMOGENIZERS

As a leader in the field of homogenizing, Omni International recognized that as needs change so must Omni's line of homogenizers. Acknowledging these needs, Omni is introducing two new homogenizers, the improved OMNI 2000 and the Model 5100 OMNI 5000. Unlike its predecessor the improved OMNI 2000 is now available as either a post mounted or a handheld homogenizer, and the Model 5100 OMNI 5000 now has a built-in speed control.



### THE IMPROVED OMNI 2000

- Faster; Homogenizes at speeds from 5,000 to 30,000 rpm.
- Built-in speed control.
- Versatile: Accepts rotor stator generators of four different diameters (5mm, 7mm, 10mm, and 20mm).
- Process volume of 0.03 mL to 1500 mL.
- Post mounted or handheld
- Quietest homogenizer: 72 dB.

### THE NEW MODEL 5100 OMNI 5000

- Operates at speeds from 10,000 to 30,000 rpm.
- Built-in speed control.
- Versatile: Accepts rotor stator generators of four different diameters (7mm, 10mm, 20mm, and 35mm).
- Process volumes of 0.25 mL to 20 L.
- Post mounted or handheld.
- Quiet homogenizer: 76 dB.

Contact: Watson Victor Ltd  
P O Box 1180, Wellington  
Ph: (04) 385-7699, Fax: (04) 384-4651

Offices also in: Auckland - Ph: (09) 579-3039  
Christchurch - Ph: (03) 366-9282  
Dunedin - Ph: (03) 477-7291

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# NEW PRODUCTS

## OPTICAL MICROSCOPY MEETS SCANNING PROBE MICROSCOPY (SPM)

Near-field scanning optical microscopy (NSOM) overcomes the diffraction barrier that limits the image magnification of an optical microscope and allows features much smaller than the wavelength of light to be imaged. With NSOM, the advantages of optical microscopy are combined with the extremely high magnification and image resolution of a scanning probe microscope. NSOM is set to play a key role in the advancement of semiconductor technology, materials science, medical research, DNA sequencing and gene mapping, among many other important fields.

Prior to NSOM there were two major light microscopy techniques - the optical microscope and the confocal microscope. In an optical microscope a sample is illuminated with a flood of light and a series of glass lenses is used to image and magnify the sample. A colour magnified image of the sample may be seen directly, or displayed on a TV screen. Even if the lenses could be made perfectly, the resolution and magnification are limited by diffraction to approximately half the wavelength of the light used. Optical microscopes are used routinely to image the general shape of samples as small as human chromosomes or compact disk bits.

In a confocal microscope, the sample is illuminated with a focused spot of laser light. This spot is scanned over the sample and the light collected in a way which allows an image to be displayed on a computer screen. The magnification system is the same as the optical microscope, however, a confocal microscope offers about a 40% improvement in image resolution, as well as the ability to produce a three-dimensional image.

In a near-field scanning optical microscope the sample is illuminated with laser light, but the light is confined in a fibre-optic guide. The fibre is prepared by tapering one end to allow an exit for the light - the final shape looks like a microscopic eye dropper. This fibre guide is no bigger than a human hair and is tapered so that the end is a few atoms wide. Using techniques common for a scanning probe microscope, this fibre end is held close to a sample surface and scanned. The magnified image of the sample is then displayed on a computer screen. This small, nanoscale light source allows more than an order of magnitude better resolution than the best optical or confocal microscope. A near-field scanning optical microscope can be used to see extensive details in a chromosome, image much smaller structures such as viruses, and even make measurements from single molecules.

For more information:-

Contact: Science & Technology (NZ) Ltd  
P O Box 663, Dunedin  
Ph: (03) 477-7860 Fax: (03) 477-7870  
circle number 30 on the reader reply card.

## NEW TEXTURE PROFILE ANALYSIS (TPA) INSTRUMENT FROM LLOYD INSTRUMENTS

Texture profile analysis (TPA) is an integral part of the testing of new products and quality checks on established ones. The new LRX texture analyser from Lloyd Instruments, together with the wide range of test cells available, provides the food industry with a complete TPA system.

Conventionally, texture testing is carried out by food tasting panels which are an essential part of the development of new products or the review of older production methods. Mechanical testing removes the subjectivity associated with human testing panels and attempts to quantify some of the parameters involved in TPA and other types of food testing such as hardness, cohesiveness, stringiness etc.

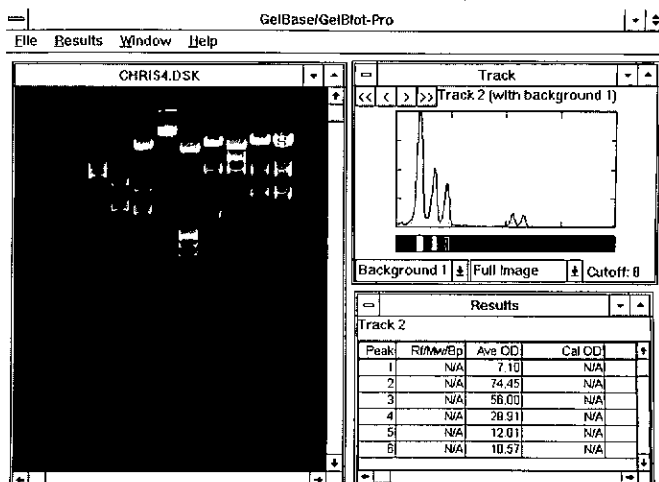
The LRX machine from Lloyd Instruments is a small bench mounted system which can be used on its own or in conjunction with a PC for rapid data analysis, comparison, storage and graphic facilities. A range of test cells developed specially for the food testing industry, including the Warner Bratzler shear test cell, back extrusion food cell, and a texture probe set are available.

In addition to providing a complete food texture testing system, with the addition of different testing grips and fixtures, the LRX can be adapted to perform a range of tests on packaging materials and finished cartons.

Contact: GBC Scientific (NZ)  
P O Box 68330 Newton, Auckland  
Ph: (09) 373-5765 Fax: (09) 360-0683  
circle number 31 on the reader reply card.

## PRO SERIES - A NEW GENERATION OF SOFTWARE FOR GEL ANALYSIS

Cambridge-based Ultra-Violet Products has introduced a new generation of software for its advanced gel documentation systems. GelBase Pro Series software comprises a number of advanced modular packages and is designed to analyse images from DNA/RNA and protein gels, autoradiographs, spots and blots. Routine analysis is accurate and can be performed in less than three minutes - all software programs operate through a series of standardised windows and menu options.



# NEW PRODUCTS

This powerful package operates on both the PC and Apple Mac platforms. Users can start with GelBase Pro, a one-dimensional track analysis package, and add new software modules as their requirements grow. Programs for two dimensional spot and blot analysis (GelBlot Pro), gel sequencing (GelSeq) and matching (GelMatch) are available. To speed up analyses, the software can be configured to operate via a series of default settings or, alternatively, users can customise the software to match specific applications.

Typically, the gel documentation system is positioned in the laboratory while the computer is located in a less vulnerable area. Images captured by the Imagestore 5000 or Gds 7500 are transferred to the computer by floppy disk or via a computer network if a networking version of the Imagestore is in use. Numerous image formats can be handled thereby ensuring image analysis from virtually any source. Alternatively, to eliminate disk transfer, the computer (PC or Mac) can be linked directly to the documentation system and a compatible frame-grabbing card (GRAM-IT) fitted to the computer. Once the card and its operating software are installed, an image can be captured directly by the software.

GelBase Pro is the series' entry-level module. This one-dimensional program is specifically designed to analyse ethidium bromide-stained DNA/RNA gels, Coomassie blue and silver-stained gels, and protein gels as well as autoradiographs.

After the image has been loaded and analysis is underway, separate image, track and results windows clearly display the progress of the analysis - each window has its own standardised menu options to manipulate and process the image. The track window displays a series of peaks or a histogram which corresponds to the bands from the track under investigation. Any results are shown in the third window.

Standard tracks can be calibrated either manually or from a user-maintained library, with or without background subtraction. Molecular weight and base pair calculations of unknown tracks can now be made - these are instantly displayed in the results window together with all appropriate data relating to the track's peaks e.g. area, height, position and volume. Hard copies of each window can be made, and if necessary the data can be exported in appropriate formats to database DTP, spreadsheet or word-processing programs.

Gelblot Pro, UVP's two-dimensional analysis package for spots and blots, can initially be supplied as a unified program (GelBase/GelBlot Pro) or added to the GelBase Pro software at a later date. Automatic and manual modes are available for rapid analysis of either individual spots or regions - optical density calibrations are made with ease and all data is shown in the results window. Standard help facilities are available for both types of software.

Pro Series software can run on most IBM PCs and compatibles or Apple Mac systems. For PC users, UVP recommends either a 386 or 486 model with at least 4MB RAM, a coprocessor when appropriate, a hard and floppy disk drive (3.5") - hardware to meet the minimum requirements to operate Windows 3.1. A SVGA colour monitor with a 256 colour display card (1MB RAM) must be fitted. Apple Mac users should employ an LC or better machine with a System 7 operating system and floating

point processor. All software is supplied with a security device and comprehensive user manual including a Quick-Start chapter designed to get the user up and running quickly.

Contact: Watson Victor Ltd, P O Box 1180, Wellington  
Ph: (04) 385-7699, Fax: (04) 384-4651

Offices also in: Auckland - Ph: (09) 579-3039  
Christchurch - Ph: (03) 366-9282  
Dunedin - Ph: (03) 477-7291

circle number 32 on the reader reply card.

## GBC LC1650 ADVANCED HPLC AUTOSAMPLER

The GBC LC1650 Advanced Autosampler has been designed to satisfy the requirements of automated HPLC sample processing in modern analytical and research laboratories. This "all electric" X-Y-Z sample processor features improved performance, sample capacity and reliability with reduced instrument footprint. The LC1650 utilizes a modular stand-alone format with advanced microprocessor control and can be integrated into any existing HPLC setups via I/O contacts. The autosampler's in-built IEEE interface enables the LC1650 to be configured with other GBC LC modules and provides a complete HPLC system with single computer user interface control.

The LC1650 provides extended variable sample volume injection versatility (1 to 200  $\mu$ L) with precision of <0.5% RSD (<5  $\mu$ L injections). This is achieved with the adoption of an advanced sampler metering arrangement consisting of a high resolution stepper motor driven SPSP (*Sapphire Plunger Self Priming*) syringe in combination with a "flow through" needle fluidic path. In addition, the unique SLG (*Spring Loaded Grooved*) format of the autosampler's needle allows accurate needle positioning and side entry of sample, providing the delivery of a precise unprecedented minimum sampler pick-up volume. A 1  $\mu$ L injection can be performed by the LC1650 with only 3  $\mu$ L of sample!

The LC1650's proven "flow through" needle fluidic path design assures high sample recovery and improved cycle time with minimal wastage. In addition, the needle is flushed automatically with the mobile phase internally and eliminates any possible sample carry-over. The simplicity of this elegant design improves instrument ruggedness and eliminates the necessity of complex injection operations.

The LC1650 has been developed to deliver maximum autosampler sample capacity for extended automation and minimized instrument footprint to conserve valuable bench space. The autosampler has a standard sample capacity of 160x1 mL and 2 mL or 112x4 mL vials but is less than 34 cm in width! This increased capacity with reduced footprint is achieved by adopting a rectangular-array vial arrangement and an X-Y-Z robotic sampling probe. This X-Y-Z sampling feature not only provides the users with complete flexibility in sampling but also in the automation of pre-injection sample preparations. An optional external needle wash station can also be fitted when vial-to-vial chemical contaminations need to be minimized. Automatic sample dilution, internal standard addition, solvent extraction and pre-column derivatization can therefore be performed as integral parts of the LC1650's sample processing routines with high precision.

# NEW PRODUCTS

The autosampler uses an "all electric" scheme and utilizes an electrically actuated 6-port high performance injector valve. This simplifies installation requirements by eliminating compressed air and tubing requirements that are associated with the traditional pneumatic arrangement. The LC1650's "open view front access" design permits visual observation of the autosampler's robotic operations while the transparent door protects the compartment from possible external interferences. This arrangement allows fast sample loading and facilitates improved equipment serviceability during routine maintenance with excellent accessibility.

The LC1650's advanced processing capabilities can be readily utilized by the user with its logical instrument control keypad and dual line backlit liquid crystal display. The autosampler has in-built sample processing templates for unattended batch analysis and pre-column sample preparation. Extended automated HPLC sample processing can therefore be conveniently made routine by the LC1650 Advanced Autosampler with high precision and reliability.

Contact: GBC Scientific (NZ)  
P O Box 68330 Newton, Auckland  
Ph: (09) 373-5765 Fax: (09) 360-0683  
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## NEW CAPILLARY COLUMN PHASES FROM J&W SCIENTIFIC

### DB-XLB

This new low polarity stationary phase has been developed for the rigorous demands of highly sensitive mass spectrometers. DB-XLB (eXceptionally Low Bleed) exhibits the lowest bleed levels to provide the "cleanest" mass spectra. Like all J&W's low bleed columns, DB-XLB exhibits exceptional inertness for acidic and basic analytes. DB-XLB's unique polymer formulation provides a low polarity - roughly equivalent to a 14% diphenyl methyl polyisiloxane - and an extended temperature range (to 340/360°C) resulting in a new stationary phase well suited to most GC/MS applications and confirmational analyses. Available in a variety of lengths, inner diameters and film thicknesses, DB-XLB provides the chromatographer with the very latest in low bleed stationary phase chemistry.

### DB-35ms

DB-35ms columns exhibit low bleed for increased signal-to-noise and excellent inertness for active compounds with an upper isothermal temperature limit of 340/360°C. DB-35ms is the best choice for those applications requiring a low bleed, mid polarity stationary phase for trace level analyses. Ideally suited as a confirmation column for applications such as pesticides, herbicides, PCBs, drugs of abuse and industrial chemicals, DB-35ms' higher upper temperature limit increases productivity by allowing the analyst to ramp up the temperature at the end of the chromatographic run to elute semi-volatile sample matrix components thus increasing sample throughput. DB-35ms columns are available in a variety of lengths, inner diameters and film thicknesses.

Contact: Clare Hodgson, Douglas Scientific  
P O Box 45027, Auckland 8  
Ph: (09) 8375447 Fax: (09) 8360668, Freephone: (0800) 735725  
circle number 34 on the reader reply card.

## MALDI-TOF FOR FAST ACCURATE ANSWERS IN MOLECULAR MASS ANALYSIS

Kratos Analytical has developed KOMPACT MALDI in conjunction with Johns Hopkins University. KOMPACT MALDI is a novel range of high performance, matrix assisted laser desorption time-of-flight mass spectrometers aimed at the biotechnology, pharmaceutical and polymer markets. KOMPACT MALDI provides outstanding performance for both routine and research applications.

You have a choice of three KOMPACT MALDI models each of which offers high mass range utilising the latest in time-of-flight technologies. KOMPACT MALDI II is a linear time-of-flight instrument, KOMPACT MALDI III is a dual instrument with both linear and reflectron capabilities. The reflectron adds the benefit of higher resolution and increased mass measurement accuracy. New exciting developments in PSD (Post Source Decay) technology will be available for the MALDI III later this year.



The KOMPACT MALDI I is a small footprint bench-top model. It has been designed for routine laboratory use and its intuitive user interface is ideal for the non-spectrometrists as well as the specialist, so, as with the other KOMPACT MALDI models you don't need to have a dedicated user. The KOMPACT MALDI I offers exceptional performance at a substantially lower cost. It occupies even less bench space than the other KOMPACT MALDI systems. KOMPACT MALDI systems are in use in laboratories around the world. Contact us for a reference users list.

All KOMPACT MALDI systems offer unrivalled sample handling facilities, high sample throughput, ease of use, and fast on-line data processing based on workstation technology. They bring quality MALDI-TOF MS systems into the reach of organisations who previously felt they could not afford this technology or could not support it with a dedicated specialist operator.

Contact: Clare Hodgson, Douglas Scientific  
P O Box 45027, Auckland 8  
Ph: (09) 8375447 Fax: (09) 8360668  
Freephone: (0800) 735725  
circle number 35 on the reader reply card.

# NEW PRODUCTS

## ADVANCED HPLC COLUMN TECHNOLOGY PROVIDES HIGH RESOLUTION FOR POLAR, ACIDIC AND BASIC COMPOUNDS

Described in a new 8-page brochure, Phenomenex PrimeSphere™ columns combine ultra-pure spherical silica with an innovative surface deactivation strategy that eliminates non-specific interaction between the base silicas and the sample components. Mixtures of acidic, basic and zwitterionic compounds resolve into sharp peaks on these high efficiency columns without the need for extreme mobile phase conditions or competing acids or bases. PrimeSphere™ columns are manufactured for both analytical and preparative HPLC separations, in 5, 10 and 15 µm materials. Preparative columns offer high loading capacities while precisely duplicating the bonding chemistries of the analytical packings. PrimeSphere is available in silica, C<sub>8</sub>, ODS-MS and ODS-HC in 120 Å pore size and in C<sub>4</sub> and C<sub>18</sub>-MC in 300Å pore size. Bulk material and packed columns in sizes ranging from 1 mm to 100 mm IDs can be purchased.

Contact: Chromspec Distributors Ltd  
P O Box 31180 Milford, Auckland  
Ph: (09) 4791442 Fax: (09) 4791442  
circle number 36 on the reader reply card.

## NEW ADDITIONS IN CARBOHYDRATE COLUMNS

Supelco offers a complete line for the separation of mono-, di-, tri-, and oligosaccharides. New to the line are the SUPELCOGEL Ca column, and the SUPELCOGEL Ag column.

The SUPELCOGEL Ca column, a calcium-loaded ion exchange column, provides excellent separations of monosaccharides and sugar alcohols. Di-, tri-, and oligosaccharides are separated by class, and the column is also useful for the separation of sugars in high fructose corn syrup.

The silver-loaded SUPELCOGEL Ag column provides rapid oligosaccharide separations, and is particularly well suited for analyses of carbohydrates in beer, corn syrup, and hydrolyzed starch products.

Contact: Chromspec Distributors Ltd  
P O Box 31180 Milford, Auckland  
Ph: (09) 4791442 Fax: (09) 4791442  
circle number 37 on the reader reply card.

## NEW HPLC PRODUCTS FROM UPCHURCH SCIENTIFIC

### *Flexible Plumbing*

The Upchurch Scientific Spiral \*Link tubing loops provide you with a wide variety of inert PEEK tubing for use as an HPLC sample loop or; a convenient, self-adjusting tubing coil that allows you to easily move your pump, valve, column and detector when a system configuration change is needed.

These Spiral \*Links come in an assorted range of the more common volumes to meet most laboratory needs. They are made from Upchurch's own in-house extruded PEEK tubing which is held to the most exacting tolerances on the market today.

Upchurch Scientific's Spiral \*Link loops can be connected into your system easily by using any fingertight or flangeless fittings for 1/16" OD tubing.

### *Supercharged!*

Upchurch Scientific's 1995 Chromatography Fittings and Accessories Catalogue and Technical Reference Manual is available NOW! Featuring the newest, most updated products for use in any brand of system, manufactured to the highest quality. As in past years the catalogue features technical information, materials solvent compatibility, detailed full colour illustrations, charts, complete product descriptions and photographs, making it an invaluable resource for the chromatographer.

To receive your free copy of the most dynamic, energised and complete catalogue of laboratory products for HPLC and fluid transfer fittings in the industry today...

Contact: Clare Hodgson, Douglas Scientific  
P O Box 45027, Auckland 8  
Ph: (09) 8375447 Fax: (09) 8360668  
Freephone: (0800) 735725  
circle number 38 on the reader reply card.

## LIKE TO LEARN MORE ABOUT THE BENEFITS OF GILSON AUTOMATED SAMPLE PREPARATION?

You're probably being asked to do more with less ... fewer people and fewer resources. One way to improve your lab's productivity is through automated sample preparation.

Pepsi-Cola Co., USA, were spending considerable time in beverage flavour compounding, preparing small-scale samples for flavour optimization. The samples had to be extremely accurate and consistent. Manual sample preparation posed several problems, including accidental omission of one or more ingredients.

The company installed a Gilson automated liquid handler to automate sample preparation. The robotic sampler treats each sample identically and doesn't skip ingredients, for totally reproducible results.

New Jersey - based McNeil Specialty Products wanted to analyse a high-intensity sweetener in various food products, including a low-calorie ketchup.

Originally the research involved solid phase extraction as the sample cleanup technique. But it was a rather lengthy process and required up to two days for sample preparation and analysis. The laboratory then switched to ultrafiltration which reduced the sample preparation time to three hours. But this was still too time-consuming.

So, McNeil Specialty Products installed Gilson's ASTED XL System. ASTED XL technology is a unique, patented combination of dialysis and trace enrichment which speeds up and streamlines complex sample cleanup. The result? Sample preparation and analysis was reduced to less than one hour per sample. The system saves time and it runs unattended, another benefit for this food ingredients manufacturer. The laboratory also reduced the amount of solvent used as well as its solvent disposal costs.

# NEW PRODUCTS

These applications are just two examples of how Gilson instruments can improve preparation of food and beverage samples. Also available from Gilson is an applications report devoted to food analysis. Included in this 8-page report are two applications where automated solid-phase extraction is used in detecting pesticide levels in fruits and vegetables, and in a cleanup procedure of polychlorinated dibenzodioxins and dibenzofurans in milk. Two other reports demonstrate the use of the ASTED XL System in QC analysis of orange juice, dairy products, soft drinks and fermented beverages.

To receive a copy of the 222 XL brochure, the ASTED XL System brochure, or the Food Preparation and Analysis Application Report,

Contact: John Morris Scientific Ltd  
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To determine the density of solids, the density values of the auxiliary liquids "distilled water" and "ethanol" over a temperature range of 15...30°C are stored in the application software and can be called up at a keystroke. The user also has the possibility to enter the density of any auxiliary liquid.

For the weighing of a solid in air, one of the two arms of the weighing pan can be loaded in a convenient manner. For weighing of the solid in the auxiliary liquid, two holders of diameter 42 mm are available. These can also be used to weigh solids which float in the auxiliary liquid.

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# NZIC NEWS

## FUTURE OPTIONS FOR THE NZIC

*William A Denny, President NZIC*

As I noted in letters to the editor in *Chemistry in New Zealand* (November 1994 and January 1995), a strategic review committee has been set up to review the role, structure and function of the NZIC. The committee met for the first time during and after the Council meeting on February 22nd 1995, and we now offer this background summary to members of the NZIC.

### **Why the committee was set up.**

There are several good reasons why a review of the NZIC is timely.

- (i) We have a declining and ageing membership.
- (ii) World-wide, but in New Zealand especially, the last few years have seen drastic changes in the scientific environment.
- (iii) There have been a number of calls, from both individuals and branch representatives, suggesting that changes be made to the goals, structure and management of the Institute.

### **How the review committee was set up.**

This was done late last year by me, following discussions with the two Vice-Presidents (Nath Pritchard [ECNZ] and Rob Whitney [Coal Research Ltd]). From the point of both representation and continuity, I felt it was important that the three elected officers be members of the review committee. In addition, we co-opted Gill Norris [Massey University; former chair of the Manawatu Branch] and Doug Wright [former NZIC president]. I believe the review committee members have a suitably broad range of backgrounds and experience. The committee's brief is to consult as widely as possible, both within and outside the NZIC membership, on the role and function of the NZIC in the year 2000.

### **What the review committee has done so far.**

We felt it was important to begin with as few biases or directives as possible. In letters to the editor in *Chemistry in New Zealand* (November 1994 and January 1995), I requested both written and verbal submissions on any aspect of the NZIC. Convenors of Specialist Groups were also asked for their views. I would like to thank everyone who responded to this deliberately vague request. All of these submissions were circulated to members of the committee, together with other documents such as the most recent Presidents' reports. At the 22nd February 1995 Council meeting, branch representatives also made submissions on behalf of their committees. The review committee then met separately after the Council meeting. It was agreed that the NZIC faced four broad possible options, listed below. Note that these options are not listed in any perceived order of merit, but rather in rough order of the degree of change which they would entail. Perhaps more importantly, they also broadly represent different basic philosophies about the role of the NZIC.

### **1. Refine and adapt the existing structure.**

This course would accept that the existing goals of the NZIC are still broadly correct. These goals, which every member has a copy of (somewhere), are summarised as follows. The NZIC is not a professional body in the sense that it does not control registration to practice, cannot enforce a code of ethics, and does not offer a personal advocacy for members in disputes. However, in addition to the "personal" services to members, including fellowship, networking and scientific exchanges (significantly now through the Specialist Groups) and peer recognition via awards, the NZIC also performs a number of more "corporate" services on behalf of members, notably provision of education in chemistry and submissions to the Government on chemical issues. All of these services, and especially the latter, require a significant central secretariat. The vast bulk of the Institute's income is from membership fees and, with about 900 fee-paying members, it is probably at the lower limit of viability as an organisation in this format.

If this option is taken, several issues need to be addressed. These include (but probably are not limited to):

(a) *The role of the Specialist Groups within the Institute.* Several of these are functioning extremely successfully, clearly fulfilling a major role for members in terms of scientific exchange, and also attracting a large number of non-members. We need to extend and support their function, while at the same time seeking benefits for the NZIC as a whole.

(b) *The workload of the secretariat.* We need to find out what members think about the relative importance of "personal" and "corporate" services (to use the terminology defined above), and provide appropriate direction to the secretariat based on this.

(c) *The membership fee structure.* Consideration could be given to making this simpler, with perhaps just two grades (student member and member). Fellowships etc. could still be granted, but as internal honours by the Institute.

The overall aims of such changes would be to provide a more relevant and responsive organisation. To the extent this resulted in increased membership, fees could be reduced somewhat.

### **2. Convert to a much less centralised "scientific society".**

If members feel that "personal" services to members are of much more importance to them than "corporate" services on behalf of members, we could consider moving towards being a discipline-based "scientific society" in the manner of several other groups in New Zealand (e.g., the Biochemical Society). Presumably the Branch and Specialist Group structures would remain (indeed would become more prominent), and *Chemistry in New Zealand* in its present format would probably not be affected. However, the central secretariat would be reduced to a "house-keeping" function and there would be a single membership fee, much lower than at present.

### **3. Seek amalgamation with a number of other smaller groups to form a much larger organisation based more broadly in science, rather than chemistry.**

An extension of the above option, but seeking an even broader (less discipline-based membership). This could result in lower costs for any central secretariat, with lower fees. The option would require much negotiation with other groups, and may be

best seen as a possible longer-term development along the lines of option 2.

**4. Recognise that changing times and special conditions in New Zealand work against the continued existence of the NZIC, and seek to wind it up in an orderly manner.**

No explanations needed.

**What the review committee intends to do in the next six months.**

The immediate role of the review committee will be to continue to gather views both from members and others on the role and function of the NZIC. It is hoped that the above discussion of "options" (which are really representations of different basic philosophies about the Institute) will assist people in commenting. I will be discussing this with members during my tour of the branches in March, and reporting back to the review committee. We will then decide whether it is necessary to seek further input from members in the form of some type of questionnaire; if so, this will be circulated via *Chemistry in New Zealand*. We will then seek to reach a consensus on what members wish concerning the future role of the NZIC in time to report to the May meeting of the Standing Committee of Council. This report and the Standing Committee discussions on it will be published as soon as possible in *Chemistry in New Zealand*.

The review committee will then prepare a report for the August Council meeting, containing its recommendations for the structure and management of the NZIC to ensure that the organisation can achieve the goals set by the membership. This report and the Council discussions on it will be published as soon as possible in *Chemistry in New Zealand*.

## COUNCIL NEWS

The February meeting of Council was held in Wellington and in spite of the current difficulties of Air New Zealand most members managed to attend. It preceded Branch submissions to the Strategic Review Committee. The following are the highlights from the Council meeting.

### FINANCIAL

Some considerable time was spent discussing the Treasurer's Report which indicated that there was a potential deficit of some \$15,000.00 at the end of the financial year. This is because of the slow return of subscriptions. The Secretariat was instructed to chase these up and make personal contact if necessary. On the expenditure side the projected budget was revised to give a small surplus for the ensuing financial year.

Subscriptions will not be increased - the same level for six years now.

A request from Otago Branch for financial support from the Overseas Visitors' Fund for a visiting speaker, Dr Gerloch, was approved. Dr Malcolm Gerloch from Cambridge University is an internationally recognised expert on magnetism, ligand-field theory and coordination chemistry, and will visit during March 1996.

Mr Tim Oughton MNZIC, the winner of the British Council/British Airways NZ Science Teacher Award will be a guest at the Edinburgh International Science Festival. Assistance was given for him to attend the RSC Conference on Chemical Education.

### MEMBERSHIP

New members admitted are reported separately below. It is with regret that we report the deaths of the following members:

Fletcher, Edwin Charles Geard, MNZIC, Manawatu Branch  
Swaney, Alan Harding, MNZIC, Wellington Branch

### HONOURS

Mr D J Hogan Hon FNZIC, received the Queens Service Order and Mr T J Sprott became an Officer of the Order of the British Empire in the New Year Honours.

### PRIZE WINNERS

The Shell Prize for Industrial and Applied Chemistry has been awarded to Dr Neil Milestone of Industrial Research Ltd, Lower Hutt.

The Pacificchem '95 Student Travel Awards were won by Ms R M Lorimer (Auckland University) and Mr D C W Reid (Massey University). The travel awards have been jointly sponsored by the Ministry of Research, Science and Technology and NZIC. The assessors made the comment that all of the student submissions were of an exceptionally high standard, and all were eminently suitable for presentation at the Pacificchem Congress later this year.

### PUBLICATIONS

"*Chemistry in New Zealand*". Dr L James Wright has been appointed to the Editorial Board, replacing Dr Jim Metson who is about to go on sabbatical.

"*Chem NZ*". Our contract to supply the Royal Society of Chemistry with "*Chem NZ*" has been renewed for another year. It is distributed with RSC's Schools Publications Services.

"*Chemical Milestones*". Most of the material for this publication is now to hand, and we are now seeking financial support for publication.

### SUBMISSIONS

The Institute has made a submission to the select committee on the Hazardous Substances and New Organisms Bill and this is reported elsewhere. Council noted the inputs from Keith Hunger, Brian Robinson, Norman Thom, Philip Tse and Jim Waters and the collation by Norman Thom.

A copy of the submission is available from Alan Turner, the Executive Officer.

## SCIENCE OF THE NZ ENVIRONMENT COMMITTEE

The Royal Society of New Zealand recently established this Committee to advise it on matters of concern in the environment. Four NZIC members have been asked to join - Dr Craig Stevenson (ESR), Dr Michael Taylor (Ministry of Health), Mr Norman Thom (University of Auckland) and Mr Jim Waters (Ministry of Health).

### NZIC MEMBERSHIP UPDATE

#### Fellows Admitted Recently

Auckland	CLARKSON	Thomas Stephen
Manawatu	SHAW	Gary John
Canterbury	WILLIAMSON	Bryce Elliot

#### Members Admitted Recently


Auckland	MANSFIELD	Stephen Michael
Manawatu	SKIBBE	Ute
Wellington	GLADKIKH	Olga
Otago	MUNASIRI	Bhadra

#### Associates Admitted Recently

Auckland	MIHEC	Damian Francis
Wellington	AMMUNDSEN BOWLES GRIBBLE PILOTTO	Brett Graeme Richard Kerr Craig Peter Paul Joseph
Otago	DAVEY KELLY McLEAN	Tim Warren Robert Carlene

#### Local Member Admitted

Waikato	BURTENSHAW	Greg James
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# NZIC BRANCH NEWS

## Otago Branch

The Marine/Analytical Group of the Department of Chemistry, University of Otago have just taken delivery of a new Flameless Atomic Absorption Spectrometer. It is a Perkin-Elmer Model 4100ZL with FIAS-400 flow injection accessory. This machine replaces an older less reliable AA and compliments the ICP-OES to greatly enhance the groups metal analysis capabilities.

Benefits of the 4100ZL include a significantly improved sample throughput due to excellent control of furnace conditions giving better reproducibility. The furnace tube design means unknown samples can be compared against a simple standard with much more confidence than before. The ZL in 4100ZL indicates that longitudinal Zeeman background correction is used. This employs a large magnetic field which affects the atoms but not the background and hence allows the accurate analysis of elements in very complicated samples - e.g. lead in blood, chromium in seawater, etc.

The FIAS-400 accessory can be used for producing hydride gases of elements such as arsenic, selenium, tin and germanium. It can also generate gaseous mercury. The gases are passed through an electrically heated "T-cell" for detection by the spectrometer. The FIAS-400 can also perform "tricks" like on-line pre-concentration and ion-exchange for matrix removal.

The Nutrition Department are interested in selenium and will use the new machine to supplement their existing techniques. The 4100ZL will be available for commercial work.

## Manawatu Branch

Congratulations to Christine Mahy, awarded the NZIC Prize for 300 level Biochemistry, and to Steven Kennedy, awarded the NZIC Prize for 300 level Chemistry. By the time you read this they will have received their prizes from NZIC President Bill Denny at our annual students' meeting on 14 March.

In the excitement of last year's AGM we missed a nomination to the committee. So apologies and welcome to Harold Percival, Harold works at Landcare Research and brings a wealth of experience to our committee from his time as President of the NZIC.

Terry Thomas of the Dairy Research Institute has been promoted to Deputy Chief Executive. He will be responsible for the day to day operation of DRI leaving Chief Executive Kevin Marshall free to concentrate on worldwide dairy industry research strategy.

Past branch chairman Gill Norris contributed to the first meeting of the Strategic review committee and reports that good progress was made. If you have any comments about the future direction of the NZIC please pass them to any member of the committee.

Massey University has announced the candidates of the short-list for the Vice Chancellor's position. They are:

<i>Prof. Robert Anderson</i>	Dean, Faculty of Agricultural and Horticultural Sciences, Massey University.
<i>Prof. Jack MacDonald</i>	Vice-President Academic, University of Guelph, Ontario.
<i>Dr James McWha</i>	CEO, Horticultural and Food Research.
<i>Prof. Charmian O'Connor</i>	Deputy Vice-Chancellor, University of Auckland.
<i>Prof. Ian Reid</i>	Deputy Vice-Chancellor, Curtin University of Technology, Perth, Australia.

The candidates will be interviewed at Albany and Massey from 3-10 March 1995. It is unlikely that the successful candidate will be known for some time.

Grant Boston

## NZIC PRIZES AND AWARDS

### SGS PRIZE

This prize of \$1,000.00 and a plaque has been donated by SGS (New Zealand) Ltd.

1. The prize shall be awarded to a member of the Institute who, in the opinion of the Council, has made a significant contribution to some branch of chemical science, the contribution to be judged by research work published during the five years immediately proceeding 30th April in the year of the award.
2. Applications by members or nominations which may be submitted by Branch Committees or individual members must be received by the Executive Officer, P O Box 12347 Wellington by 30th April each year and must be accompanied by copies of papers presented in support of the entry. The Council itself may nominate candidates for the award.
3. A nomination or application, once made, shall stand for five years and material which fails to satisfy clause 1 shall automatically be deleted and additional material may be presented at any time.
4. If, in the opinion of Council, there is no candidate of sufficient merit, the Council may refrain from making the award.
5. The prize shall be presented at the Annual Conference of the Institute or at a meeting of the Branch to which the prize-winner belongs.
6. A member to whom the prize has been awarded shall not be eligible for re-nomination.

### SHELL PRIZE FOR INDUSTRIAL AND APPLIED CHEMISTRY

A prize of \$1,000.00 and a certificate will be awarded annually by Shell New Zealand Holding Co. Ltd. to further the recipient's studies in industrial chemistry and to commemorate the achievement.

1. The prize will be awarded for meritorious achievement in the field of industrial or applied chemistry.
2. The prize will be restricted to financial members of the New Zealand Institute of Chemistry of any grade of membership. In the case of joint work the prize may be shared between two or more members.
3. Applications should include a written statement of the industrial or applied chemistry activities or achievements of the candidate(s) and their significance in terms of improved technology, new products or other benefits to industry or the community. Supporting documents and publications may be submitted with the application and **will be held to be confidential to the assessors**. If possible, the value of the work should be attested by an accompanying statement from the manager or directors or head of the organisation. There is no limit on the period of time over which the work was carried out.
4. Applications for the prize may be made by individual members or nominations may be made by Branch Committees or by corporate members of the Institute. A nomination or application, once made, shall stand for five years.
5. Two or three assessors will be appointed by the Council of the Institute to consider the applications and make recommendations. The final decision on the award will be made by the Council. Council reserves the right to make no award in the absence of a suitable candidate.
6. Applications or nominations must be received by the Executive Officer, P O Box 12347, Wellington by 30th April each year.

### AWARD FOR CHEMICAL EDUCATION

Council of the Institute has established an award for chemical education consisting of a certificate and a prize of \$250.00. The award is to be made in compliance with the following rules:

1. The award will be made annually unless, in the opinion of Council, there is no candidate of sufficient merit.
2. The award shall be made to a person who, in the opinion of Council, has made an important contribution to Chemical Education in New Zealand. (Note: the award will normally be made to a secondary teacher actively involved in teaching chemistry.)
3. The award shall consist of a certificate and a prize of \$250.00.
4. The award is not restricted to financial members of the Institute.
5. Application for the award may be made by individuals, or nominations may be made by any Branch Committee or by any individual financial member of the Institute.
6. Applications or nominations must be received by the Executive Officer, P O Box 12347, Wellington by 30th April in the year of the award. Each application or nomination must include a full curriculum vitae and two independent supporting statements from referees commenting on the educational activities of the candidate and their significance to chemical education.

# DESIGNING OPTIMIZED INDUSTRIAL PROCESS ANALYSERS FOR CLOSED LOOP CONTROL

*By Bernard Grevesmuehl, Cynthia Kradjel and Hanno Kellner  
Bran + Luebbe, Germany*

Manufacturers today are looking closely at ways to optimize "quality" and increase process efficiency while reducing manufacturing costs.

Near infrared (NIR) technology is ideally suited for this challenge. NIR provides manufacturers with rapid and reliable in-process analysis. Thousands of systems have been installed in the food, chemical, pharmaceutical and agricultural markets.

For more than ten years, near infrared has successfully been applied to at-line process analysis. Rugged and easy-to-operate filter analysers are traditionally located in the control room. Process operators can then "grab samples" and obtain results in less than one minute. There are many practical advantages to using at-line filter systems. Products from many lines can be run on one system and since there is no direct process interface, installation, operation and maintenance are quite simple.

However, manufacturers are now striving to achieve on-line closed loop control. In these cases, the benefit of obtaining continuous measurements is worth the effort required to automate the analysis.

This paper will discuss actual installations of liquid and solid applications. The Bran + Luebbe InfraAlyzer 600 (IA600) is the basic unit used in all examples. In some cases, the system is used in the non-contact mode and in other examples it is used in the contact mode.

The optical unit is based on patented Reference-Sample-Dark (RSD) optics. This optical configuration is called 'precision scanning' because for each wavelength measured, several real-time readings of the gold-plated integrating sphere and the dark current are also measured. The design enables a constant check against changes in the environment or optics of the system.

The wavelengths are achieved by high-precision bandpass filters which are mounted on a filter wheel. The optical head is a separate unit from the electronic unit. The optics module can be connected either directly to the liquid cell or sampling device or mounted above the production line. The electronic unit fits into a standard 19-inch rack.

The IA600 has been used on liquid, solid and semi-solid applications.

## Liquid Applications

The first Bran + Luebbe on-line process liquid analysis systems were installed in the mid 1980s.

The InfraAlyzer 600 is successfully used in the sugar processing industry. Calibrations have been developed in co-operation with the University of Ferrara in Italy for the determination of Brix value and degree of polarization in process liquids, including raw, thin and thick juices.

These parameters are used to optimize efficiency when processing the beet to crystalline sugar. The Brix value is related

to the total solids content. The degree of polarization is related to the ratio of sugars, such as saccharose, glucose and fructose.

The juice is pumped through a stainless steel pipe to a suitable point in the plant and then thermostatted at 50°C. The juice flows continuously through a small tank to ensure a homogenous stream and a constant load. Pneumatic valves, which are controlled by a PLC are used to direct the liquid to the thermostatted liquid manifold for NIR analysis and then to discharge. An automatic rinsing stage takes place.

Sugar manufacturers can now easily blend process streams to achieve the "target value" every time.

Another on-line process liquid measurement system that operates without "human interface" in a closed loop control process is installed in the brewing industry. The system is used to control a blending station by a PLC where water is continuously added into a high gravity beer line.

## Solid Applications

Solid sampling systems can be either contact or non-contact. Each has its advantages and disadvantages. In general, contact analysers require more complicated sample interfacing and are not easily adaptable to pasteous products. However, they are ideally suited for fine powders. They can often achieve very high levels of precision and accuracy because the sample is presented in a very reproducible fashion and process variables are controlled. Non-contact systems are typically mounted above a production line. This is practical for sticky or adhesive products. However, the system will be susceptible to variations in the distance of the sample from the analyser.

The earliest contact system for powders was developed in conjunction with the Federated Milling and Baking Research Association (FMBRA) in the UK. Flour is diverted through a slip stream pipe and automatically pressed against the window. The sample is stationary while analysis is taking place, and then sent back to the line with a compressed air line. The flour is measured for moisture and protein. When necessary, the system signals a valve to open and automatically add gluten to the flour to increase the protein content.

Fat, moisture and protein are critical parameters in milk powder processing. Since milk powder can be adhesive, Bran + Luebbe has developed a non-contact sampling approach for this application.

The optical module is fitted into a compact sampling system and mounted on a bypass pipe after the main milk powder fluid bed. The powder travels to a point underneath the optics module via a magnetic vibrator. A uniform density and constant distance from the optical window is achieved via precise setting of the magnetic vibrator. The flow stops during the measurement cycle. When analysis is completed, the sample is returned to the main line through a spiral conveyor. Fat and protein are determined to an accuracy of  $\pm 0.1\%$  and moisture is measured to an accuracy of  $\pm 0.02\%$ . Approximately 60 samples are

analysed per hour. The sample temperature ranges between 18 and 22°C.

The last example illustrates the use of the InfraAlyzer 600 for the analysis of fat in cream cheese. The optics module is mounted vertically, reading cream cheese at 80°C as it comes out of a pressure stabilized bypass and runs down a spade-like dispenser. The optical module is sealed against dust and water. The electronic unit is located in a control room and is connected to the PC for statistical process control. The fat content ranges from 46-53% with an accuracy of  $\pm 0.25\%$ .

There are many other applications of the InfraAlyzer 600 for on-line NIR process analysis, including moisture in textiles, additives to thermoplastics and fat and protein in meat.

For applications that require full spectral scanning, Bran + Luebbe has introduced two High Performance Crystal Spectroscopy Systems: the InfraPRIME, (Process Integrated Monitoring Equipment) based on the technology of Acousto-Optical Tunable Scanning (AOTS) and the InfraPROVER, a polarizing interferometer optimized for the near infrared region of the spectrum.

Bran + Luebbe has over 15 years experience with NIR process analysis and over 70 years experience with dosing, weighing electronics and process systems, making Bran + Luebbe a strong partner for implementation of closed loop process control.

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## 10TH NATIONAL CONVENTION RACI

## The Royal Australian Chemical Institute announces the 10th National Convention

### "Chemistry Serving Society"

Adelaide Convention Centre, South Australia

27 September - 2 October 1995

### Expression of Interest and Call for Papers

The Organising Committee has the Convention program in place and is now calling for Papers and for Expressions of Interest from those interested in attending the Convention. The following RACI Divisions are participating formally in 10NC:

Analytical, Chemical Education, Electrochemistry, Environment, Industrial, Inorganic, Medicinal and Agricultural, Physical, Polymer, Solid State and The History and Archives Committee.

*All chemists, including those with interests in Divisions not formally represented, are encouraged to contribute to these programs.*

Full Call for Papers details and Expression of Interest Forms are in a pull-out centre section in the November 1994 issue of *Chemistry in Australia*. Or available from Alan Turner, NZIC Executive Officer,

P O Box 12-347 Wellington. Phone: (04) 473-9444, Fax: (04) 473-2324

**Register early and win  
Return your  
Expression of Interest Form  
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at \$60 each.**

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o te Ika a Maui*



### CHAIR OF CHEMISTRY

Applications are invited for a Chair of Chemistry, available from 1 February 1996. Applicants should have an excellent teaching record and an international reputation for outstanding research in any area of Chemistry, although preference may be given to those with a research interest in Inorganic, Physical or Materials Science. The successful applicant will make a substantial contribution to the teaching and research activities of the Department of Chemistry, including a commitment to teaching introductory courses to first year students, and will be expected to provide high quality academic leadership. Further enquiries may be made to the Chairperson, Dr David Weatherburn (tel: 0-4-471 5335; fax 0-4-495 5241; email: David.Weatherburn@vuw.ac.nz).

Salary range for professors is currently \$80,080-99,840 per annum.

Conditions of appointment and method of application are available from the: **Appointments Administrator, Victoria University of Wellington, PO Box 600 Wellington (fax: 0-4-495 5238; email: appoint@vuw.ac.nz).** Closing date: 15 May 1995.

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# WANTED

The Historical Studies Group of the Geological Society of New Zealand is researching the life of Dr John Robert Don.

In the early 1880s Dr Don taught in both Chemistry and Geology at the University of Otago and in 1896 he became the second person to be awarded a D.Sc from the University of New Zealand and the first in Chemistry. His thesis for the degree, 'On the Genesis of Auriferous Lodes from a Chemical Point of View', was published in a condensed form in the *Transactions of the American Institute of Mining Engineers* in 1898.

Dr Don was an unsuccessful applicant for the foundation chair of Chemistry and Physics at Victoria University College, Wellington in 1898. He was Rector of Waitaki Boy's High School from 1897 to 1906 after which he seems to have 'faded out', spending the rest of his life until his death in 1919 as a school inspector in Dunedin.

If anyone can provide any information or leads on Dr Don would they please make contact with A. P. Mason, Convener Historical Studies Group, Geological Society of New Zealand, 65 Argyle Street, Herne Bay, Auckland 1002.

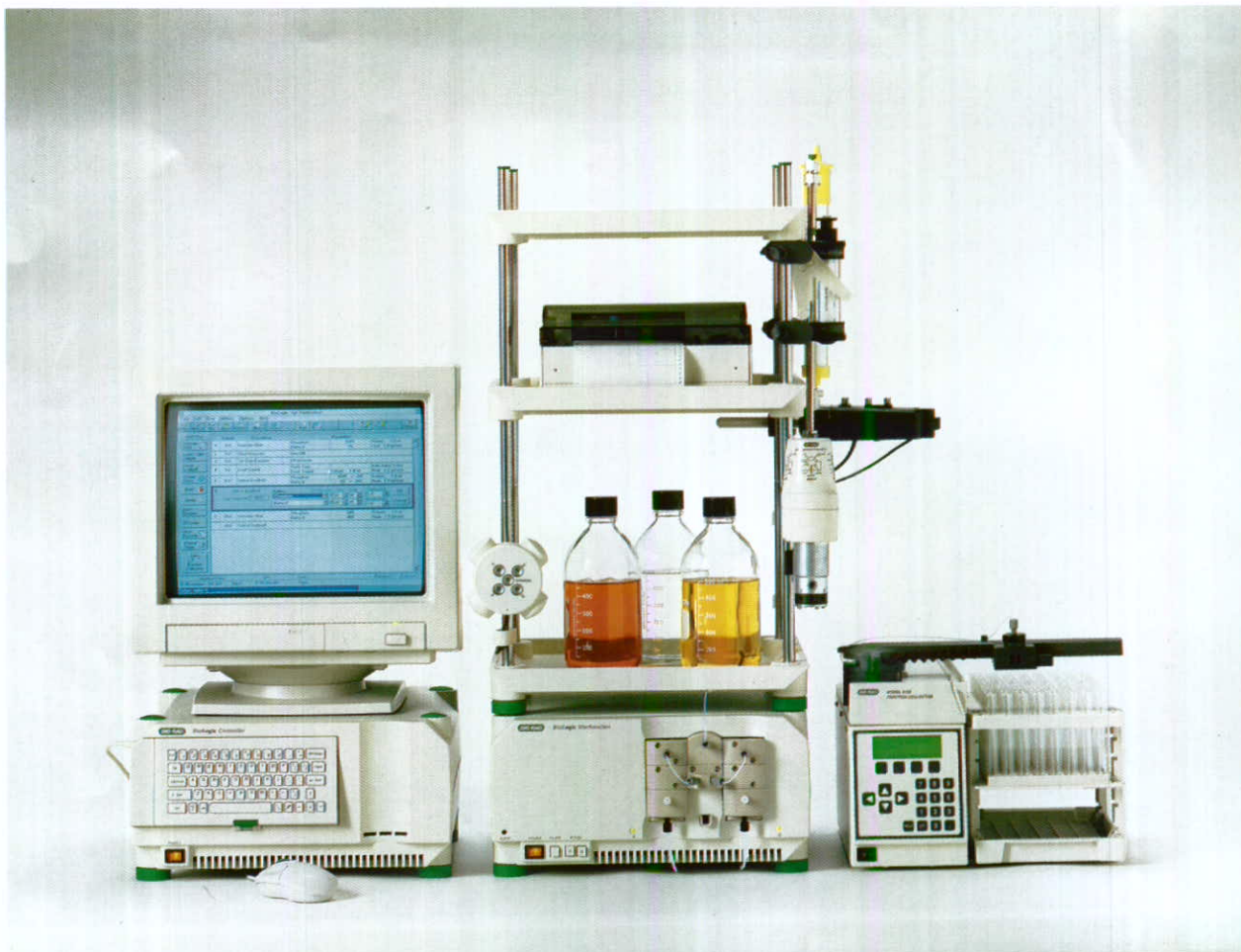
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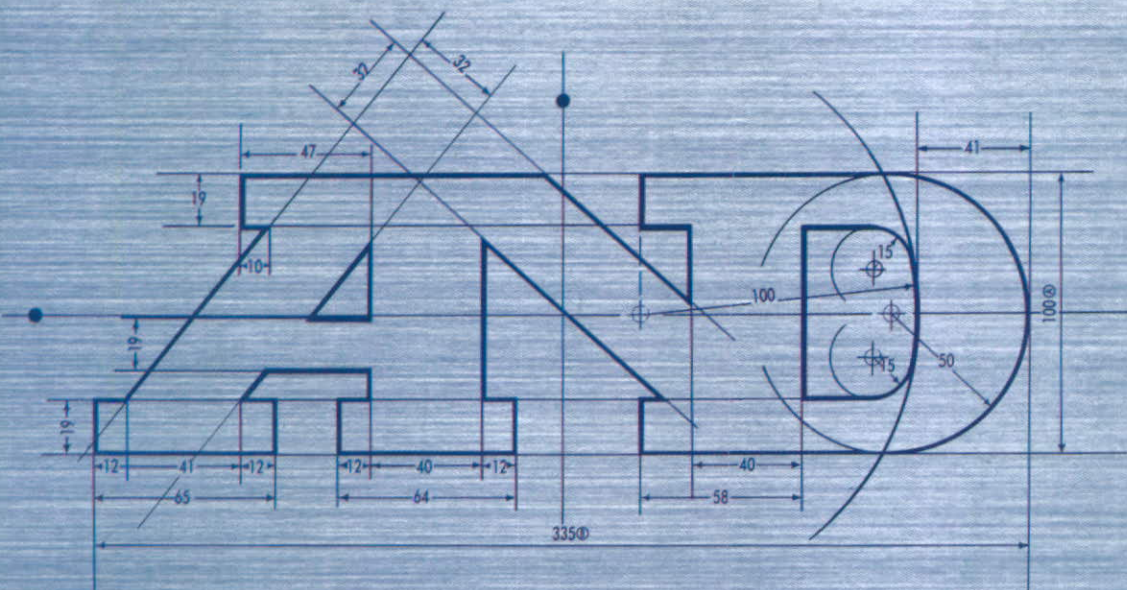


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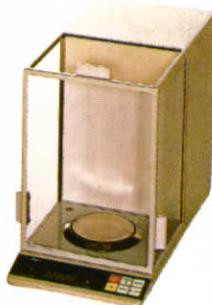
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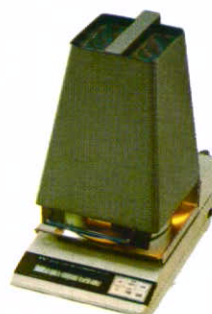
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