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# Chemistry

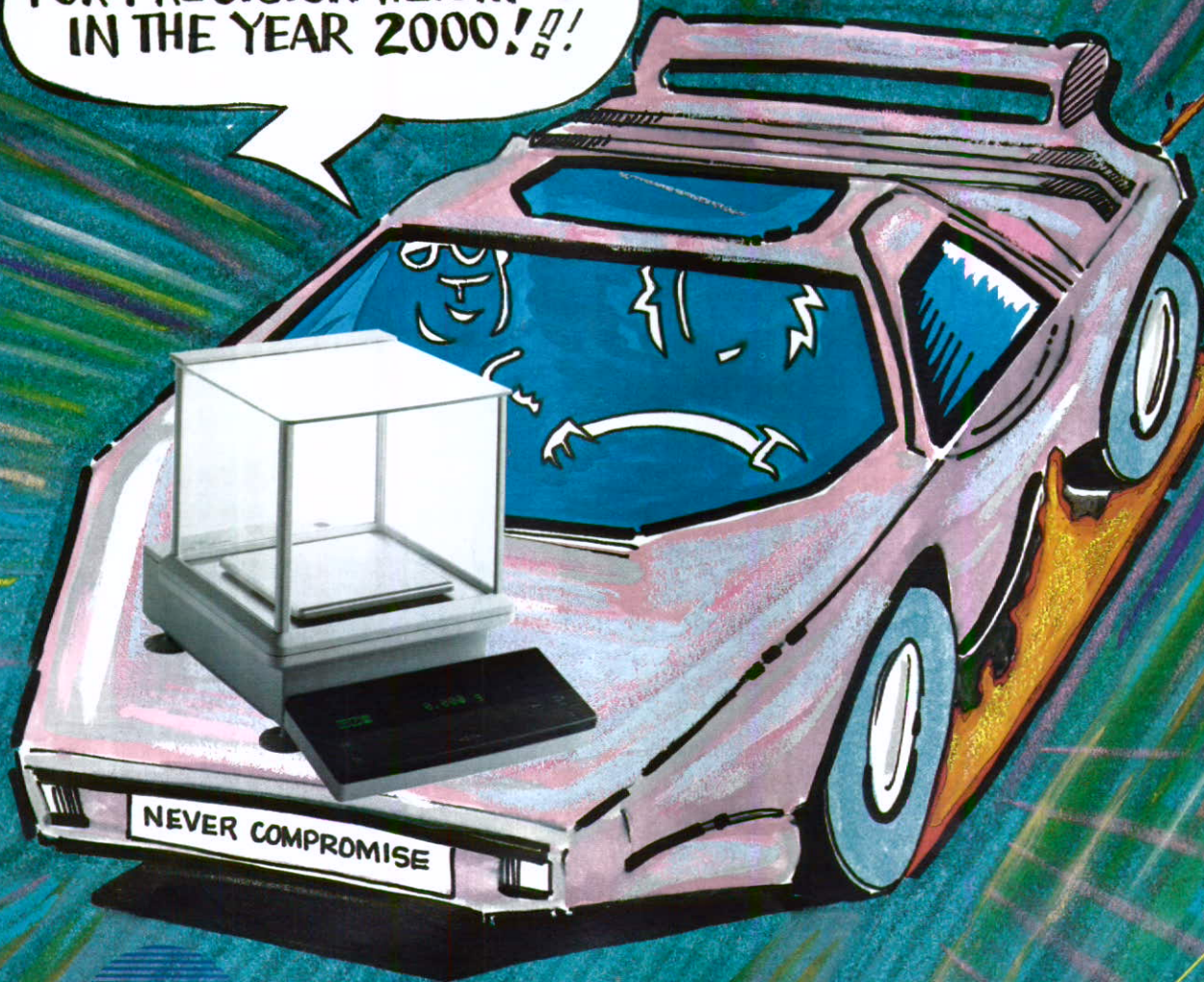
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For further details see the cover story on page 2



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## COMING UP ...

July 1994 - Focus on Chemical Pathology,  
Forensics, Clinical Chemistry

September 1994 - Focus on Forestry, Timber,  
Pulp and Paper

Deadline for material:  
10th of the month of publication

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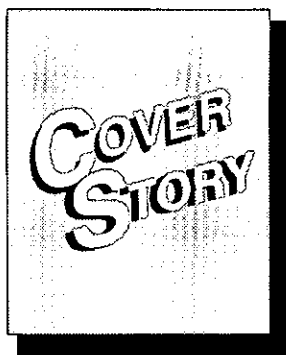
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## JAMES METSON NAMED CO-RECIPIENT OF 1993 TMS LIGHT METALS AWARD

James Metson, a Senior Lecturer in the Department of Chemistry and Director of the Research Centre for Surface and Materials Science at the University of Auckland, was chosen co-recipient of the Minerals, Metals and Materials Society's (TMS) Light Metals Award. The award is presented for a technical paper, published in the preceding year's volume of the Light Metals proceedings, which exemplifies the application of science to the solution of a practical problem in light metals technology.

Dr Metson received his PhD in chemistry from Victoria University. His current research interests include adsorption and adhesion at metal oxide surfaces, catalysis on modified metal/metal oxide surfaces and the further development of surface analytical methods, particularly secondary ion mass spectroscopy. He has published over 40 papers, a similar number of technical reports, and has received two patents.

Dr Metson was recognised for his work on the paper "Influence of Petroleum Coke Sulphur Content on the Sodium Sensitivity of Carbon Anodes" which he co-authored with S M Hume, R Terry, K Baker, Raymond C Perruchoud, and Werner K Fischer.

The Light Metals Award was presented at the 1994 TMS Annual Meeting and Exhibition in San Francisco, California, February 27 - March 3, 1994.

## DOW ELANCO CHOOSES NEW PLYMOUTH

Chemical company Dow Elanco has closed its Sydney manufacturing plant and will base its Australasian manufacturing on its site in New Plymouth. This is expected to double the output of the New Plymouth plant.

## COBALT-60 IRRADIATION PLANT IN TOKOROA?

Recent announcement of plans to build a Cobalt-60 irradiation plant in Tokoroa has brought a wave of comment in the media over the past few weeks.

Potential uses of the planned plant have ranged from irradiation of food, through sterilisation of medical equipment and food packaging to disposal of toxic waste (polychlorinated biphenyls - PCBs).

The South Waikato Trust which is behind the project has held a series of public meetings and forums to explain the mechanisms of Cobalt-60 sterilisation. They hope to avoid the public relations problems which thwarted a similar proposal by Ansell Steritech in 1987 for a food irradiation plant in Mangere, Auckland.

However Greenpeace has already objected strongly to the proposal claiming that use of Cobalt-60 contravenes New Zealand's nuclear free policy and warning of the danger of potential accidents from transport of Cobalt-60 and possible discharges from the plant.

A detailed article by Dr Peter Roberts of the Institute of Geological and Nuclear Sciences (Lower Hutt) in the *NZ Herald* (Friday May 6, 1994, page 9) discussed the application of Cobalt-60 irradiation for food sterilisation at length. He outlined the plant set-up, how the Cobalt-60 is contained, the stability of Cobalt-60 itself, and how the irradiation process is carried out.

*Chemistry in New Zealand* is interested in comments from our readers on Cobalt-60 irradiation plants, their construction, safety, applications and particularly use in destruction of toxic waste.

Robert Lyon  
Managing Editor  
*Chemistry in New Zealand*

## IRL NOTES

(Some of the goings-on in chemistry at Industrial Research Limited)

Mike Brown, an MSc student with Nigel Sammes at Waikato University spent four weeks with the Ceramics Team working on ceramic/metal composites (cermets). The Ceramics Team also has had a visit from Rod Stead of Rojan Ceramics in Perth, to discuss collaborative projects with the mining industry.

Allan Read has returned after a year spent collaborating on high pressure/high temperature reactions with Terry Seaward at ETH in Zurich. Terry Seaward is an ex DSIR Chemistry Division scientist.

Joining the Carbohydrate Chemistry Team is Gary Evans. Gary did his PhD with P K Grant at Otago University. Before returning to New Zealand he spent a number of years with a small startup company, Oxford Asymmetry, which synthesise chiral intermediates. Jenny Mason has left the Carbohydrate Chemistry Team for a year's maternity leave.

The Carbohydrate Chemistry Team is expanding the number of clients it supplies custom synthetic chemicals to, and recently supplied carbohydrate derivatives to Astra Arcus (a Swedish chemical company). Cambridge Research Biochemicals also received another shipment of a glycosidase inhibitor.

A joint venture between IRL and ESR, managed by Barry Dent, to produce deuterated drug standards for ESR and foreign clients (through Radian Corporation) is proving successful after a year in operation.

Steven Bloor has joined the Plant Chemistry Team headed by Ken Markham to work on the chemistry of floral pigmentation. This work is part of a sizable contract with Calgene Pacific of Melbourne. The Plant Chemistry Team also had a visit from Dr Markus Veit of the University of Wurzburg and his student, Berndt Ibscher. In the two months that they were at IRL they examined the effects of ultraviolet light on allelopathy. And to save you having to reach for your dictionary; allelopathy is concerned with how plants chemically interact with one another.

#### **50TH JUBILEE OF DEPARTMENT OF CHEMICAL AND PROCESS ENGINEERING AT THE UNIVERSITY OF CANTERBURY, 4-6 MAY, 1994**

In October 1943 the University Council approved a grant of £1100 to establish Chemical Engineering at Canterbury University College. Chemical Engineering had long been championed by H G Denham, Professor of Chemistry. Denham had asserted the need for both Engineering Chemists and Chemical Engineers, the former is a chemist "with a wide knowledge of physical chemistry and imbued with the spirit and method of the best type of engineer". The latter were to be "fully trained engineers - the more chemistry they know the better it will be".

Certainly the beginnings of Chemical Engineering at Canterbury conformed to Denham's desires. The Diploma of Industrial Chemistry was admirably suited to taking a chemist with a firm basis of physical chemistry and grafting on the spirit and method of the engineer. The degree course took students through engineering in parallel with a rigorous requirement in chemistry. So rigorous indeed that all the Chemical Engineers spent a year longer in training than other engineers and graduated with a double degree B.E./BSc.

Over the years both chemistry and chemical engineering have changed in character in ways that have lead to a divergence from Denham's concepts. Chemistry has become a more

theoretical subject and its perceptions have moved from the "bulk" behaviour of materials to molecular concepts. Water is viewed not less as a colourless liquid than as a triangular structure. Chemical engineering training has become more concerned with the generalisation of "process" behaviour - its dynamics and control than with the details of the particular process such as its chemistry. Indeed it is now possible for a chemical engineer to graduate with a formal exposure to chemistry which corresponds to only a part of second year university chemistry.

By 1951 the University Council had resolved after requesting Stan Siemon's views to establish "a department of Chemical Engineering within the framework of the School of Engineering" and in 1956 the Minister of Education wrote to the Registrar assenting to the establishment of a Chair of Chemical Engineering. In 1960 with the move to Ilam, Chemical Engineering became physically part of the School of Engineering and ties to the Chemistry Department began to break.

It is interesting to trace the developments of some of the classical chemical topics in chemistry and chemical engineering. My own field of thermodynamics had a strong position in both chemistry and chemical engineering and is one in which the chemical engineers are expected to have a broader grasp than the chemists - their interest include not only the chemical thermodynamics of the chemists but also the engineering thermodynamics of compressors, expanders, "heat engines", compressible fluid flow and so on. Of particular interest is the thermodynamics of fluid phase equilibria which started as the preserve of physical chemists and has now become very much the concern of chemical engineers. Statistical Thermodynamics - esoteric stuff of the 1960s - is now firmly established as a tool in the design packages used by chemical engineers to estimate phase equilibria for distillation design and the separation processes. However it is perhaps a matter of concern that not only do many chemical engineers not know the limitations of these techniques, but they are often barely aware that they are using them. I quote this as just one example of the way in which chemists and the chemical engineers are drifting apart. Chemists now see classical thermodynamics as "old hat" and tend to whip through the subject as quickly as possible and the chemical engineers use sophisticated tools based on physical chemistry which they have never been taught.

The 50th Anniversary of the establishment of Chemical Engineering at Canterbury was celebrated with a two day seminar on May 4-6 which was attended by about 170 graduates whose careers span almost the whole history of the department.

Stan Siemon, the first lecturer in Chemical Engineering and first Head of Department was the guest of honour and graduates of the Department arrived from all parts of the world - Australia, South East Asia, the United Kingdom, and the United States of America. A commemorative publication has been produced containing reprints of a number of papers, some relating to the development of the department, some describing its recent activities and some expressing the views and experiences of graduates now in industry. The publication is available for \$15 from the Department of Chemical and Process Engineering, University of Canterbury, Private Bag, Christchurch.

Arthur Williamson

# ENVIRONMENTAL ISSUES



## THE MANAGEMENT OF HAZARDOUS WASTE CONTAINING CHLORINATED ORGANICS.

It is probably not widely recognised that most hazardous wastes arising in this country can be properly treated and disposed of by local commercial operators. The major exception is for those wastes containing chlorinated organics such as the polychlorinated biphenyls, (PCBs), and some pesticides.

High temperature incineration is regarded as a satisfactory disposal option for these wastes by regulatory authorities in Europe and North America, but proposals for the installation of incinerators for this purpose in Australia or New Zealand are opposed particularly by environmental groups. As a result, and because of the quantities and cost involved, local chlorinated organic wastes are currently either being stored or are exported to Europe for destruction.

Destruction efficiencies for chlorinated organics in excess of 99.9999% can be achieved by incineration and the hydrogen chloride which results is removed from the combustion gases by alkaline liquor scrubbing. Exhaust gases can contain trace amounts of potentially hazardous compounds and regulatory limits on the concentration of these are usually expressed in terms of nanograms per cubic meter in permitted gaseous emissions. Included in these trace emissions are the dioxins and furans, probably the most renowned being 2,3,7,8,tetrachlorinated dibenzo para dioxin. The toxicity and carcinogenicity of these compounds at trace levels in the environment has been the subject of major scientific debate for more than a decade and this is the principle reason for opposition to incineration by the environmental groups.

There are however chemical methods for the destruction of chlorinated wastes which have been developed and are offered commercially in countries overseas as alternatives to high temperature incineration. Two of these processes which have received favourable reports from the USEPA under the Superfund Innovative Technology Evaluation (SITE) program, and are available in Australia, are known as Base Catalysed Decomposition (BCD) and Thermal Gas Phase Reduction respectively. Each is briefly described below.

### Base Catalysed Decomposition

This process was developed by scientists in 1989 working for USEPA's Risk Reduction Engineering Laboratory, Cincinnati, and has now been licensed for operation in several overseas countries. BCD is a relatively inexpensive treatment process which can dechlorinate pollutants in liquid and solid matrices.

Process chemistry involves nucleophilic substitution of chlorine atoms with hydrogen radicals. The reagents that are mixed with the contaminated matrix in a reactor vessel, are fine grained sodium hydroxide, a paraffinic oil as the hydrogen donor, and a finely divided carbon catalyst. The mixture is

then heated to 320 to 340° C for 1 - 3 hours. Products from the reaction are the dechlorinated organic compound, (e.g. biphenyl is produced from PCB), salt (NaCl), and water. Vapours arising from the process pass through a condenser and an activated charcoal adsorber with the result that there are no discharges to the atmosphere. GC/MS analyses of samples from the contaminated matrix and of the reaction products have illustrated destruction efficiency for the chlorinated pollutant in excess of 99.9999% with similar efficiencies for trace contaminants such as any dioxins and furans that may have also been present.

The USEPA scientists who developed the BCD process, have demonstrated that, in addition to PCBs, it can be effectively used to treat pentachlorophenols, DDT, lindane, 2,4,D, aldrin, dieldrin, 2,4,5,T and hexachlorobenzene. The solid matrix containing the pollutant can be soil or clay. As such this process also has a potential use in clean up operations for contaminated sites.

### The Thermal Gas-Phase Reduction Process

This also, is a highly efficient chemical treatment alternative to high temperature incineration for chlorinated waste materials, particularly for those with a substantial water content.

The process is based on the gas-phase thermochemical reaction of hydrogen with chlorinated organic compounds. At 850°C or higher, hydrogen replaces chlorine forming hydrogen chloride and lighter hydrocarbons; in the case of PCBs the reaction products are hydrogen chloride and methane. The reaction is enhanced by the presence of water, which acts as a reducing agent and a hydrogen source. Up to 40% of the methane produced is in turn converted to hydrogen, thus the process, under predefined conditions, can operate without an external source of hydrogen. In the case of highly concentrated wastes the process produces an excess of methane which can be used as a fuel gas. Gases exiting the reactor are scrubbed with a caustic solution to remove the hydrogen chloride, and are then dried. The exiting gas consists of a mixture of hydrogen, methane, carbon monoxide and light hydrocarbons which is recirculated back into the reactor after reheating, or when in excess, are diverted off, compressed and stored as fuel gas.

This process has treated PCBs and PAHs during full scale operations which were audited by the Federal Canadian Department for the Environment (Environment Canada) and by the Ontario Ministry of the Environment. Destruction efficiencies for these compounds in the wastes treated were shown to achieve 99.99999%. Bench scale trials have illustrated the effectiveness of this process for treating a wide range of waste containing other chlorinated organics including pentachlorophenol. In the case of solid wastes the contaminants must first be thermally desorbed or solvent extracted before they can be treated. This process has also been used in successful clean up operations of contaminated sites overseas.

Both of these processes are transportable and the costs quoted for treatment of waste material compares favourably with that for high temperature incineration.

Other treatment processes are being evaluated locally as alternatives to high temperature incineration for chlorinated

At the Waipa sawmill, which was identified by the National Task Group Investigating Site Contamination from the use of timber treatment chemicals as a "likely worse case", a physical chemical process involving the use of hydrogen peroxide and UV light in the presence of proprietary catalysts, is being trialed. Bioremediation using bacteria to degrade chlorophenols has also been used successfully, particularly in situations where contaminant levels are low. This method however has limitations with the destruction of trace contaminants such as the dioxins and furans which are also likely to be present.

If high temperature incineration for the treatment of waste material contaminated by chlorinated organics, either in

purpose built incinerators or in cement kilns, continues to be publicly unacceptable, there are efficient chemical treatment methods that should be considered. Chemists, and the NZIC, should not be reluctant to bring informed opinion into public debate on issues of this type and express views, particularly on the need to indefinitely store such wastes locally, or to ship these overseas to be treated in a process not regarded as publicly acceptable in this country.

Norman Thom  
 Convenor, NZIC Environment Committee.  
 C/- Environmental Science Dept,  
 University of Auckland.

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# LOCALISED CORROSION OF STAINLESS STEELS IN DAIRY AND FOOD PROCESSING INDUSTRIES

A J Betts

Industrial Research Limited, P O Box 2225, Auckland

## INTRODUCTION

Originally developed for use in "rustless" cutlery by Harry Brearley in Sheffield over 80 years ago, the iron-based stainless steel alloys have been extensively researched and steadily improved over the intervening period. These alloys are now an essential component of much of New Zealand's Dairy and Food and Beverage industrial plant, where they serve in a wide variety of applications. Good corrosion resistance (where corrosion represents the interaction of a material with its environment) combined with relatively high strength, smooth surface texture and good abrasion resistance make stainless steels ideal for use in storage tanks, piping, valves, homogenisers, evaporators, heat exchangers and other equipment in these industries.

Their good corrosion resistance stems largely from the ability to form an extremely tenacious chromium-rich oxide (passive) surface film, typically of the order of a nanometre or so thick. This film is normally coherent above a threshold chromium level of about 12 wt%.

Understandably this passive film which largely governs their corrosion behaviour has been the subject of intensive research. Both electrochemical and surface analytical methods including Auger electron spectroscopy, and X-ray photoelectron spectroscopy have been used to investigate its structure, which still remains the subject of much debate.

Another active area of research is the study of the highly localised nature of most stainless steel corrosion problems. Although a comparatively rare event, localised corrosion can have particularly damaging consequences in the food-related industries, where hygiene and contamination issues are very important, in addition to causing expensive plant downtime and costs involving component replacement. Such problems are in practice almost invariably associated with use of these alloys outside their design specifications, most probably due to a process upset or a quality assurance-related failure either during design or fabrication.

However the development of more differentiated food products and the need to store more waste products has made it increasingly important for plant owners and operators to understand more about their behaviour and limitations. This paper reviews some of their localised corrosion problems and briefly discusses some recent research in the field.

## LOCALISED CORROSION PROCESSES

Although general corrosion can occur when in contact with some organic fatty acids, under certain conditions in aqueous environments, localised corrosion processes are more common. Pitting, crevice corrosion, stress corrosion cracking (SCC) and intergranular corrosion (IGC) can typically affect only extremely small regions but can, under some circumstances lead to potentially catastrophic consequences.

Many factors can have a significant impact on corrosion performance of stainless steels and these are now well-documented, together with numerous case histories describing various failure investigations (1). Examination of such failures can often provide some indication of the alloy's failure mechanism and hence advance our understanding of its behaviour under certain conditions. Broadly-speaking three distinct groups of factors may be identified: they are associated with solution composition, metallurgical and compositional factors and physical conditions respectively.

### (a) Solution Composition

The influence of halide ions, particularly chloride, bromide and iodide on the corrosion performance of stainless steels is well-known (2). For chloride ions there is a logarithmic relationship between chloride ion activity and the experimentally-determined critical potential for pitting,  $E_p$  such that

$$E_p = A - B \log (Cl^-)$$

where A is a constant and  $(Cl^-)$  represents chloride ion activity

Measurements performed on artificial single pits manufactured from grade 302 stainless steel microelectrodes of 10 $\mu$ m diameter have confirmed that the value for B is approximately 90mV at room temperature (3). This value in turn is in excellent agreement with the widely accepted localised acidification model of stainless steel corrosion. However sometimes even at extremely high halide concentrations, pitting and other localised corrosion processes do not take place, as other anions such as hydroxyl, phosphate, nitrate, chromate and sulphate also present in solution effectively act as inhibitors. In the dairy and food industries, lactates and polypeptides (proteins) can also act as inhibitors in process fluids. In addition the solution's pH and buffer capacity (i.e. ability to counteract significant pH change) can also have a marked influence on corrosion performance. The stability of localised corrosion processes is dependent on the development of a localised, low pH environment next to the metal (i.e. pH < 2). Under these localised conditions the passive film does not reform and active dissolution is maintained. Buffer capacity acts by inhibiting the formation of the low pH environment, such that, for example, the minimum critical dissolution rate for stable pit formation is not obtained. The most corrosive process fluids contain unbuffered acid chlorides, especially in the presence of oxidants such as chlorine, peroxide or ferric ions. Such oxidising species can alter the electrochemical corrosion potential of a stainless steel significantly and are often used in sanitising (clean-in-place) or other cleaning agents. However provided such chemicals are correctly used in conjunction with careful process monitoring and control and with exposure to the stainless steel surface kept to a minimum, they can effectively kill unwanted micro-organisms and clean surfaces without causing unnecessary corrosion damage.

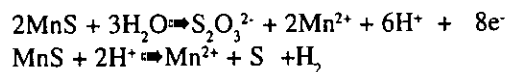
## (b) Metallurgical and Compositional Influences

There are five classes of stainless steel alloys, the chemical composition and heat treatment during manufacture determining the resultant alloy microstructure and ultimately its corrosion performance. Two of the most common grades are the austenitic types AISI 304 and AISI 316 (based on a face centred cubic lattice structure) and the duplex alloys such as alloy 2205. These latter alloys, originally developed in the 1930's have a mixed austenitic-ferritic microstructure and possess some benefits over the more traditional austenitic grades, most notably improved SCC-resistance and strength.

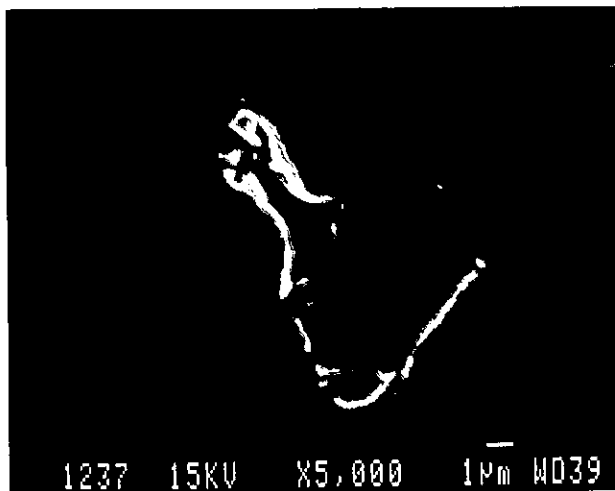
Certain elements, such as molybdenum and nitrogen are known to be beneficial in corrosion resistance terms. For example the molybdenum-containing AISI grade 316 stainless steel has better pitting and crevice corrosion performance than the non-molybdenum bearing AISI grade 304. Several explanations have been offered for this, including the inhibition of active dissolution during both pit initiation and propagation stages by submonolayer surface enrichment of molybdenum (4). Many new alloys have also been developed with increased chromium levels, and an empirical relationship between pitting propensity (the Pitting Resistance Equivalent, PRE) is often quoted:

$$\text{PRE} = \% \text{Cr} + 3.3 \times \% \text{Mo} + 30 \times \% \text{N}$$

Another important influence is the existence of intermetallic inclusions in stainless steels especially MnS. Most modern stainless steels have reduced S levels as a consequence, making them often more corrosion-resistant than older alloys containing higher sulphur levels. However some residual MnS is invariably always present in a stainless steel. Lott and Alkire (5) for example implicated the dissolution of MnS in crevice corrosion experiments according to the following reactions:



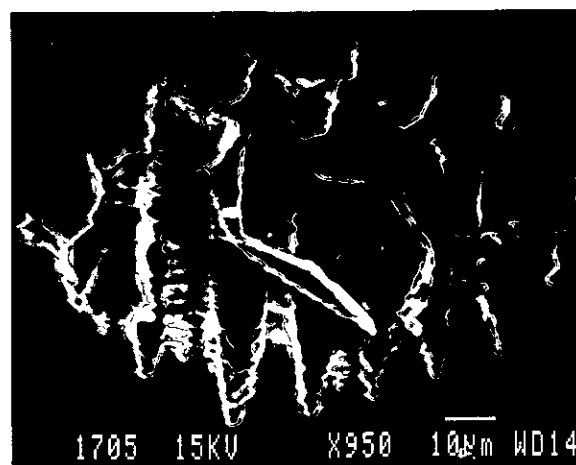
More recently Baker and Castle (6) using Auger/EDX spectroscopy, in conjunction with high spatial resolution imaging techniques and scanning electron microscopy, proposed a detailed dissolution mechanism based on deposition of a  $\text{MnCl}_2$  salt film which prevents repassivation and promotes stabilisation of the metal corrosion process during MnS dissolution.



**Figure 1.** Scanning electron microscopic image of titanium carbo-nitride particle in stainless steel (x5000). Note crevice on left corner.

Other inclusions, including titanium carbo-nitrides (such as that shown in Figure 1) and various oxides have also been implicated in pitting corrosion (7).

Corrosion problems of stainless steels are often associated with weldment regions and in cast materials, as microstructural variations can play a significant role in alloy service performance. Secondary phase precipitation resulting in formation of delta ferrite, sigma phase (an Fe-Cr-Mo compound) and microsegregation effects (e.g. Mo-depleted austenite in the vicinity of delta ferrite formed as a result of welding) can have an adverse affect on the corrosion performance. Figure 2 shows preferential attack of the austenite phase of a high alloy stainless steel weldment. Chromium carbide formation at grain boundaries (often referred to as sensitization) is also detrimental to the corrosion resistance of austenitic stainless steel alloys. Good quality, well-controlled welding procedures (with adequate but not high heat input), use of low-carbon alloys (L-grade alloys with less than 0.03wt%C) proper post-manufacture/fabrication treatment and moderate service temperatures can obviate many of these problems.



**Figure 2.** Scanning electron microscopic image of corroded stainless steel weldment region (x950). Note preferential attack of interdendritic region of weld.

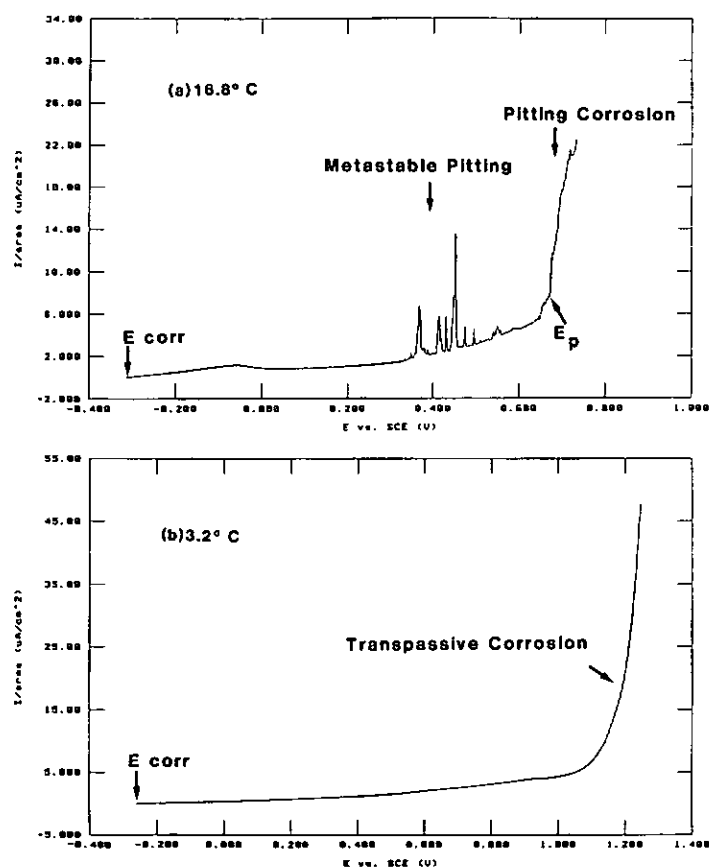
Some microstructural damage may result from plant fires. Such fires can lead to the formation of deleterious secondary phases, especially metal carbides, if the stainless steel is exposed to high temperatures (above about 500°C) for a sufficient length of time. Fortunately techniques are available which can be used to detect such damage on site, and the decision whether to refurbish or replace expensive components can then be made based on these evaluations.

## (c) Physical Influences

Corrosion, like most chemical reactions is sensitive to both temperature and pressure effects, though competition between concomitant oxidation and reduction reactions can lead to a slowing of reaction rate under some conditions at higher temperatures. For example crevice corrosion on stainless steels generally slows above about 70°C as the reduction reaction predominates. The effect of temperature serves as the basis for a number of stainless steel corrosion tests, including the Critical Pitting Temperature test, above which localised pitting occurs and below which transpassive corrosion occurs (which

is associated with the general dissolution of the passive film). This is illustrated in Figure 3 where potentiodynamic pitting scans performed both above and below the CPT are shown.

Crevices such as those found in flanges, weld spatter, mechanically-produced indentations and under fasteners, as well as under deposits such as scale and dirt, are particularly significant as they can lead to the onset of crevice corrosion problems. Microbes including bacteria and other organisms can also thrive in such environments, making strict hygiene and sanitation procedures necessary in the dairy and food processing industries. In addition these organisms may lead to microbiologically influenced corrosion (MIC), often involving sulphur species. Removal of scale and other surface deposits (such as iron contaminants) serves to mitigate against corrosion, as does sealing of crevices. Good design and materials selection (e.g. use of molybdenum-bearing stainless steel) also helps alleviate the problem.



**Figure 3.** Potentiodynamic polarisation curves commencing from corrosion potential ( $E_{\text{corr}}$ ), conducted on welded grade 2304 stainless steel at 0.33 mV/s (a) above CPT. Note region of metastable pitting (b) below CPT.

Stresses, resulting either from applied loads, or residual stresses resulting from manufacture or fabrication procedures are obviously an important component in SCC. Chloride ions, sodium hydroxide and some sulphur-containing compounds are known agents of SCC, which may be a problem with austenitic stainless steels when above about 60°C. The modern duplex stainless steel alloys are however more resistant to this type of attack, which is the subject of much intensive research.

Vibration resulting in constant cyclical loadings can also cause fatigue failures either in the presence (corrosion-assisted fatigue) or absence of an initiating corrodent.

Another corrosion problem encountered with stainless steel occurs under thermal insulation material. As insulation ages, water can leak into the metal-insulation annulus at joints or breaks and act as a source of chloride salts, albeit often at low concentrations (e.g. of the order of 10 ppm in tapwater). Elevated steel surface temperatures can however cause evaporative concentration and accelerate initiation of SCC. Various solutions to the problem have been proposed including use of less-SCC susceptible duplex stainless steels, use of barrier coatings and improved waterproofing.

Pitting and crevice corrosion of stainless steels are far more likely to occur in quiescent liquids than those in which there is a significant flow of fluid past the metal surface. One of the reasons often cited for this is the constant replenishment of the protective oxide surface film that results from interaction of the stainless steel with dissolved oxygen in the fluid. Beck and Chan (9), however, reported that pits in type 304 stainless steel tended to repassivate above a critical velocity where the salt film formed during the pit initiation process is removed. In sea water for example (which contains about 2% chloride ions) a minimum critical velocity of about 1.5m/s is often recommended.

However, erosion-corrosion can be a problem at higher flow rates when solid particles are present in a corrosive environment. Attack, which invariably manifests itself in a highly directional pattern, is most often found in elbows, of T-junctions in piping and on impeller blades in pumps. Solutions to this problem lie in the use of harder more corrosion-resistant stainless steel grades, changes in design (e.g. through lessening the severity of the bend) removal of suspended solids and process temperature reduction (where appropriate).

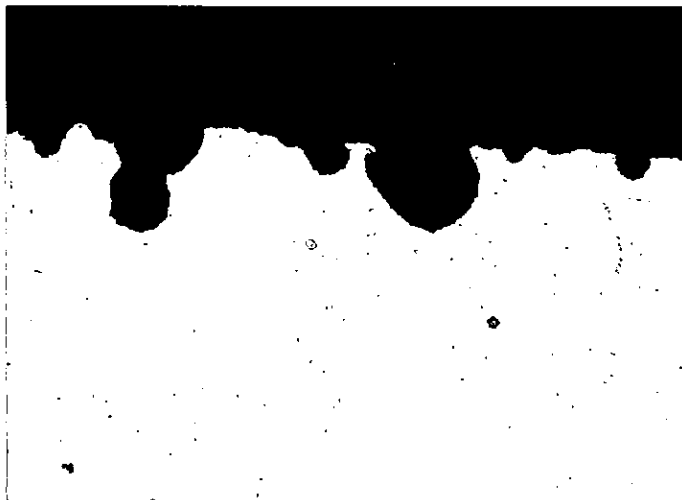
It has long been known that the stainless alloy surface finish can have a considerable influence on its corrosion performance, through the changes it can cause to the passive film and/or the underlying metal. In general, the smoother the surface, the higher is the resistance to initiation of pitting and crevice corrosion. The surface finish of stainless steel supplied by a manufacturer can vary depending on type of material and end use. Stainless steel supplied with a 2B (general purposes cold-rolled) finish often has good localised corrosion resistance.

Post-manufacture surface treatments such as pickling and passivation with nitric acid mixtures and electropolishing can also markedly improve a stainless steel's corrosion resistance.

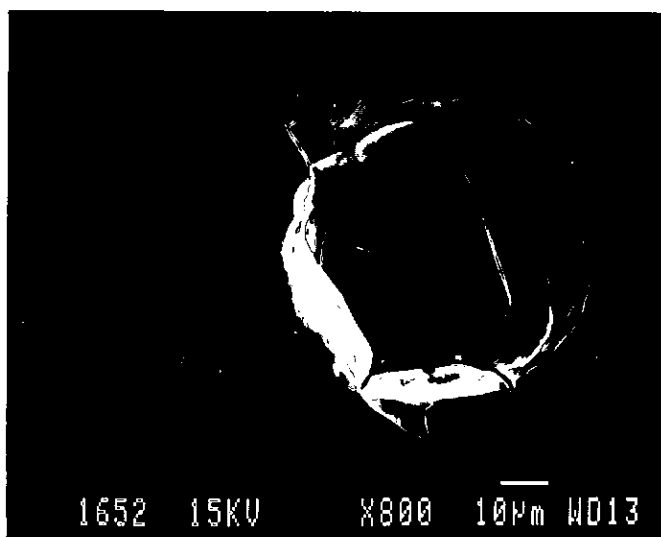
## RECENT RESEARCH

Much of the recent research work conducted on the localised corrosion of stainless steels has been directed towards a fundamental understanding of the corrosion mechanisms and processes in order to provide alloy selection and life prediction advice. Attention has increasingly been focused on the initiation stages of localised processes and significant advances have been made in this area in recent years. A good example is the investigation of metastable pitting, which is apparent in the potentiodynamic polarisation plot presented in Figure 3 where current transients are apparent well before the dramatic current increase that is normally associated with pitting ( $E_p$ ). It is now believed that pitting can be separated into a nucleation or initiation stage, where the passive film is destroyed locally and is then followed by a metastable growth stage. The next stage is either a rapid repassivation (i.e. film reformation) or a

stable pit growth (propagation) phase. This occurs when the active state within the pits eventually stabilises over a sufficiently long period of time and rapid metal dissolution ensues. The resulting pit morphologies can vary markedly ranging from open polished hemispherical pits (such as those shown in Figures 4 & 5) to etched irregularly shaped cracks.



**Figure 4.** Photomicrograph showing cross-section of a pitted stainless steel surface (x75). Note open-mouthed hemispherical pit morphology.



**Figure 5.** Scanning electron microscopic image of shallow repassivated pit on a high alloy stainless steel.

The actual pit shape depends largely on the type of rate-controlling reactions that occur during the growth stage which may be due to charge-transfer (i.e. electron flow), ohmic (salt-film) or simple diffusion control mechanisms. In one recent analysis (9), the transition from metastable to stable pit growth for open-mouthed hemispherical pits is thought to be governed by the "pit stability product". This was derived from consideration of the range of concentrations of metal ions produced by dissolution (between 75% and 150% of saturation of the chloride salt) and diffusion of the metal ions within the pit cavity during the growth stage. This product is thought to range between about  $3\text{mAcm}^{-1}$  and  $6\text{mAcm}^{-1}$  for an AISI type 304 stainless steel with stable pit growth favoured above the lower limit, although a contribution from a perforated cover over the pit mouth (eg. a remnant from the original passive film and/or some of the remaining surface metal) may be able

to sustain the high metal cation concentration and hence promote stable pit growth below this limit. Alternatively a high osmotic pressure of the concentrated solution inside the pit may lead to rupture of the cover, followed by loss of metal ions from the pit interior and hence repassivation of an essentially metastable pit.

This mechanistic approach to pit initiation using artificial pit microelectrodes (simulating pits) in conjunction with electrochemical methods, has yielded valuable information about the causes of stainless steel pitting, with important consequences for the life prediction of stainless steel plant. In addition to a range of other electrochemical methods (such as critical pitting temperature determinations and low-noise multiple specimen potentiodynamic tests), measurement of open circuit corrosion potentials and/or currents (so-called "electrochemical noise"), is being used at Industrial Research Ltd to detect and measure the onset of corrosion and hence relate localised corrosion initiation to changes in operating conditions with reference to some of the factors described earlier.

These specialised techniques also provide some mechanistic information as to the parameters controlling corrosion rate such as diffusion or ohmic resistance. A recent development is the use of a neural network, based on a computer system incorporating artificial intelligence concepts, to distinguish between initiation of pitting and crevice corrosion from electrochemical noise data (10).

Finally Industrial Research Limited have also developed a modular expert system suitable for capturing knowledge on the selection and use of stainless steels. The PC-based system has been tested and proved using a materials selection module for dairy industry applications. A second concerned with design and fabrication is currently under development. The developed system is strongly oriented to quality control and quality assurance with the objective of avoiding corrosion failures through provision of known technology.

In New Zealand, the number of skilled and semi-skilled corrosion and materials experts is limited, but the range of corrosion problems is not diminished by comparison with other more populous developed countries. There is a need for comprehensive systems addressing the needs of a relatively diverse audience. In these situations the use of a modular expert system structure, developed to meet the needs of a specific industry sector can have immediate benefits, particularly when the expert system structure can be transferred from one application to another.


## CONCLUSIONS

Stainless steels are extremely important alloys in the Dairy and Food Processing Industries whose corrosion performance depends upon a large number of factors. New processes and products are constantly being developed along with new alloys with improved properties. Research into the corrosion performance of these remarkable alloys continues and many issues have only comparatively recently been addressed, such as the mechanism of metastable pitting. There is still much controversy in the literature (eg. concerning the structure of the passive film, the mechanism of pit nucleation and the mechanism of SCC initiation) and many commonly held views

Much of this knowledge is being used to formulate predictive models about plant life and to determine likely causes of failure, through the use of electrochemical monitoring techniques and the expert system approach.

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# DOCTOR PEAK'S CHROMATOGRAPHY MADE EASY

## "D.I.Y. METHOD DEVELOPMENT IN GAS CHROMATOGRAPHY - WHERE TO START"

By Roger Whiting, Auckland Institute of Technology

*While the development of chromatographic methods is not a mechanical exercise a systematic approach can greatly speed up the search for a satisfactory method.*

### IS GAS CHROMATOGRAPHY A SUITABLE METHOD?

This is really the first question that should be asked - but one that is all too often ignored in the haste to get started. Very often gas chromatography is chosen because an instrument is available but it is not a suitable technique for all analytes. Compounds which can be determined by gas chromatography are those with significant volatility at temperatures of 350°C or less. These compounds must also be thermally stable at the temperature at which they are volatile. The quickest way to check if gas chromatography may be a suitable technique for analysis of a sample is to inject the sample onto a short non-polar column and take the temperature from low to high. If gas chromatography is suitable for the sample its various components should be detected coming through the column. To do this however a decision needs to be made whether the column to be used is to be a packed column or a capillary column.

Capillary columns are now selling much more rapidly than packed columns and as they give much higher resolution they appear to be the columns of the future. Thus if effort is going to be put into developing a new method, the time would be more profitably put into a capillary column based method.

A typical column to check if gas chromatography will suit a particular sample would be a 15m x 0.25mm, 0.2µm film thickness DB-5 (or equivalent). Helium should be used as the carrier gas at a flow rate of 40cm/s. This gives near optimum separation while giving short run times (see the Van Deemter plot). A suitable injection volume would be 1µL used with a split ratio of 50:1 and the initial column temperature of 60°C rising to 350°C at approximately 20°C/min.

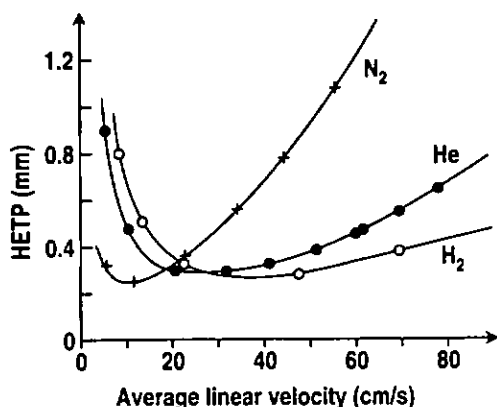


Figure 1. Van Deemter plot for hydrogen, helium and nitrogen

If the chromatogram shows only a solvent peak the assumption can be made that the sample cannot be analysed by gas chromatography unless the analytes are derivatised to make them volatile. If other peaks appear then gas chromatography would probably be a suitable method for this sample.

### HARDWARE - WHERE TO START?

The first choices to make in the development of the method are those relating to the hardware. This covers detectors, injectors and the column.

### CHOOSING THE DETECTOR

The choice of detector is probably best considered before columns and liquid phases as the detection of the analytes is necessary for optimising the choice of the other parameters. A range of specialised detectors can be used but are best left to those situations where the particular analytes are known to require special treatment e.g. Electron Capture Detector (ECD) for pesticide residues. Most samples can be analysed using the two most common detectors.

**Flame Ionisation Detectors (FID)** - These are usually the first choice of detectors as they exhibit high sensitivity and linearity and are robust to such influences as minor changes in column temperature or flow rate or to trace amounts of water or oxygen. Their disadvantage is that they can only be used to detect organics - however for many analyses this is not a problem.

**Thermal Conductivity Detectors (TCD)** - These are the second choice of detectors as they are sensitive to organic and inorganic molecules. However they are not suitable for capillary columns of less than 0.32mm inner diameter and are not sensitive to trace amounts of analytes in the sample.

### CHOOSING THE INJECTOR

Capillary columns require a very small sample injections (<10<sup>-6</sup> g). The gas flow rates in capillary columns are also low (0.75 to 2.0mL/min) so direct injection of the sample onto the top of the column while feasible, can be difficult in practice. There are several ways of injecting the required amount of material onto the column. The most common are split and splitless injectors.

**Split Injectors** - In these the sample is injected into a hot vaporising inlet liner - the sample and solvent vaporise and the vapour flow is split between the column and a injector vent. Thus a small portion of it (about 2%) passes onto the column. These injectors have the several advantages. The sample volume can be easily controlled by adjusting the split valve. The technique is simple and easy to automate. It gives good column efficiencies and the high flow rates produce narrow injection profiles. Samples do not need dilution. Unfortunately they do have some disadvantages. The loss of

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most of the sample out the split valve makes trace analysis difficult especially for high boiling point compounds. The hot vaporisation step can damage thermally labile compounds.

**Splitless Injectors** - Here a relatively large sample is injected into the inlet liner and passes onto the column which is cold. After a little time the split valve is opened and the inlet liner is purged to remove any compound remaining. The column temperature is then increased and the compounds start moving down the column. These injectors have the advantage of increased sensitivity as more sample is put on the column, however it is a more complex process than split injection and hence has more parameters requiring optimisation to achieve good resolution. It is also slower as the column is cold for 45 - 60s before the start of a temperature programme.

### CHOOSING THE COLUMN

The chromatography industry is generally moving towards capillary columns because of their greater efficiencies. Thus new methods should be developed if possible on capillary columns. There are some application where this is not practical or possible.

**Length** - The best starting point for development is usually a 30m column which is a balance between high resolution and run time. If the resolution is not sufficient then attention to such parameters as temperature, bore or liquid phase will probably pay bigger dividends than increasing the length of the column. Increasing the length will improve resolution but will also increase the run time more rapidly

**Internal Diameter** - The common starting points for method development are 250 or 320 $\mu$ m columns. The 250 $\mu$ m columns give better resolution while 320 $\mu$ m columns have a higher capacity and hence allow better sensitivity. The 320 $\mu$ m columns should be used if a thermal conductivity detector is to be used. If the resolution needs to be improved and the column temperature cannot be reduced then a 100 $\mu$ m column can be tried. If sensitivity needs to be increased then a 530 $\mu$ m column may be appropriate.

**Film Thickness** - The most common film thickness to start with is 0.2 $\mu$ m. A thicker film may be appropriate if the sample is very volatile.

### Choosing the Liquid Phase

The best starting point for development is with a non-polar liquid phase, either a 100% methyl silicone or a mix of phenyl and methyl silicone. These give good coverage of the capillary surface which is essential for good resolution. While ideal for non-polar or semi-polar analytes, having low polarity themselves, these columns have been found to be satisfactory for many polar analytes as well. If satisfactory resolution cannot be achieved by optimising the instrument parameters then more polar phases should be tried starting with the less polar e.g. OV-17 and working towards the more polar such as the Carbowaxes.

### OPTIMISATION OF INSTRUMENT CONDITIONS

Having assembled the hardware the development of the method requires the optimisation of the carrier gas flow and the column oven temperature.

**Carrier Gas** - A good starting point is helium as carrier gas. It has a flat Van Deemter plot so that choosing a flow rate of 40cm/s - about twice that for optimum resolution - will give a short run time while not greatly decreasing the resolution. If the resolution proves marginal then decreasing the flow rate to 20cm/s will improve it.

### Column Temperature

The optimisation of column temperature is one of the most critical steps in the method development. The starting point is to consider the initial test carried out to decide if the sample was suitable for gas chromatographic analysis. If there are only a few peaks and they appear over a temperature range of less than 100°C then the sample can probably be analysed isothermally. The initial temperature to try is approximately 30°C below the temperature at which the last peak appeared. From this point, to improve the resolution the temperature can be reduced. This will also have the effect of increasing the analysis time. If a satisfactory compromise between resolution and run time cannot be found then temperature programming can be used. Temperature programming should also be used if there are a lot of peaks appearing or they appear over a wide temperature range. The decision to use temperature programming then requires the choice of initial temperature, heating rate and final temperature. The initial temperature should be chosen to freeze the sample - but not the solvent - on the column. A temperature about 45 - 60°C below the temperature that the first peak appears will normally be satisfactory. The temperature can then be increased so that each component starts to move off down the column. A rate of temperature increase of 10°C/min is a suitable starting point. If the resolution needs to be improved the rate can be reduced. If the run time needs to be reduced the temperature can be increased more quickly. The final temperature of the column should be chosen to ensure that all the sample is eluted off the column. This can involve taking the column up to a temperature approximately 50°C above the temperature at which the last peak appears.

### CONCLUSION

While the development of analytical methods for gas chromatography cannot be regarded as a simple mechanistic process the application of a systematic approach to selection of each element in the system can greatly assist the analytical chemist. By starting from a point of commonly used equipment the optimisation process can ensure that these elements are only changed when it is essential to achieve satisfactory results. The use of capillary columns for method development is recommended as the higher column efficiencies of capillary columns has resulted in them replacing packed columns in most applications and most new developments will be in this area.

### Reference

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\* \* \* \* \*



## INTERNATIONAL NEWS

### *Surface Science Spectra* Issues a Call for Papers on Materials Used in Head and Disk Manufacturing.

An upcoming special issue of *Surface Science Spectra*, guest edited by Dr C R Brundle, will feature X-ray absorption photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) data on materials relevant to the magnetic recording industry. This special issue will provide high-quality, peer-reviewed reference spectra and related data useful for characterizing magnetic materials.

Data applicable for consideration for this issue include surface and interface characterization of materials commonly used in head manufacturing (such as that used in multilayer structures and slider materials) and materials used in disk manufacturing, including substrates, adhesive layers, overcoats, magnetic media and lubricants.

*Surface Science Spectra*, an official journal of the American Vacuum Society published by the American Institute of Physics, is devoted to archiving spectra of technological and scientific interest. It is an evolving database of standardized, peer-reviewed spectra and related data published in both a hard copy journal and in a digitized data format. Currently publishing AES and XPS data, the journal will expand to include other spectroscopies in upcoming volumes. Data published in previous issues cover a wide range of industries - from optics and the electrical properties of materials to dielectrics and condensed matter.

For submission guidelines and more information on this special issue, contact Melissa Beers, Technical Marketing Programs, 150 West Iowa Avenue, Suite 104 Sunnyvale, CA 94086, Ph 001-408-7370288, Fax 001-408-7379529. The deadline for submissions is August 1, 1994.

### Millipore Divests Waters Chromatography Division

Completing a six month sale process, the Millipore Corporation has found buyer for their Waters and Milligen Divisions. Millipore will concentrate on their traditional membranes market, while the Waters Management Team, backed by Waters Holdings, a private investment group are the successful bidders for the largest liquid chromatography business in the world. Purchased in 1982 as a US\$80 million company, Waters has grown to US\$400 million under the Millipore umbrella with worldwide sales 40% higher than its nearest competitors. Waters Holdings Inc. is a new corporation owned equally by AEA Investors Inc. and Bain Capital Inc.

With solid financing now assured Waters aim to continue their upward growth and have announced significant new technologies for delivery by the 2nd quarter of 1994 including Networked Chromatography Information Management Systems for Novell and DEC based Networks, new pumping systems compatible with their Extrel Division's recently unveiled ThermaBeam Mass Spectrometer Interface and a complete LC/MS system, INTEGRITY incorporating these technologies and a new Mass Detector into a fully integrated analysis tool.

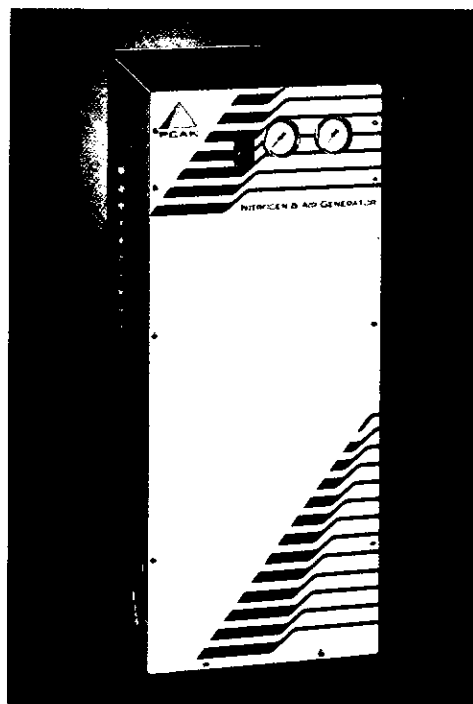
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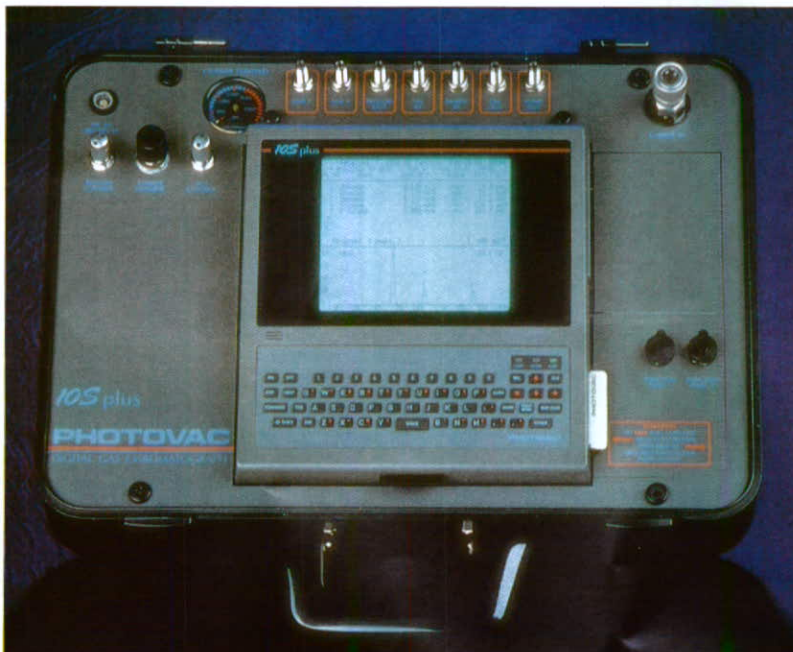
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Self-contained and weighing less than 14kg, the 10S PLUS™ Portable Gas Chromatograph features a built-in computer, menu-driven software and instant access memory cards for specific application storage. Stainless steel valving/tubing eliminates carryover and contamination from high sample concentrations. Wide bore capillary columns with isothermal ovens enhance chromatographic resolution for concentrations down to sub PPB levels.

**Alltech**

For further details, or a demonstration, contact:  
or circle no. 8 on the reader reply card

**Alltech New Zealand**  
P O Box 100-352, NSMC, Auckland 10  
Phone: 09-444 3230 • Fax 09-444 2399  
Freephone: 0800-652 766

The *Infralab TM5000* consists of a sampling unit fitted with the appropriate infrared sensors, an IBM 486 style desk top computer and full function software. Infrared Engineering's established infrared sensor technology allows the product to be measured at-line, without any preparation.

The *Infralab TM5000* uses Infrared Engineering's current gauging technology and scans the sample to simulate on-line presentation. Thus it has the freedom to use samples taken directly from the production process.

Measurement, accuracy, is frequently limited only to the quality of the reference technique chosen and comprehensive statistical software is included to calculate optimal calibration coefficients.

Used as an at-line analyser, the *Infralab TM5000* brings the power of the QA laboratory to produce quality measurements at the workplace. No special operator skills are needed to provide rapid and accurate monitoring of your production process.

CHEESE samples can be tested in seconds rather than hours, no sample preparation and presentation is required, just a simple grate.

The *Infralab TM5000* will measure moisture, fat and protein in all types of DAIRY POWDERS whether fine or agglomerated without the need for grinding or sample preparation.

- \* No Sample preparation
- \* Instant at-line measurements
- \* Save thousands of dollars
- \* Save hours of staff time

We are currently developing calibrations and tray modifications for moisture/fat/protein in viscous or semi liquid products e.g. ice cream, yoghurt, dairy desserts etc.

*Note:* All functions are performed by the function keys on the computer. Results can be sent directly to the factory computer network.

For further information contact:  
Watson Victor Ltd,  
P O Box 1180, Wellington,  
Ph (04) 385 7699, Fax (04) 384 4651  
Product Specialist - Simon Ganley

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

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## LYNFIELD FOOD SERVICES

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CONSULTANCY SERVICES



### WE CAN OFFER:

Analytical Services to the Dairy and Food Industries in the following areas:

- ⊙ Compositional Chemistry
- ⊙ Inorganic Testing
- ⊙ Carbohydrate Analysis
- ⊙ Ingredient Testing
- ⊙ Vitamin Testing
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- ⊙ Microbiological Testing

*A Telarc Registered Laboratory*

Contact: Laboratory Manager

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P.O. BOX 12-416, AUCKLAND  
PHONE 0-9-573 1110  
FAX 0-9-573 1106

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# NEW PRODUCTS

## LARGE VOLUME HELIUM PYCNOMETER

Particle and Surface Sciences Pty Limited announce a new version of the already successful Micromeritics 1330, Automatic Helium Pycnometer.

In response to market demand a 350cc sample capacity version has been introduced to satisfy the situation where the density of large samples is required.

All the features of the smaller versions such as, high accuracy, totally automatic operation, ease of use, etc, are incorporated into the new system.

Contact: Particle and Surface Sciences Pty Limited  
Tel: +61-43-237822, Fax: +61-43-237629  
or circle number 28 on the reader reply card

## DEMANDS OF LC/MS LEAD TO NEW PUMP TECHNOLOGIES

WATERS low pressure gradient forming pump technology developed during the mid 1980's contributed a great deal towards their success. RPS software incorporated into their 600 series controllers ensures precise gradient formation and stabilises flow variation resulting from pressure fluctuation which is essential for high sensitivity detection.

The acquisition by WATERS of Extrel Mass Spectrometry accelerated the development by WATERS engineers of a novel feedback mechanism called SILK Technology which augments the original RPS performance to eliminate flow variation completely, especially at extremely low flow rates consistent with interfacing liquid chromatography with mass spectrometers.

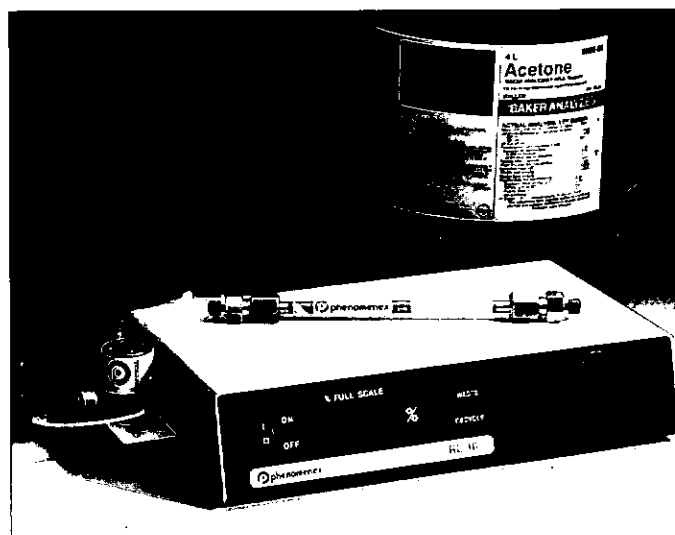
Previously only syringe pumps satisfied the demanding requirements of LC/MS interfacing, resulting in limitations of run duration and very high cost for gradient capability. SILK Technology has changed all that, making the world's most popular pumping system available both for the demanding LC/MS application and standard flow rate routine analyses. WATERS have improved the response times of their RPS control system as well improving their low pressure mixing gradient systems so that their performance is virtually indistinguishable from WATERS traditional high pressure mixing gradient systems, for years the industry standard. The new WATERS 626 polymeric and 616 metallic pumps incorporate this SILK Technology while the original 600E pump has been upgraded to the new RPS software and hardware.

LC/MS capability has been offered for many years but has fallen short of ideal for routine use. With the development of these new pumping systems and the Extrel INTEGRITY benchtop MS with ThermaBeam interface it seems that finally the technology has reached the state of development that will allow it to challenge GC/MS as the ultimate detection tool with the

wide range of molecular weights available to the liquid chromatographer.

Contact: Stuart Tyler,  
Alphatech Systems Ltd & Co.,  
P O Box 37-583, Parnell, Auckland  
Ph (09) 377-0392, Fax (09) 309-8514  
or circle number 29 on the reader reply card

## HPLC SOLVENT RECYCLER FROM PHENOMENEX SAVES UP TO 80% OF SOLVENT PURCHASE AND DISPOSAL COSTS



An amazing breakthrough in environmental technology now available from Phenomenex will save chromatographers up to 80% of their HPLC solvent purchase and disposal costs.

The Phenomenex Model RC-100 Solvent Recycler is a truly remarkable and reliable product which allows HPLC users to routinely recycle up to 80% of their mobile phase. In response to the signal from any LC detector, a two-position valve is activated to send uncontaminated mobile phase back to the solvent reservoir and contaminated solvent to waste. The unit is programmable and capable of storing up to 10 methods in non-volatile memory for easy recall. The Recycler can be used with any system from microbore to prep, and has exceptional utility in labs where continuous and unattended operation are required.

This is a simple and highly reliable solvent recovery system which will quickly pay for itself in just 2 to 3 months of ordinary use.

And the benefits to the environment are important to all chromatographers concerned with reducing chemical waste.

Contact: Chromspec Distributors Ltd  
P O Box 367, Warkworth  
Ph (09) 425 9032, Fax (09) 425 9032  
or circle number 30 on the reader reply card

**PERKIN-ELMER APPLIED BIOSYSTEMS  
DIVISION'S MODEL 172 MICROBORE HPLC  
SYSTEM**

*Offers unparalleled sensitivity coupled  
with very low solvent consumption*

The Model 172 microbore HPLC system has been optimized for use with microbore (1.0mm i.d.) and narrow bore (2.1mm i.d.) columns. It offers the higher sensitivity with smaller sample size and lower mobile phase consumption benefits of microbore HPLC in a compact, precise system. The Model 172 is now available with the Turbochrom Chromatography Workstation.

**Key features**

- \* Up to 20 times more sensitive than conventional HPLC
- \* Extremely low mobile phase consumption, <15mL per day, greatly reduces solvent disposal costs
- \* Uses smaller samples, saves time and money
- \* Microbore gradient syringe pump design for high performance at very low flow rates
- \* Model 785A UV/Vis detector provides industry standard performance
- \* Oven/Injector for stable chromatographic performance
- \* Ideal for LC/MS

Contact: Perkin-Elmer New Zealand  
P O Box 22-159 Otahuhu, Auckland  
Ph (09) 276 2230, Fax (09) 276 5602

**or circle number 31 on the reader reply card**

**PRICE-PERFORMANCE BREAKTHROUGH IN  
HPLC PC INTEGRATION**

Bio-Rad announces a breakthrough in price-performance in HPLC PC integration. For a remarkably low price, the new Bio-Rad ValueChrom™ PC integrator combines powerful new Windows HPLC software with a new advanced instrument interface to provide a complete PC based integration package. This powerful, user-friendly package is designed to be easily set up in minutes to quickly replace your aging integrator or outdated HPLC analysis software. The ValueChrom PC integrator will equip any system with the latest in advanced easy to use HPLC analysis software.

Software features include multitasking, integration and baseline manipulation, multilevel calibration, an advanced chromatographic database, and chromatogram overlay. The ValueChrom PC integrator can collect four channels of data from two systems simultaneously and can be used to replace integrators on two separate systems.

A control option is available for direct PC control of Bio-Rad's automatic samplers and pumps, and a variety of other popular HPLC pumps. The ValueChrom PC integrator is also available with an EPA energy star approved 486 computer.

Contact: Bio-Rad Laboratories  
P O Box 100-051, Auckland 10, Freephone 0508 805 500,  
Ph (09) 443 3099, Fax (09) 443 3097

**or circle number 32 on the reader reply card**

**THE NEW 720 KFS TITRINO FROM METROHM  
FOR KARL FISCHER AND END POINT TITRATIONS**

This new member of the Titrino family complements the range of Titrinos with a high class KF titrator for forward and back titrations (with an optional Dosimat). The method and silo memory allows automatic operation with sample changers. Like all Titrinos, the 720 KFS Titrino combines extremely easy routine operation with high flexibility. All Titrino functions are accessible via the separate keypad. For routine operation, the keypad can be disconnected and the instrument operated via the keys on its front panel. The two line, 24 character, backlit LCD allows easy communication and shows the volume of reagent added as well as the measuring signal. Parameters can be viewed and adjusted "live" while titrating. This allows the operator to modify titration methods at any time.

**Titrator and meter functions**

In addition to the Karl Fischer mode, the 720 KFS Titrino is a powerful end-point titrator with an optional conditioning routine. Two endpoints can be set per titration, and the calculation formulae are freely programmable. The two electrode inputs can be converted to a high impedance differential amplifier for non-aqueous titrations. The programmable polarizer allows biamperometric (dead stop) or bivoltagmetric titration with no additional hardware. On top of all that, the 720 KFS Titrino can be used for measuring pH, mV,  $I_{pol}$  and  $U_{pol}$ .

**Applications**

*Karl Fischer titrations*

- Water determination in oil, fats, plastics, hydrocarbons, solvents, acids, dyes, shampoos, soaps, creams, gases, milk products such as cheese, butter or margarine.

*End-point titrations*

- Standard acid/base titrations
- p- and m- value determinations
- Kjeldahl nitrogen determinations
- Bromine numbers in petroleum products
- Chloride in food etc
- Reducing sugars according to the Fehling method
- Vitamin C determination
- Determination of antimony in lead
- Zinc in plating baths
- Chromium in iron and steel
- Digestibility of fish meal (pH stat application)
- Free SO<sub>2</sub> and acidity in wine

The built-in bi-directional RS 232C interface allows connection of a PC for further data processing, printing and storage of results or as a link to a laboratory data system e.g. LIMS. The Metrodata 720 menu programme allows full communication with the 720 KFS Titrino as well as the back up of titration data for GLP documentation. The method memory can be extended to the PC if the Titrino memory should not have enough space for all your methods. Titration curves can be viewed on screen while titrating ("live") and data previously stored can be reviewed and recalculated at a later date. For a comprehensive documentation of your analyses and for result reports, a printer can be connected. With a balance connected, automatic transfer of weights reduces entry errors by operators.

Contact: John Morris Scientific  
P O Box 6348, Wellesley Street, Auckland  
Ph (09) 366 3999, Fax (09) 366 3060

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## INTRODUCING: THE PERKIN-ELMER MPLC<sup>®</sup> ANALYTICAL CARTRIDGE COLUMN SYSTEM

Brownlee, today's most popular cartridge column system, packed with Spheri-5<sup>®</sup>, Spheri-10<sup>®</sup> and Aquapore<sup>®</sup>, is now available from Perkin-Elmer's Applied Biosystems Division. The conventional fixed-end fitting format is also available for the most popular chemistries.

These cartridges are:

- \* Reproducible - quality columns manufactured under stringent control standards
- \* Easy to use - no tools required
- \* High performance - each analytical cartridge individually tested
- \* Versatile - 2.1mm and 4.6mm ID; choice of 3cm, 10cm and 22cm lengths
- \* Economical - low cartridge column investment

Contact: Perkin-Elmer New Zealand  
P O Box 22-159 Otahuhu, Auckland  
Ph (09) 276 2230, Fax (09) 276 5602

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### 100% PEEK FRITS

These new frits, manufactured by Upchurch Scientific, are the only frits on the market today made of 100% PEEK (Polyetheretherketone). Because these frits are made entirely of PEEK, they are chemically inert to solvents used in HPLC with no extractibles - only concentrated nitric and sulphuric acids will attack them.

Our 100% PEEK frits offer exceptional physical strength and uniform porosity and, our tests indicate, that the porosity is more constant than all other frits on the market - including the steel frits Upchurch offer!

If you run applications where chemical inertness is a concern, we highly recommend these new 100% PEEK frits.

For further information contact Douglas Scientific,  
P O Box 45 027, Auckland 8.  
Ph (09) 837-5447, outside Auckland Ph (0800) 735-725,  
Fax (09) 836-0668

or circle number 35 on the reader reply card

### iLIMS TO MANAGE LABORATORY SAMPLES

Laboratory Information Management Systems simplify sample tracking and help to ensure that all tests are done and meet your release criteria for quality and safety. At a fraction of the cost of a Networked LIMS system, and designed to be compatible with the code of Good Laboratory Practice, iLIMS from es<sup>2</sup>c provides full LIMS capability to the smaller lab.

The design of iLIMS, is an extended version of the application developed in the publication of the Royal Society of Chemistry "An Introduction to the Management of Laboratory Data: A Tutorial Approach using Borland's Paradox Relational Database". However the development of this full secure version of the application is beyond the scope of the introductory text. Every alteration of the data is recorded in an audit trail table which holds the identity code of the person logged on, data and time. The initialization of the system for use, all transfers of data to and from floppy disks and all backup and restore

operations are recorded in a supervisory audit table. Data tables are encrypted with a password key, so that they may not be altered from outside the application without knowing the encryption key and it is not possible to accidentally delete a record.

Improvements in laboratory efficiency, sample tracking, analysis forms, material test protocols, standard operating procedures, verification of results, certificate of analysis, summary reporting, efficiency reporting, quality audits, are all included with this organizational management tool designed for use on a single PC.

Contact: Steve Lawson, Alphatech Systems Ltd & Co.,  
P O Box 37-583, Parnell, Auckland  
Ph (09) 377-0392, Fax (09) 309-8514

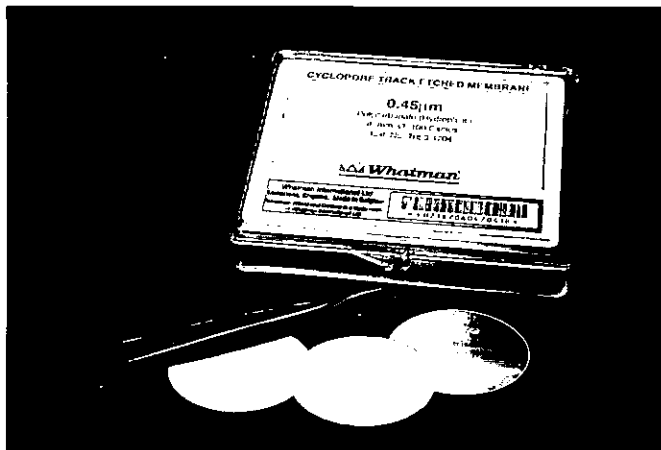
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### WHATMAN CYCLOPORE MEMBRANE

The new range of Whatman Cyclopore Polycarbonate and Polyester membrane filters are true pore size microporous screens and feature the sharpest cut off and reproducible results among track etch membranes available.

The smooth float membrane ensures that particles are retained on the surface so that they are easily visible under a microscope, unlike most other membranes where particles may be retained within the pores.

The very even distribution of the pores on the filter surface is achieved by the unique Cyclotron technology. Whatman offers totally transparent membranes with no cleaning necessary.



Whatman Cyclopore membranes are made of pure polymeric films and result in an exceptional chemical cleanliness. They are the material of choice for most analytical applications whenever collected particles must be measured, counted, recovered or transferred. The pore sizes range from 0.1µm to 12µm and the diameter range from 13mm to 142mm.

Typical applications include air monitoring, water analysis, general filtration, microscopy, micro-organisms analysis, nucleic acid studies, gravimetric analysis, X-ray fluorescence, blood filtration and cells analysis.

Contact: Labsupply Pierce (NZ) Ltd  
P O Box 34-234 Birkenhead, Auckland  
Ph (09) 4435867, Fax (09) 4447314

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## THE PERKIN-ELMER FLOW INJECTION MERCURY SYSTEM (FIMS)

**Fully automated mercury trace analysis  
- fast and cost-effective**

The Perkin-Elmer Flow Injection Mercury System (FIMS) is a small, easy-to-operate atomic absorption system dedicated to mercury trace analysis. Based on flow injection techniques, it is fully automated, fast and cost-effective. The FIMS consists of a high sensitivity spectrometer with a high energy mercury source, precision long-path absorption cell and solar-blind detector; and a built-in flow injection system. Mercury detection limits of 0.01 µg/L (relative) or 0.005 ng (absolute) are obtained routinely with FIMS. These levels are well under the current maximum allowable limits for mercury in drinking water set by regulatory agencies such as the US EPA.

Equally impressive is the performance of FIMS for the analysis of samples with complex matrices. Use of the flow injection technique reduces matrix effects and because the Hg is released from the matrix, measurements are virtually free of non-specific absorption. In addition, memory effects are much lower compared to continuous flow mercury analyzers, even with large variations in Hg concentration.

FIMS provides maximum sample throughput of up to 2 analyses per minute. In addition, the optional AS-91 autosampler can hold up to 218 samples and analyze them in any desired sequence. The software monitors and controls the entire process, including periodic recalibrations and check sample analyses performed according to protocols set by regulatory groups, such as the US Environmental Protection Agency (EPA). FIMS reduces operating costs. Reagent and carrier gas consumption are much lower with FIMS than with conventional continuous flow systems. FIMS offers the choice of using SnCl<sub>2</sub> (according to EPA protocols) or NaBH<sub>4</sub> reducing agents. With its peristaltic pumps, FIMS can also automatically add additional reagents such as KMnO<sub>4</sub> for stabilization of the mercury.

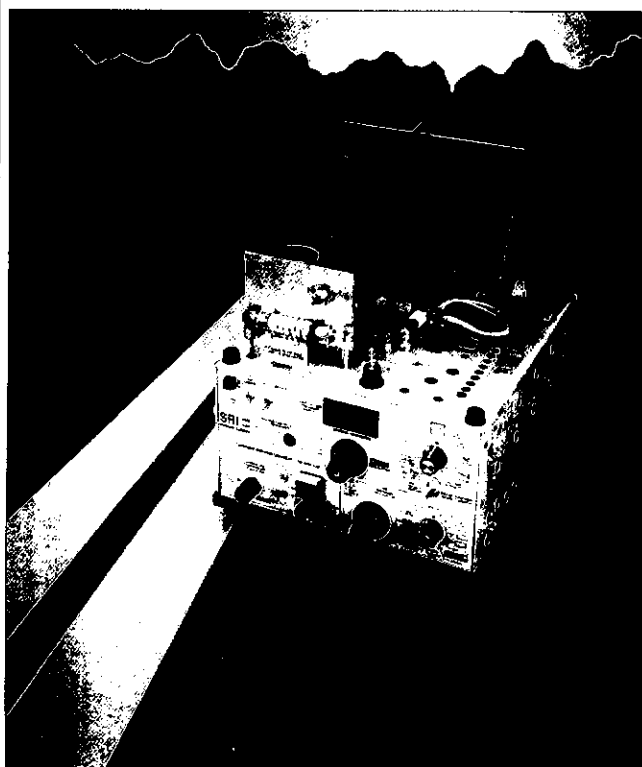
FIMS and its accessories are fully controlled from a personal computer. A graphical user interface based on the Microsoft Windows® operating environment and adhering to Windows conventions greatly simplifies operations. This software offers a variety of useful features: Stored methods can be recalled to provide fast, easy, reproducible system set-up; all analytical data - raw absorbance data, processed results, even the date and time the analysis was performed - are stored to provide you with the complete documentation of your results vital for regulatory compliance; built-in quality control procedures, including the ability to monitor analytical results, identify results that are outside pre-set limits, and modify and rerun the analysis - on-line and automatically; on-line HELP function providing a context-specific HELP function. FIMS is a small instrument requiring minimal bench space.

Contact: Perkin-Elmer New Zealand  
P O Box 22-159 Otahuhu, Auckland  
Ph (09) 276 2230 Fax (09) 276 5602  
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# LOW COST

## GAS CHROMATOGRAPHS

Take it on the road...



...or leave it in the lab!

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*High performance, portable  
capillary or packed column gas  
chromatographs for all applications.*



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**NO MORE BROKEN ELECTRODES !!!**  
**Orion Solid State pH/ Temperature Systems**  
**An Ideal Solution for the Dairy and Food Industries**



ORION Solid State pH/Temperature Systems are designed to be reliable workhorses, and are ideal for busy, multi-user laboratories or for field, or plant measurements where conventional glass pH electrode breakage or cleaning is a problem.

Featuring non-glass construction, the pHuture™ probe is reliable and rugged no matter what challenges your environment offers. Your ORION pHuture probe will survive the inevitable accident that occurs in the laboratory, plant, or field. Proven results in samples ranging from wastewater to pharmaceutical, environmental to food and beverage; the ORION pHuture pH measurement system will provide you with fast, accurate pH measurement no matter what you sample.

In thick, viscous samples, two problems have plagued industrial technicians for years. Stable pH measurements and carry-over from sample to sample or sample to buffer. Measurement is *no problem* with the ORION pHuture probe in samples that are aqueous, viscous or even semi-solid. Stable reproducible pH results are typically reported in seconds. Cleaning the ORION pHuture probe is also *no problem* as polarization does not occur even when aggressive cleaning procedures are used.

**Key System Features:**

- \* Simultaneous pH and Temperature Display
- \* Autocalibration or Manual Calibration
- \* Automatic Temperature Compensation
- \* User Prompting
- \* Ready Indicator
- \* Self-Diagnostic Test

For further information about this new Orion product, 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.

**or circle number 39 on the reader reply card.**

**NEW REFRACTIVE INDEX DETECTOR**  
**FROM PERKIN-ELMER**

The new Series 200 Refractive Index Detector from Perkin-Elmer is a highly stable and sensitive LC and GPC detector. The compact design features internal temperature control of the flow cells, offset adjustment, autozero and autopurge of the reference cell - all with easy keypad entry and a LED display.

Refractive Index (RI) detectors are universal (non-specific) detectors, providing sensitivity in the µg-ng range. RI detectors are most useful for the analysis of sugars, organic acids, triglycerides, and plastics (GPC). RI detection is ideal for compounds with no chromophore's or for compounds with low absorptivity in the ultra violet/visible spectral region.

**Series 200 RI Features:**

- \* Microprocessor-controlled, deflection-type design for ultra-sensitive detection with low noise and drift characteristics.
- \* High-sensitivity RI detection
  - Low noise ( $\pm 2.5 \times 10^{-9}$  RIU)
  - Low drift ( $2 \times 10^{-7}$  RIU/L), thermostatted flow cell at 35°C or 75°C)
- \* Excellent flexibility and reliability
  - Wide RI range 1.00 - 1.75 RIU (no need to change prism)
  - Compact and very rugged design, easy-to-use
- \* Convenience features
- \* Contact keyboard, LED display, remote functions, autopurge, liquid drain outlet.

Contact: Perkin-Elmer New Zealand  
P O Box 22-159, Otahuhu, Auckland  
Ph (09) 276 2230, Fax (09) 276 5602

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**GAS GENERATORS THAT PROVIDE**  
**PEAK GC PERFORMANCE**

Peak Scientific gas generators provide an economical alternative to using cylinder gas. Using gas generators will improve your laboratory safety, and reduce time and effort used in changing cylinders, and it will save you money!

There will be fewer problems with column bleed, detector response, baseline noise and drift. Fewer ghost peaks and increased productivity from your instruments. Peak Scientific's systems produce greater stability and readability thus eliminating cylinder to cylinder variations. All this allows you to run instruments 24 hours a day and over weekends. Safety is built into every system. Whether your requirement is hydrogen as a carrier gas, nitrogen for a mobile laboratory or carrier gas for a TOC, there is a system which will meet your requirements. In fact, with all the advantages of a Peak Scientific system, your expectations will be far exceeded. Products include; Nitrogen, Hydrogen, Zero Air, TOC Gas, Dry Air, Purge Gas and Tri-gas Generators.

Contact: Stuart Tyler, Alphatech Systems Ltd & Co.,  
P O Box 37-583, Parnell, Auckland  
Ph (09) 377-0392, Fax (09) 309-8514

**or circle number 41 on the reader reply card**

## NEW DSC 6 DIFFERENTIAL SCANNING CALORIMETER FROM PERKIN-ELMER

Perkin-Elmer announce the availability of the new DSC 6 Differential Scanning Calorimeter. The DSC 6 is targeted primarily at the routine, quality control/quality assurance segment of the thermal analysis market. The DSC 6, a heat flux design, together with DSC 7 forms a family of calorimeters that only Perkin-Elmer can offer.

Key DSC 6 features:

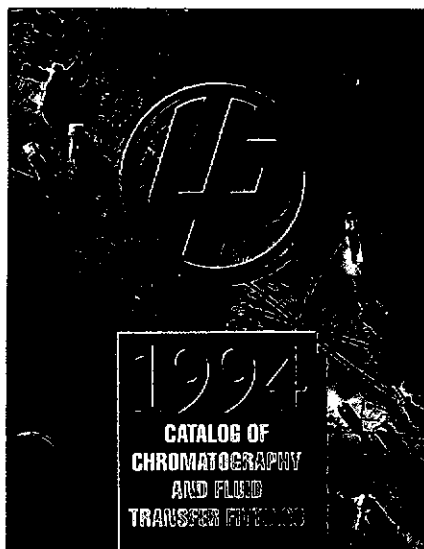
- \* Exceptional value and performance in one DSC package
- \* Low mass, corrosion-resistant furnace with a robust construction
- \* Exceptional reliability due to modular construction
- \* Compact design that requires very little bench space
- \* Integral cooling chamber for flexible, low-cost operation
- \* Simple and inexpensive subambient operation
- \* Built-in LCD panel displays instrument status
- \* Microsoft Windows-based user interface that is simple to learn and use
- \* Extensive software features including real-time manipulation and calculation
- \* Purity software included as standard

Contact: Perkin-Elmer New Zealand  
P O Box 22-159 Otahuhu, Auckland  
Ph (09) 276 2230, Fax (09) 276 5602

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## NEW 1994 UPCHURCH CATALOGUE AVAILABLE NOW!

Upchurch Scientific's 1994 Chromatography Fittings and Accessories Catalog and Technical Reference Manual is available now. The new catalogue includes an expanded Tech Talk section, complete product descriptions, and detailed four-colour illustrations, charts, cross-sections and photos. The catalogue is available free from Douglas Scientific. If you have not received your catalogue already, call us and order today - we have the solutions to your fittings problems.



Contact Douglas Scientific,  
P O Box 45 027, Auckland 8.  
Ph (09) 837-5447, outside Auckland Ph (0800) 735-725,  
Fax (09) 836-0668  
or circle number 43 on the reader reply card

## WHATMAN ION EXCHANGE CELLULOSE MEDIA

The Whatman range of ion exchange cellulose media for the separation and purification of biopolymers is currently used widely in small-scale laboratory research as well as large-scale industrial processing throughout the world. The wide selection of grades are available in the form of pre-swollen microgranular, pre-swollen fibrous, dry microgranular and dry fibrous.



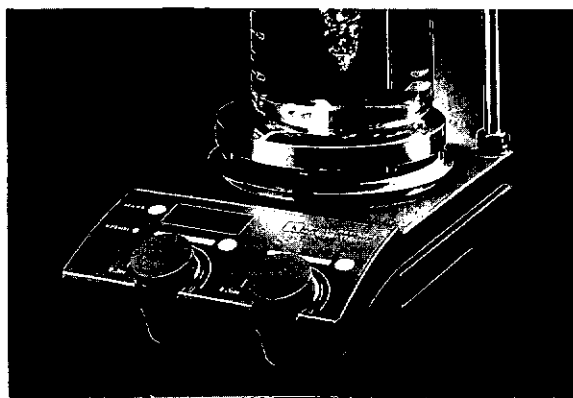
The media includes functionalities such as diethylaminoethyl, quaternary ammonium, carboxymethyl, sulphonyethyl and orthophosphate for separation and purification of proteins, peptides and hormones. The different media is designed to match the majority of requirements in performance and cost-effectiveness.

Contact: Labsupply Pierce (NZ) Ltd  
P O Box 34-234 Birkenhead, Auckland  
Ph (09) 4435867, Fax (09) 4447314

or circle number 44 on the reader reply card

## THE WORLD'S FIRST MAGNETIC STIRRERS WITH FUZZY LOGIC FROM IKA

IKA have released their new range of IKAMAG Magnetic Stirrers, the first magnetic stirrers/hotplates to feature integrated FUZZY logic control to regulate rotational speed and temperature. These new circuits offer faster heat-up without overshoot and maintain the target temperature within a very narrow range.

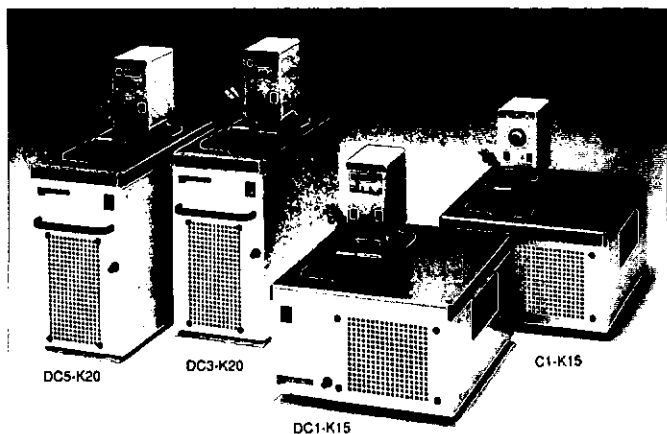


Available in four models: RCT basic, RET basic, RET digital and RET control, the new stirrer/hotplates have an appealing, eye-catching low profile, slim-line design made possible by the latest microelectronic components but incorporating the same high safety standards and reliability that has made IKA famous. All housings are completely enclosed to protect the electronics from corrosive fumes and spilled solvents. Electric sockets are recessed and protected from spills by a housing lip.

Contact: Labsupply Pierce (NZ) Ltd  
P O Box 34 234 Birkenhead, Auckland  
Ph (09) 443 5867, Fax (09) 444 7314  
or circle number 45 on the reader reply card

**NEW CFC FREE  
REFRIGERATED CIRCULATORS  
FROM HAAKE**

Refrigerated circulators are a combination of a temperature control module and a bath equipped with an integral cooling compressor.



**Features of the Haake CFC Free Refrigerated Circulators**

- \* The cooling circuit and insulation are CFC free (R134 is the coolant).
- \* Rapid cooling to the desired set temperature.
- \* Efficient removal of reactive heat even at high working temperatures.
- \* Sealed plastic bath cover reduces evaporation loss.
- \* Built-in drain nozzle fitted as a standard feature.
- \* A removable ventilation grid simplifies the cleaning of the liquefier. This in turn helps to ensure a high, long term cooling capacity.

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.

or circle number 46 on the reader reply card.

**IKA'S NEW  
MINIATURIZED DISPERSING INSTRUMENT  
THE ULTRA-TURRAX® T8  
FOR THE SMALLEST QUANTITIES**

In all fields of natural science, miniaturisation is in full swing. In order to achieve reproducible results, grain size reduction as well as homogeneity of the samples to be processed is of vital importance.

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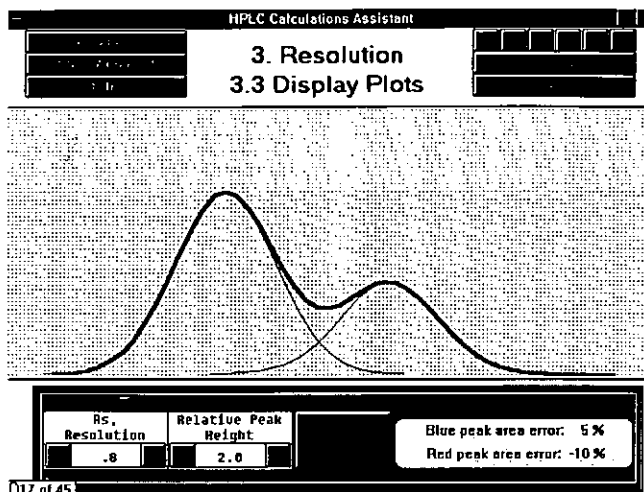
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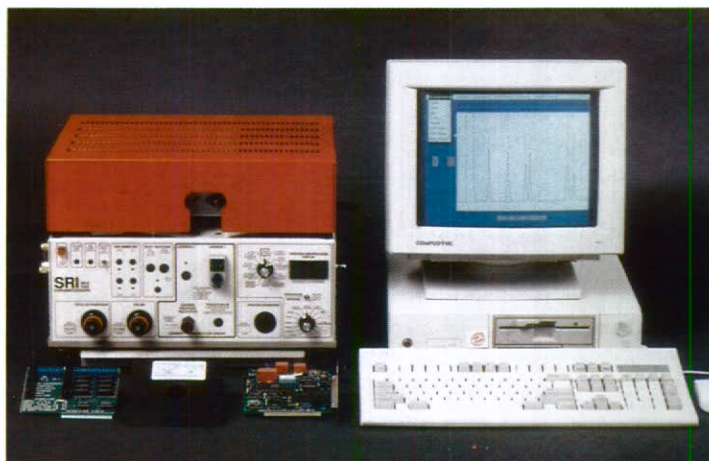
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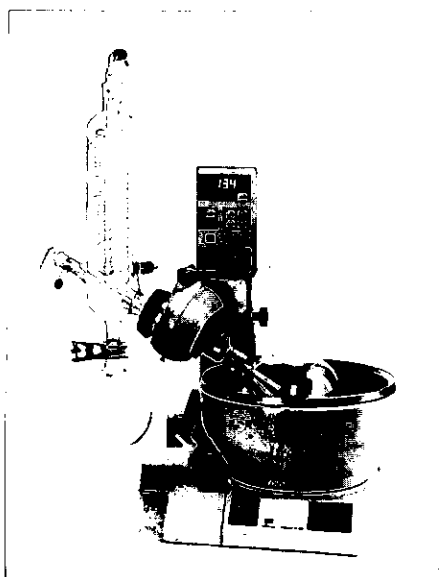
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#### Titrator and meter functions

The 719 SET Titrino is a powerful end-point titrator with an optional conditioning routine. The end-points can be set per titration, and the calculation formulae are freely programmable. The two electrode inputs can be converted to a high impedance differential amplifier for non-aqueous titrations. The programmable polarizer allows biamperometric titration with no additional hardware.

On top of all that, the 719 SET Titrino can be used for measuring pH, mV, temperature,  $I_{\text{pot}}$  and  $U_{\text{pot}}$ .

#### Applications

- Standard acid/base titrations
- p- and m-value determination
- Kjeldahl nitrogen determination (conventional)
- Bromine numbers in petroleum products
- pH-stat applications in synthesis laboratories
- Chloride in food etc
- Reducing sugars according to the Fehling method
- Vitamin C determination
- Vinegar in mayonnaise or ketchup
- Determination of metals in wine (decaassage of wine)
- Digestibility of fish meal (pH-stat application)
- Free  $\text{SO}_2$  and acidity in wine

The built-in bi-directional RS 232C interface allows connection of a PC for further data processing, printing and storage of results or as a link to a laboratory data system, e.g. LIMS. The Metrodata 719 menu programme allows full communication with the 719 SET Titrino as well as securing of titration data for GLP documentation. The method memory can be extended to the PC if the Titrino memory should not have enough space for all your methods. Titration curves can be viewed on screen while titrating ("live") and data previously stored can be reviewed and recalculated later.

For comprehensive documentation of your analyses and for result reports, a printer can be connected. With a balance connected, automatic transfer of weights reduces entry errors by operators.

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Step forward to 50 - method storage of all your titrations with the Mettler Toledo DL25 Titrator.

### *DL21 - Efficient Single Method Automatic Titrator*

An economical automatic titrator, the DL21 is easily configured for your specific application. After set up, run the titration with a single keystroke.

### *DL25 - Versatile Multi-Method Automatic Titrator*

The DL25 has 15 common titration methods in permanent memory for instant recall. You can use them as is or optimise them via interactive menus for your own needs. A security code protects the methods against inadvertent change.

### *Compact, Precise Burettes*

Burettes are available in four different sizes, fit all commercial reagent bottles and dispense precisely - ensuring reproducibility of results.

### *Fifty Methods At The Touch Of A Key*

The method key of the DL25 activates any stored method - up to 35 methods you can create, or one of the 15 permanently stored standard methods for acid, base or redox titration, photometric titration, the determination of chlorides, pH statings, etc.

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### *Designed for Efficiency*

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### *Designed for Reproducibility*

Key in the calculation constant. Even the titrant concentration is automatically determined. The titrator automatically reconciles both values with titrant consumption. This assures reproducible results. Calculation and transcription errors are eliminated.

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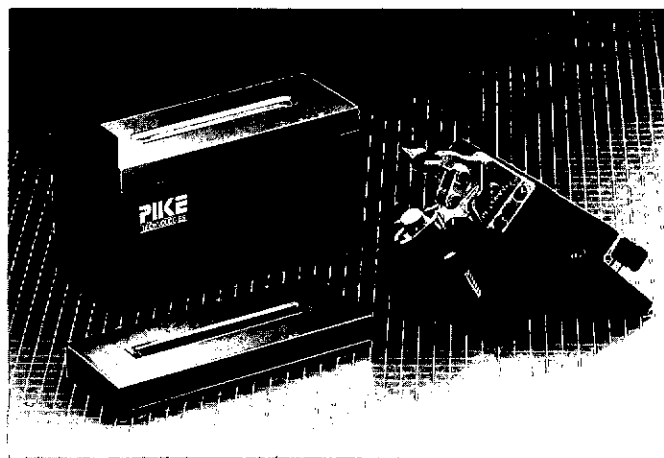
### *ATRPlus™ ATR Accessory*

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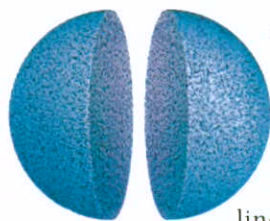
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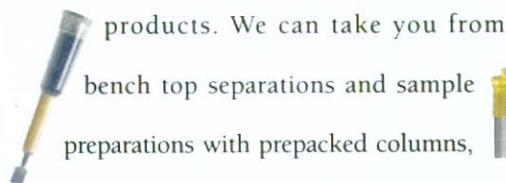
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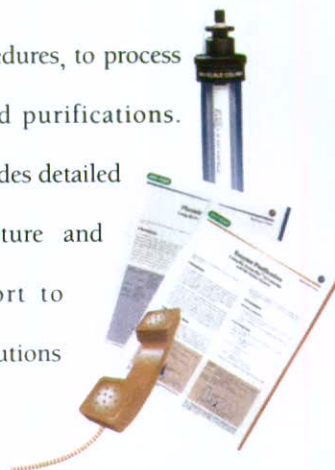


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## KARL FISCHER TITRATION

The DL18 Karl Fischer titrator from Mettler Toledo gives you precise water content determination from low ppm levels to 100%. Whatever the water content, the DL18 gives you precise determination with push button simplicity.

### *Reliable*

The titrator handles all calculations, automatically and accurately. Calculation and transmission errors are virtually eliminated. Attach a printer and you even prevent human error in results recording.

### *Precise*

A series of sophisticated precautionary measures guarantees exceptional reproducibility of results. The DL18 determines both titrant concentration and drift per minute automatically. These values are factored into the final results calculation for outstanding titrant concentration and drift per minute accuracy, particularly in trace analyses.

### *Precise Burettes*

Specially-designed METTLER TOLEDO burettes deliver precisely the right amount of reagent for increased reproducibility of results. The burettes fit all commercial reagent bottles, eliminating the need to manually transfer Karl Fischer reagents to another container.

### *Simple Operation*

The titrator indicates the next step. You confirm with the touch of a key. In a surprisingly short time, the titration is complete, the typical titration time is only two to three minutes with freely available water.

### *High Sample Throughput*

Productivity is assured by the simple operation of the DL18, and by its many automatic features, results recording with an attached printer further streamlines operation.

### *Hands-Free Titrant Transfer*

At the touch of a button, the pump siphons off the titrated solution and dispenses fresh solvent.

### *Automatic Transfer of Weight Values*

The DL18 Karl Fischer Titrator accepts weight values from a METTLER TOLEDO analytical or precision balance. The weighting data are included in the final result calculation after the titration is complete.

### *Designed for the Future*

The DL18 can be connected to a printer through its built-in CL interface, for automatic recording of results. Extensive automation is possible with an optional RS232C interface through attachment to a computer, a robot or a complete lab information system (LIMS).

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## IKA'S NEW

### UTL 25 ULTRA-TURRAX®-INLINE LABORATORY AND PILOT SCALE DISPERSERS

"Scale-Up", the establishment of process stages from laboratory to production scale can be very costly and requires in the process step "dispersing" large quantities of test material. Test samples/batches which mostly have to be disposed of; requirements for installation, cleaning, sterilisation and manual documentation are further costs. In order to minimize the number of pilot plant tests recognised as necessary in order to be able to simulate production-like conditions on the laboratory table, IKA-Labortechnik has developed the UTL 25 ULTRA-TURRAX®-Inline machine. This dispersing instrument which operates according to the rotor-stator-principle, has a modular construction consisting of:

- flow chamber DK 25.11
- ULTRA-TURRAX®-drive T25, S5
- dispersing element S 25 KV-25G-1L
- mounting support AD 25

All materials contacting the product are made of stainless steel 1.4571 and Kalrez®, so practically all chemical and biotechnological processing up to a temperature of +220°C can be carried out.

The UTL25 is suitable for in-line processes, circulation processes and processes in which quickly reacting species are injected directly into the shearing head. The chamber DK25.11 can also be immersed directly into liquids and so ensures airfree dispersing with simultaneous circulation. In addition, the UTL25 can also be used as a centrifugal pump. The ULTRA-TURRAX®-drive T 25 S5 is equipped with a connection for the IKA-TRON®-revolution counter DZM 5, which has an analog output for recorder or PC-documentation. Temperature measurement in the bypass is made using the DTM 11 (with interface) with corresponding PT-100 measuring sensor. Further parameters of interest are e.g.: Period of dwell, particle size, rate of flow, rheological properties.

### *Throughput UTL 25*

with 1/min	8000: 3 1/min*
	24000:121/min*
	(* water, free outlet)

### *Particle fineness with S 25 KV-25 G-1L*

- 1 - 10 µm emulsions
- 5 - 25 µm dispersions

### *Key Features*

- Simple - complete - sturdy
- can be sterilised as well as autoclaved
- table or stand instrument - low space requirements
- ULTRA-TURRAX®-drive T 25, S5, standard versions with connection for revolution counter
- newly developed dispersing element S 25 KV-25 G-1L, suitable for vacuum and pressure
- material contacting the medium: stainless steel 1.4571 and Kalrez®
- for suspending - emulsifying - disagglomerating
- airfree - sterile - in-line

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# VOLATILE SULFUR COMPOUNDS IN WINE: THEIR CONTRIBUTION TO FLAVOUR, AROMA AND COLOUR

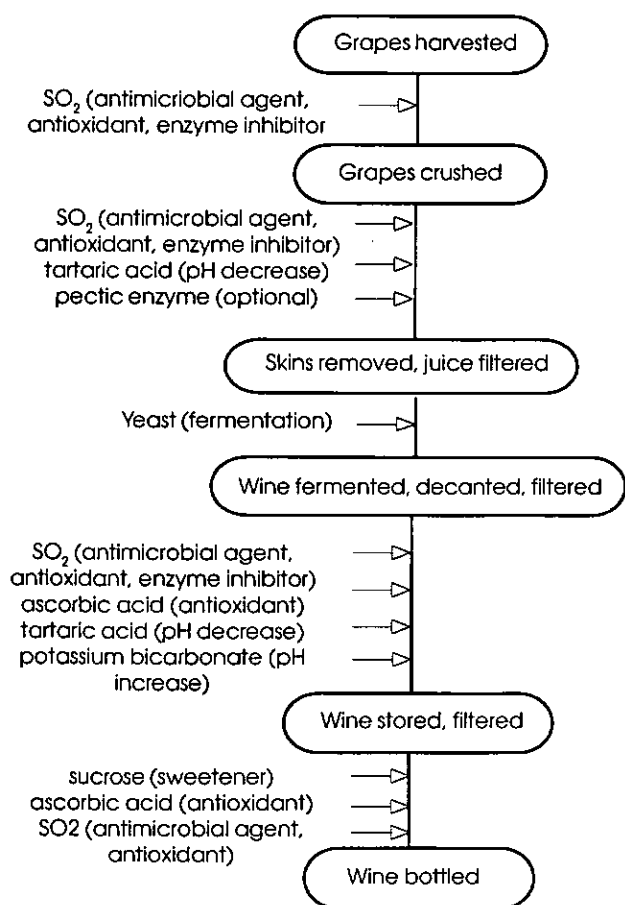
by David Shooter

Chemistry Department, University of Auckland, Private Bag 92019, Auckland

## INTRODUCTION

Sulfur compounds occur naturally in all wines often in very low concentrations, but they play an important role in the determination of the flavour and aroma of wine. These compounds are supplemented by the reaction products of the sulfur dioxide which is added at various stages of the wine making process. The detection of sulfur in subnanogram quantities became a reality in 1966 when the first report of the flame photometric detector for sulfur and phosphorus was published, [Brody and Chaney, 1966]. By 1987 thirty seven volatile sulfur compounds had been identified as contributors to the flavour and taste of wine, [Eschenbruch and de Mora, 1987].

Over 1300 volatile components of alcoholic beverages have been identified since the late 1950s [Nykanen, 1986]. The range covers virtually all classes of compounds from alcohols, aldehydes, acids, esters, aromatics and heterocyclics to amino acids, proteins, inorganic sulfates and pesticides. Capillary GC-MS has played a major role in the identification and quantification of flavour components where concentrations can vary from micrograms to grams per litre, [Williams, 1982].



The aim of this article is to identify those sulfur compounds in wine which contribute to wine flavour, aroma and colour and discuss aspects of their chemistry. In particular the role of reduced volatile sulfur compounds will be highlighted.

## THE WINE MAKING PROCESS

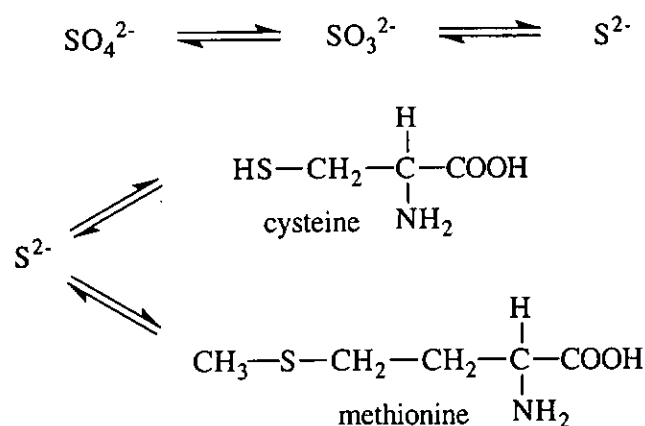
There are many variations to wine making. It is however useful to have a general overview of the process so that an appreciation can be obtained of the importance to wine chemistry of added chemicals and the natural sulfur compounds in grapes, grape juice and wine. A simple flowchart of the wine making process is shown in Figure 1.

## SULFUR CONTAINING COMPOUNDS IN WINE

Wine contains a variety of sulfur compounds, ranging from low molecular weight gases such as hydrogen sulfide to relatively involatile vitamins and thiophenes. A more complete list would include thiols, sulfides, thiocyanates, isothiocyanates, sulfoxides, sulfones, thioethers and thioacetic acid esters as well as reduced sulfur gases such as carbonyl sulfide. Many of the volatile reduced sulfur compounds in wine arise from the action of grape juice enzymes, or are products of yeast metabolism. Non-enzymatic reactions are also involved in their production.

Recently Rauhut [1993] compiled a table of concentrations of volatile sulfur-substances in wine. Thirty three compounds are listed varying in size from hydrogen sulfide to N-acetylmethionine ethyl ester. Concentrations range from less than 1.5 µg L<sup>-1</sup> for hydrogen sulfide in a normal wine to 2300 µg L<sup>-1</sup> for 3-(methylthio)-1-propanol.

Rauhut [1993] has also compiled a table of flavour threshold values published over the last fifteen years for sixteen volatile sulfur compounds in wine, beer, water or an ethanol/water mixture. The threshold values range from 0.9 µg L<sup>-1</sup> for diethylsulfide in white wine and 60 µg L<sup>-1</sup> for dimethyl sulfide in red wine, to between 1,000 and 10,000 µg L<sup>-1</sup> for 2-mercaptoethanol in a 15% (v/v) ethanol/water mixture.



Sulfate is available to yeasts in crushed grapes at concentrations between 30 and 2200 mg L<sup>-1</sup> [Rauhut, 1993]. Since almost all yeasts utilise inorganic sulfate as their source of sulfur, a sulfate deficiency is detrimental to yeast growth. Yeast are able to reduce sulfate to sulfide which is then used in metabolic processes:

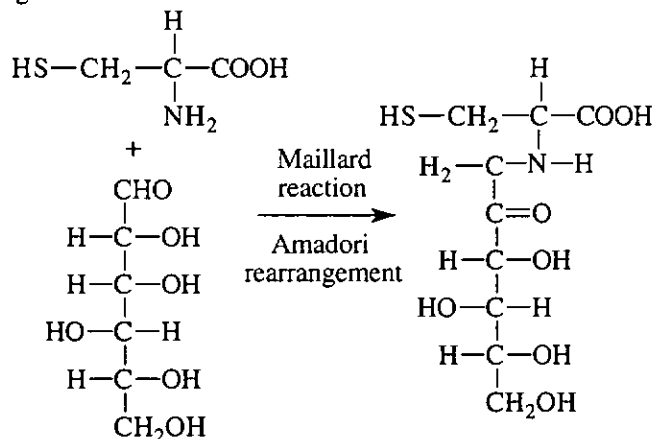
**Sulfite** is present in grape juice and wine. Yeasts normally produce between 10 and 30 mg L<sup>-1</sup>, but this is supplemented by the sulfite added during wine making, with a resultant concentration normally between 50-200 mg L<sup>-1</sup> [Rauhut, 1993].

### Amino acids

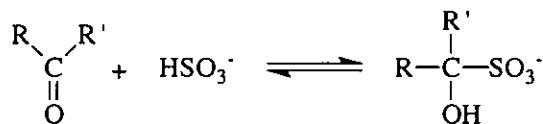
Many amino acids are found in both grape juice and wines [Sanders and Ough, 1985], those containing sulfur include methionine, cysteine, cystine, the peptide glutathione, as well as S-methyl cysteine and S-methyl methionine. Of these methionine and cysteine are found in the highest concentrations. Ough and Bustos [1969] report a maximum methionine concentration in wine of 110 mg L<sup>-1</sup> compared with 2 mg L<sup>-1</sup> for cysteine. The free amino acid content of wine is influenced by grape variety, the strain of yeast used and the conditions of the fermentation process.

Both enzymatic and non-enzymatic reactions characterise the chemistry of amino acids. Enzymatic formation occurs in both intact and crushed grapes and through the action of yeasts. Organic reactions such as the Maillard-reaction and the Strecker-degradation are responsible for non-enzymatic reactions. Both reactions are responsible for the loss of amino acids thus influencing the suite of volatile compounds in wine. They are also responsible for the production of a visible yellowing or browning in wine.

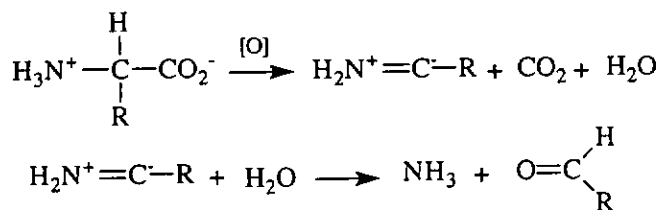
The Maillard-reaction occurs between amino groups and sugars:



The Maillard-reaction can be prevented by the reaction of sulfur dioxide with carbonyl groups on the sugars through a reversible bisulfite addition reaction:



The Strecker-degradation of amino acids is induced by mild oxidizing agents which cause decarboxylation of amino acids with concurrent oxidation to aldehydes:



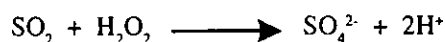
### Sulfur Dioxide (SO<sub>2</sub>)

SO<sub>2</sub> is one of the most important wine additives. It is added at various stages of the wine making process by bubbling SO<sub>2</sub> through the wine or by addition as a solid salt or aqueous solution. SO<sub>2</sub> is present in wine in 'free' state and 'bound' states, with most table wines containing a combined total of less than 100 mg L<sup>-1</sup> as SO<sub>2</sub>. Reactions between SO<sub>2</sub> and compounds such as acetaldehyde create the 'bound' state while the 'free' state consists of dissolved SO<sub>2</sub>, bisulfite (HSO<sub>3</sub><sup>-</sup>) and sulfite (SO<sub>3</sub><sup>2-</sup>) ions. At normal wine pH (3.0-4.0) HSO<sub>3</sub><sup>-</sup> is the predominant species.

'Free' SO<sub>2</sub> is effective as a fungicide against spoilage yeasts and bacteria and problems with odour and flavour can arise if insufficient sulfur dioxide is used. Lactic acid and acetic acid bacteria are of most concern, and sufficient SO<sub>2</sub> must be present to inhibit their growth while not interfering with desired fermentation yeasts and bacteria. Fortunately microbial species differ in their sensitivity to SO<sub>2</sub>.

Many oxidative changes in wine which result in a brown or yellow colouring are enzymatic but are also caused by contact of the wine with air. Sulfur dioxide inhibits wine browning by combining irreversibly with phenolic quinones to form colourless addition products thus making phenolic oxidation more difficult for polyphenol oxidase enzymes, [Fleet, 1990].

The legal maximum concentration for SO<sub>2</sub> in New Zealand is 200 to 400 mg L<sup>-1</sup> dependent on the type of wine, with sweeter wines being allowed higher concentrations, (in Australia the limit is 350 mg L<sup>-1</sup>). It has been shown that a level of at least 0.8 mg L<sup>-1</sup> of 'free' SO<sub>2</sub> is required in white table wine to inhibit the growth of bacteria and prevent oxidation, [Rankine, 1991]. As an antioxidant SO<sub>2</sub> also has an important role in the destruction of hydrogen peroxide which can cause discoloration and oxidizing aromas and flavours in wine.



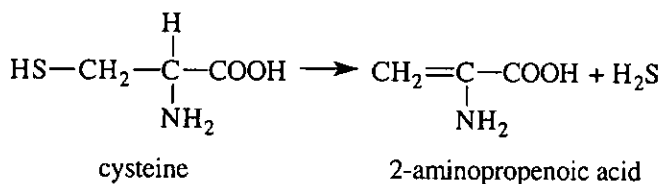
### Thioformaldehyde (H<sub>2</sub>CS) and dithioformaldehyde (H<sub>2</sub>C=S-S=CH<sub>2</sub>)

Neither of these compounds have been detected in wine, thioformaldehyde has however been detected in beer, but only at low concentration, [Nykanen and Suomalainen, 1983]. The volatility and reactivity of these compounds may mean they have evaded detection.

### Hydrogen Sulfide (H<sub>2</sub>S)

It is hard to find complimentary comments about the presence of H<sub>2</sub>S in wine. It has an unpleasant odour at all detectable concentrations, and is reported to begin detracting from the bouquet of wines when present in amounts above 1000 µg L<sup>-1</sup> [Nykanen and Suomalainen, 1983]. A lower odour threshold value of 50-80 µg L<sup>-1</sup> for hydrogen sulfide has been reported [Wenzel *et al.*, 1980].

Hydrogen sulfide can be formed in wine by a variety of processes. It is produced in small quantities during fermentation and where cysteine levels are high enzyme desulfuration can increase H<sub>2</sub>S levels:



$\text{SO}_3^{2-}$  or  $\text{SO}_2$  added to grapes prior to fermentation can also be a source of sulfur for  $\text{H}_2\text{S}$  formation by yeasts.

Elemental or molecular sulfur used as a grape fungicide can appear in the bottled product as  $\text{H}_2\text{S}$ . The sulfur is reduced non-enzymatically by compounds, e.g. glutathione, that possess a sulfhydryl group.



If  $\text{H}_2\text{S}$  is not removed by the carbon dioxide produced during fermentation, treatments for removal of excessive amounts are available. For example it can be reduced by purging or by addition of  $\text{SO}_2$ .



Removal of  $\text{H}_2\text{S}$  can also occur through contact between wine and copper or brass fittings and through reaction with added copper sulfate.



If  $\text{H}_2\text{S}$  is left in the presence of wine other volatile S-compounds may form which impart a unpleasant stagnant or rubbery odour. It has been shown that the reaction between  $\text{H}_2\text{S}$  and acetaldehyde can result in the formation of 1,1-ethanedithiol claimed as causing a 'sulfury and rubbery flavour note' [Rauhut *et al.*, 1992].

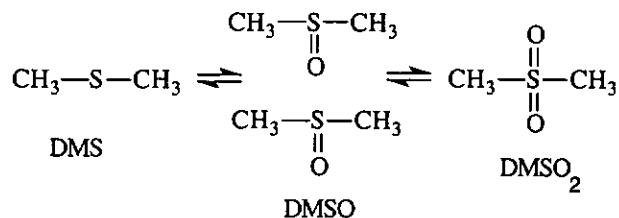
### Dimethylsulfide (DMS)

DMS is often stated to be a key volatile in the determination of both the favourable and unfavourable taste of wine. At high concentrations the odour is repulsive and cabbage like, while at low concentrations it is much more acceptable with a vegetable like smell. The reported odour threshold for DMS is  $25 \mu\text{gL}^{-1}$  for white wine [Goniak and Noble, 1987, Spedding and Raut, 1982] and  $60 \mu\text{gL}^{-1}$  for red wine [de Mora *et al.*, 1987].

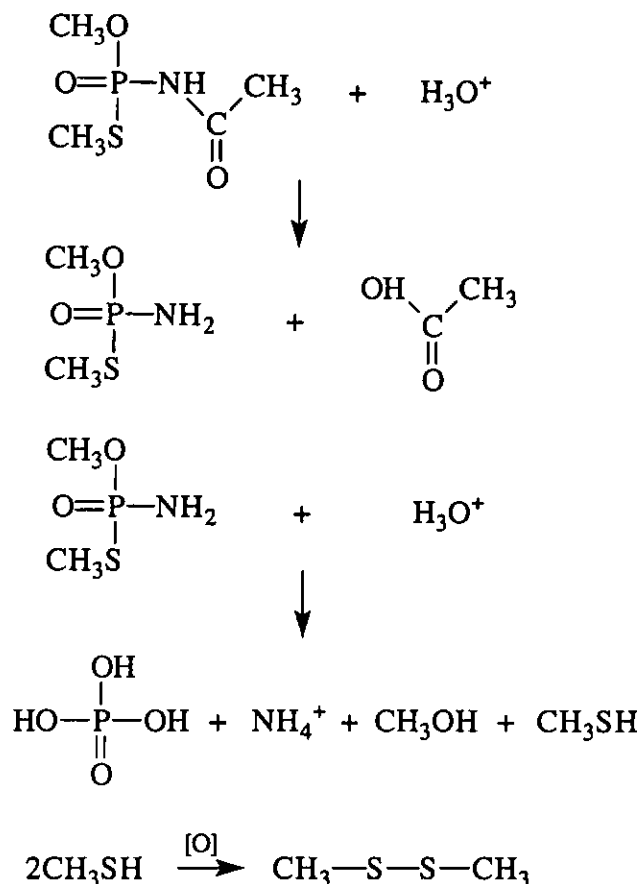
DMS was first reported in a white wine in 1974 [du Plessis and Loubser, 1974] and in red wine in 1980 [Spedding, Eschenbruch, 1980]. Concentrations in the range  $0-474 \text{mgL}^{-1}$  were reported for a variety of wines by Loubser and Du Plessis (1976). Other measurements have found levels in the range  $5-50 \mu\text{gL}^{-1}$  [summarised by Goniak and Noble, 1987]. Recent measurements made on a selection of Australian and New Zealand wines [de Mora, Lee, 1993] yielded a maximum of  $946 \mu\text{gL}^{-1}$ . As with most of the volatile sulfur compounds in wine, a feature of DMS levels is its variability which can often be related to vintage, viticulture practice and district, yeast strain, availability of precursors and storage time and temperature.

In contrast to beer, where the breakdown of S-methyl methionine and the reduction of dimethyl sulfoxide are thought to result in the formation of DMS, the precursors of DMS in wine are not well characterised with both cysteine and dimethyl sulfoxide being proposed. De Mora *et al.* (1986) using S-35 radiolabelled cysteine showed that addition of cysteine to a fermentation could result in the formation of radiolabelled DMS. Schreier (1979) states that DMS can be formed by yeasts from cysteine, cystine and glutathione, but not from methionine. De Mora *et al.* (1986) have confirmed that methionine plays a minor role as a precursor for DMS.

Recently it was proposed [Harvey and Lang, 1986] that dimethylsulfoxide could undergo disproportionation:



While there has been little confirmatory evidence for this reaction, it may play a role in wine chemistry by providing an additional pathway for DMS production.



### Diethylsulfide (DES) $\text{CH}_3\text{CH}_2-\text{S}-\text{CH}_2\text{CH}_3$

Diethyl sulfide has one of the lowest known flavour threshold values for a sulfur compound in white wine of  $0.92 \mu\text{gL}^{-1}$ . It has an odour of cooked vegetables, onion and garlic [Goniak and Noble, 1987]. However little is known about its concentration in wine.

### **Dimethyldisulfide (DMDS) $\text{CH}_3\text{-S-S-CH}_3$**

Low concentrations, in the vicinity  $1 \mu\text{gL}^{-1}$ , of DMDS have been found in both red and white wines [Leppanen, Denslow, 1980]. The aroma threshold value has been measured at  $291 \mu\text{gL}^{-1}$  [Goniak and Noble, 1987].

The presence in wine of sulfur containing pesticide residues, such as orthene, can lead to the formation of DMDS. The mechanism by which acephate, the active ingredient in orthene, is believed to be degraded is as follows [Rauhut et al., 1992]:

Acephate residues can result in wine methanethiol and DMDS levels far in excess of their odour threshold values [Rauhut, Kurbel, 1992]. Recently such levels have had a financial impact in some Western European countries.

### **Diethyldisulfide (DEDS) $\text{CH}_3\text{CH}_2\text{-S-S-CH}_2\text{CH}_3$**

DEDS can be produced by the mild oxidation of ethanethiol. The maintenance of significant  $\text{SO}_2$  levels may prevent this by causing reduction back to ethanethiol. DMDS has a odour threshold of  $4.3 \mu\text{gL}^{-1}$  [Goniak and Noble, 1987].

### **Methanethiol (MeSH) $\text{CH}_3\text{-SH}$**

MeSH is believed to be formed from methionine by yeast metabolism [de Mora et al., 1986], with the reaction being pH dependent [Rauhut, 1993]. MeSH can also arise from hydrolysis of pesticides, and on exposure of wine to light. It is a highly reactive substance with a low odour threshold in wine and a rotten eggs or cabbage flavour. Despite its potential impact on wine flavour MeSH in wine has not been subjected to intensive investigation.

### **Ethanethiol $\text{CH}_3\text{CH}_2\text{-SH}$**

In the sensory study by Goniak and Noble (1987), DMS and ethanethiol odours were compared and described in terms of five aromas. DMS aroma was composed of asparagus, corn and molasses whereas ethanethiol was described as having an onion and rubber aroma. The threshold value of ethanethiol was reported to be  $1.1 \mu\text{gL}^{-1}$  compared to  $25 \mu\text{gL}^{-1}$  for DMS.

### **Dimethyl sulfoxide (DMSO) $\text{CH}_3\text{-SO-CH}_3$**

Spedding et al., [1980] suggested that DMSO could be a precursor of DMS. Recently this hypothesis was confirmed when DMSO was measured in a number of Australian and New Zealand wines [de Mora et al., 1993]. In several instances DMSO was the most abundant sulfur-containing constituent in the wine, with a maximum concentration of  $1230 \mu\text{gL}^{-1}$  compared to the DMS maximum of  $946 \mu\text{gL}^{-1}$ . However no obvious correlation between DMS and DMSO concentrations was apparent for either the Australian or New Zealand wines. Taste and odour thresholds for DMSO in wine are not currently known, however if DMSO is a consistent precursor of DMS then it may have a significant, but indirect, influence on wine taste and odour. It has been suggested that DMSO reduction may also occur in the mouth as wine is drunk [Bamforth and Anness, 1981].

In a study of the levels of DMSO in selected fruits, vegetables, grains and beverages [Pearson et al., 1981] often found traces of its oxidation product dimethylsulfone ( $\text{DMSO}_2$ ).

### **Carbon disulfide ( $\text{CS}_2$ ) and carbonyl sulfide (COS)**

Both COS and  $\text{CS}_2$  have been found in a variety of wines at

concentrations in the  $0\text{-}10 \mu\text{gL}^{-1}$  range. The origin of both of these gases is not yet clear, although it is thought to be metabolic [Spedding et al., 1983], however their presence in the atmosphere may account for their occurrence in wine. It has been also been suggested that COS can be formed by a reaction between  $\text{SO}_2$  and  $\text{CO}_2$  [Shaw et al., 1980] and also may result from degradation of fungicides [Rauhut, 1993]. However both gases are thought not to have a distinct influence on the smell or taste of wine [Spedding et al., 1983].

### **S-methyl thioacetate ( $\text{CH}_3\text{-S-CO-CH}_3$ ) and S-ethyl thioacetate ( $\text{CH}_3\text{CH}_2\text{-S-CO-CH}_3$ )**

These two thioesters have been found in both red and white wine [Leppanen et al., 1980] at levels in the range  $2\text{-}16 \mu\text{gL}^{-1}$ . At low pHs both are subject to hydrolysis to form thiols and acetic acid which make them possible precursors of off flavours in wine through the thiol formation.

### **3-(methylthio)-1-propanol (MTP) $\text{CH}_3\text{-S-CH}_2\text{-CH}_2\text{-OH}$**

Like beer, wine can contain relatively large amounts of MTP compared to other sulfur compounds. For example, Baumes et al. (1986) have reported concentrations in the range  $507\text{-}998 \mu\text{gL}^{-1}$  for white wines and  $1363\text{-}2314 \mu\text{gL}^{-1}$  for red wines. Both MTP and its precursor 3-(methylthio)-1-propanol have an aroma which is associated with potatoes but both have high odour threshold values, [Rauhut, 1993]. MTP has also been described as having a powerful sweet soup or meat-like aroma and flavour, [Williams, 1982].

### **Sulfur containing amines**

Sulfur containing amines, such as N-[3-(methylthio)-propyl]-acetamide, have also been found in wine, but there is a lack of information on their impact on wine flavour and aroma.

### **THE FUTURE**

The complexity of wine chemistry and the subtlety of wine taste and aroma all contribute to the difficulty of providing a complete picture of the physico-chemical processes and resultant changes in composition that occur during the various process involved in wine making.

Recent research on sulfur compounds in wine has focused on their contribution to flavour and aroma and a considerable amount of work has been devoted to investigating the biochemical mechanisms by which flavour compounds are formed.

Eventually we may be able to explain the sensory characteristics of wine in terms of the chemical and physical properties of its flavour components. However further refinements of analytical techniques and a greater understanding of the wine making process are needed before this happens. Although sulfur containing compounds are numerous, they are not all of equal importance to the flavour of wine, and one of the major challenges will be to determine the relative importance of a compound in flavour and odour determination.

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# CONFERENCES & SEMINARS

## 19-24 June, 1994 20th International Symposium on Chromatography

Venue: The Bournemouth International Centre  
Bournemouth, Hampshire, England

Contact: Dr C E Roland-Jones  
Conference Coordinator  
The Chromatographic Society  
Suite 4, Clarendon Chambers  
32 Clarendon Street  
Nottingham, NG1 5JD  
United Kingdom  
Ph 44-602-500 596  
Fax 44-602-500 614

## 26-30 June, 1994 Developing Competency in Chemistry

Venue: Newcastle, NSW, Australia  
Organised by the RACI Chemical Education Division  
Further information can be obtained from:

Alan Turner  
Executive Officer, NZIC  
Ph (04) 473-9444  
Fax (04) 473-2324

## 3-8 July, 1994 RACI Division of Organic Chemistry 14th National Conference

Venue: University of Wollongong  
Wollongong, Australia

For further information contact:

Professor John Brenner (Chairman)  
University of Wollongong  
Department of Chemistry  
Locked Bag 8844  
South Coast Mail Centre  
NSW 2521, Australia  
Ph 61-42-214 255  
Fax 61-42-214 287

## 11-15 July, 1994 MACROAKRON '94, 35th IUPAC International Symposium on Macromolecules

Venue: Akron, USA  
Contact: Cathy Manus-Gray  
Institute of Polymer Science  
University of Akron  
Akron, OH 44325-3909, USA  
Fax 1-216-972-5463

## 19-21 July, 1994 Chromatography '94

Venue: Australian Jockey Club  
Alison Road, Randwick, Sydney, Australia

Contact: Dr S Grant Wylie  
RACI Analytical Group, (NSW)

## 24-29 July, 1994 30th International Conference on Co-ordination Chemistry

Venue: Kyoto International Conference Hall  
Kyoto, Japan

For further information contact:

Professor Koji Taraka  
Secretary of XXX ICC

Coordination Chemistry Laboratories  
Institute of Molecular Science  
Myodaiji, Okazaki, 444  
Japan  
Ph 81-564-55-7252  
Fax 81-564-54-2254

## 25-29 July, 1994 Polymer Networks 94, 35th Microsymposium on Macromolecules

Venue: Prague, Czech Republic  
Contact: Dr B Valter  
Institute Macromolecular Chemistry  
Academy of Sciences of the Czech Republic  
Heyrovskeho Nám 2, 162 06  
Prague, 6-Petriny, Czech Republic

## 1-4 August, 1994 9th International Drying Symposium (IDS '94)

Venue: Gold Coast, Australia  
The NZIC is co-sponsoring this conference and further information can be obtained from:

Alan Turner  
General Secretary/Executive Officer NZIC  
P O Box 12 347  
Wellington  
Ph 04-473 9444  
Fax 04-473 2324

## 8-12 August, 1994 13th International Conference on Chemical Education

Venue: Metropolitan Campus  
Inter American University  
San Juan  
Puerto Rico

For further information contact:

Professor Ram S Lamba  
Chairman 13th ICCE  
Department of Chemistry  
Inter American University of Puerto Rico  
P O Box 191 293  
San Juan  
Puerto Rico 00919-1293

## 6-9 September, 1994 Downstream Processing and Separations of Biological Molecules International Meeting

Venue: University of Cambridge, England  
This meeting is a coordinated effort of industrialists, manufacturers and scientists involved in downstream processing who have come together to provide an opportunity to present the latest advances in the technologies and to face its new challenges including regulatory and standards. Representatives from major manufacturers will be available to answer your questions and show the latest technological innovations.

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Canterbury, Kent CT3 1TP  
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14-15 October, 1994 **Citac '94 Hong Kong Symposium on Traceability and Comparability of Analytical Measurements**

Further information can be obtained from:

Alan Turner  
Executive Officer, NZIC  
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13-17 November, 1994 **3rd North American Research Conference on Organic Coatings Science and Technology**

Venue: Hilton Head, South Carolina, USA  
Contact: Dr A V Patsis  
Institute for Materials Science  
SUNY, New Platz, NY 12561, USA  
Fax 1-914-2550978

14-16 November, 1994 **IUPAC International Symposium on Functional and High Performance Polymers**

Venue: Taipei, Taiwan  
Contact: Professor Ging-Ho Hsuie  
National Tsing Hua University  
Department of Chemical Engineering  
Hsinchu Taiwan 300  
Fax 886-35-726825  
Email: ipst@nthu.edu.tw

3-5 December, 1994 **Conference of the Australasian Pharmaceutical Sciences Association**

Venue: Auckland, New Zealand  
This conference will be held jointly with the Australasian Society of Clinical and Experimental Pharmacologists and Toxicologists and will include a satellite-symposium on therapeutic drug monitoring.  
Contact: Assoc Prof John Shaw, Conference Secretary  
APSA '94 School of Pharmacy  
University of Otago  
P O Box 913, Dunedin, New Zealand  
email reception @ gandalf.otago.ac.nz

11-16 December, 1994 **10th International Organic Agriculture Conference**

Venue: Lincoln University, Christchurch  
Further information can be obtained from:  
Alan Turner  
Executive Officer, NZIC  
Ph (04) 473-9444  
Fax (04) 473-2324

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

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) 1 201-8682

17-22 December, 1995 **Pacificchem '95**

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

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Following on from the success of **CHROMATOGRAPHY '92**, the Australia wide consortium of scientific equipment suppliers in conjunction with the RACI Analytical Group (NSW) are pleased to invite you to **CHROMATOGRAPHY '94**

Australian and overseas speakers will present the latest developments in both the theory and practice of chromatography over the three days of the Conference.

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PLUS more than 26 instrument and accessory suppliers will be mounting displays for a Chromatography exhibition of working instrumentation which will be FREE for all to attend.

# NZIC COUNCIL NEWS

## *On Matters Financial - From the President*

There was an extended discussion at the recent Finance Committee meeting on the Institute's Budget projections and their implications with respect to subscriptions. As members will be aware much of the discussion of finances in the past years has been centred around the major drain on the institutes finances imposed by the Journal. Now that the problem of the Journal costs has been alleviated (so that current and projected Journal costs are around \$11,000 per year instead of \$40,000) we are able to direct our attention to other aspects of the Institutes finances.

The next largest item on the budget is our payments to IPENZ for secretarial services. This includes office space, access to computer facilities and salaries for the Executive Officer and typist. One can look back at past practice when both the editing of the Journal and the administration of the Institute were operated on a voluntary basis. Those were the times when members could count on the generosity of their employers to provide the flexibility and often the time for them to carry out professional body activities. In effect we were subsidised from time to time by some of the employers of our members. Sadly in these days of efficiency this kind of cross subsidisation is no longer accepted and we are as much subject to the "user pays" principle as anyone else. Given this situation the finance committee looked long and hard at the services provided for our IPENZ payment and concluded that we are receiving very good value for our money.

Assuming a modest surplus for 1993/1994 from the Auckland Conference, the budget projections indicate that over the next three years the Institute will average a budget that is just in balance - provided that we undertake no new initiatives.

The Finance Committee recommended a subscription package aimed at adding a small measure of additional security and flexibility to the budget, based on the changes in CPI and GST since the last subscription change (in 1990). This involved a small increase for Fellows and Members, no change for Associates and an extension of the time after election from Associate to Member before the Member subscription rate applied. This would have increased the Institute's income by about \$10,000 per annum (8%). The full Council, recognising that the Institutes's reserves, though not large were adequate to cover small budget deficits for up to three years, and taking into account that there is to be a review of the Institute structure and activities including the function of specialist groups, elected to postpone consideration of the subscription for one year until the report on specialist groups has been received. At that time the Council will be in a better position to assess the needs and wants of members and the services which can be provided through branches and specialist groups.

Arthur Williamson  
President - NZIC

### **NZIC Council Meeting, February 1994**

Minutes of this meeting are with branch secretaries should members wish to view them.

## Council Elections

Rule 16.2 states: -

"The President, Vice-Presidents, Honorary General Secretary and Honorary Treasurer shall be elected annually from nominations made by Branches, or by any six corporate members, and forwarded to the Executive Officer by June 30".

Please forward nominations to reach the Executive officer by Thursday 30 June, 1994

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A A Turner  
Honorary General Secretary For Council  
2 May 1994

## NZIC Branch News

### OTAGO

Recent visitors to the chemistry department have included two former staff members.

Dr William S Fyfe, FRS, Emeritus Professor of Geology at the University of Western Ontario spent four days in Dunedin at the beginning of March. After graduation in chemistry at the University of Otago in 1948, Bill Fyfe joined the staff and set up a high pressure laboratory for the study of mineralogical phase relations. In 1959 he joined the staff of the department of Geology on the Berkeley campus of the University of California. He was appointed Royal Society Professor of Geochemistry in 1966 at Manchester University and in 1972 moved to Canada to become Professor of Geology, Western Ontario. While he was in Dunedin in March Bill met many of his former students and colleagues. He lectured to three different audiences on "Depletion of Global Resources" with emphasis on water inventory, garbage and pollution.

Also in March Dr David Jones, from the Canadian Explosives Research Laboratory, Ottawa, presented a seminar on "Properties of Modern Commercial Explosives". David lectured in physical chemistry at Otago from 1971 to 1975.

Later in March Dr Benjamin Luberoff, founding editor of Chemtech entertained large audiences at two seminars when he talked about "A funny thing happened on the way to the analytical lab" and "Where is chemistry going?"

Prof. Taro Saito from the University of Tokyo lectured on "Synthesis of Sulfide Cluster Complexes as a Structural Model of Chevrel Phases" towards the end of March.

The inorganic research theme was continued in April by Prof. Hubert Schmidbaur from the Technical University of Munich who lectured on "Supramolecular Chemistry of Gold Complexes" and Dr Stephen Robinson from Kings College, London with a seminar on "The Coordination Chemistry of

Amidines. Amides and Carboxylic Acids - A Study in Diversity".

The Otago Branch commenced its 1994 programme on a controversial note when the Dunedin City Water Engineer, Nigel Harwood, described problems associated with the city's water supply and tried to convince the audience that the water was safe to drink despite a very low international rating.

Prof. Ian Dance from the University of New South Wales addressed the branch on the subject "If minerals could swim and fly" and followed this fascinating lecture with a departmental seminar on "New, big, elementary molecules." This first venture into an Australasian tour for the Royal Society of Chemistry lecturer was entirely successful in Dunedin.

Congratulations are extended to two Institute members on their appointments to personal professorships. Prof. Keith Hunter of the Chemistry Department has focused his research on chemical processes governing the behaviour of trace elements and carbon dioxide in aquatic systems. He has also been involved in the development of new teaching methods and the application of computer-aided learning to chemistry. He has served on several national and international committees and working groups dealing with the natural environment.

Prof Laurie Melton of the Department of Consumer and Applied Sciences has conducted research into food chemistry concentrating his investigations on the changes in cell walls in fruit during development. This has led to successes in controlling fruit ripening, making processing easier and reducing waste. His research on apples and strawberries could prove of economic benefit to New Zealand with respect to the exploitation of distant markets. He is a former president of the New Zealand Institute of Food Science and Technology.

#### MANAWATU

The Manawatu's Branch students' evening was organised as a dinner meeting on 27th April. To loosen everybody up after a hard day at the lab bench we started with a Trivial Pursuits Quiz.

After an enjoyable dinner we gathered to hear Vicki Hyde, editor and publisher of the general interest science and technology publication, New Zealand Science Monthly. She spoke of her experiences as a journalist, both in New Zealand and overseas, and stressed the importance of good general knowledge prior to specializing. As an indication of the audience's keen interest, the question time was almost as long as Vicki's excellent presentation.

Tony Burrell has just returned from Los Alamos to join the Chemistry and Biochemistry Department at Massey. Before his departure Tony was a member of the Auckland branch.

Mick Sherberne and Geoff Jamieson have also joined Massey staff. Geoff is a Kiwi who has returned from Georgetown to work in the field of protein crystallography.

Alastair MacGibbon, a dedicated member of the Branch Committee, is in Atlanta to attend the American Oil Chemist's Society annual meeting. Gill Norris, last year's Branch Chair,

has been awarded a Quill-McCarthy scholarship. She will attend a crystallography workshop in Sicily and then visit labs in Britain and the US. While in America she will attend the American Crystallography Association meeting in Atlanta.

Grant Boston  
NZDRI

#### WELLINGTON

The Branch Committee:

Mr Jim Waters (Chairman) is an adviser on toxicology to the Public Health regulatory services.

Dr David Bibby (Secretary) is general manager of CESIT (Communications, Electronics, Sensings and Information Technology) at Industrial Research Limited.

Dr Linda Parker (Treasurer) is a scientist with the Inorganic Materials Team at Industrial Research Limited.

Dr Keith Morgan (Branch Delegate) is a scientist with the Carbohydrate Team at Industrial Research Limited and a lecturer in chemistry at Victoria University.

Dr Rod Tilbury is a lecturer in chemistry at Victoria University.

Mr John Reeve is a toxicologist with the Agriculture Compounds Unit at AgResearch Limited.

Dr Jim Ellis is an ex President of NZIC and former Director General of the DSIR.

Ms Sue Freitag is a scientist at Works Consultancy Services Ltd.

Mr Graeme Murray is a science teacher at Hutt Valley High School.

Mr Noel Dowrick is a science teacher at the Correspondence School.

#### WAIKATO

The Waikato branch started off the year with the annual barbecue to recruit new members. This was held at the Common Room Club of the University of Waikato and featured experimental sausages from MIRINZ. As usual, this function was very successful and enjoyable.

Our secretary, Ron Newth is taking a break in the United Kingdom until July and in his absence, secretarial duties are being carried out by committee members Bill Henderson and Chris Miles.

Upcoming events include a visit to the Hautapu dairy factory and an accompanying talk by Trevor Locke and a visit by Brian Davis on Wednesday 21 September to give a talk to senior secondary school students.

Michèle Prinsep  
University of Waikato

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A record in the World Patents Index database describes a patent family, starting with the new invention (basic patent) and adding information about the same invention issued in other countries (equivalents). Records contain bibliographic data, Derwent-assigned titles, abstracts, general indexing and in-depth chemical and electrical indexing. Access to the chemical and electrical indexing is subject to subscription with the producer. Data from the World Patents Index database is also available in the print publications Chemical Patent Index (CPI), Electrical Patents Index (EPI), General and Mechanical Patents Index (GMPI), WPI Gazette Service, and World Patents Abstracts (WPA). Apart from its multinational coverage, the World Patents Index excels in the range of facilities for subject searching and the flexibility which this gives in formulation questions. Derwent interprets the original patent document for the searcher - providing improved titles, detailed and explicit abstracts, and a series of precise classification and coding systems.

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For the chemical researcher, the combination of the World Patents Index database and the comprehensive range of chemical databases on STN, constitutes a perfect reference source for his/her research.

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For further information on the Derwent databases on STN contact: Shirley Bailey-Wood, Marketing Department, Derwent Publications Ltd., Derwent House, 14 Great Queen Street, London WC2B 5DF. Tel. (+44) 71 344 2800, Fax: (+44) 71 344 2821, Email: sbailey-wood ccmail.derwent.co.uk.

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Currently science and technology degrees in Environmental Management, Sports Science, Electronics and Computers, and Information Technology are offered at Tamaki. Faculty of Science planning supports high quality interdisciplinary academic programmes which are complementary to those on the City Campus. An important emerging development at Tamaki is the proposed School of Environmental and Marine Science.

Applicants must be well qualified with a high level of achievement in research and with leadership qualities in teaching and administration. The appointee will be expected to lead the development of Chemistry research as well as teaching at undergraduate and graduate level at the Tamaki Campus.

Commencing salary will be established within the range \$80,000 - \$100,000 per annum (Professors) or \$71,000 - \$77,000 per annum (Associate-Professors).

Further information, Conditions of Appointment and Method of Application, should be obtained from the Academic Appointments Office, The University of Auckland, Private Bag 92019, Auckland, phone (09) 373-7999 ext 5097, fax (09) 373-7454. Three copies of applications should be forwarded to reach the Registrar by 1 August 1994.

Please quote relevant Vacancy Number UAC.428 in all correspondence.

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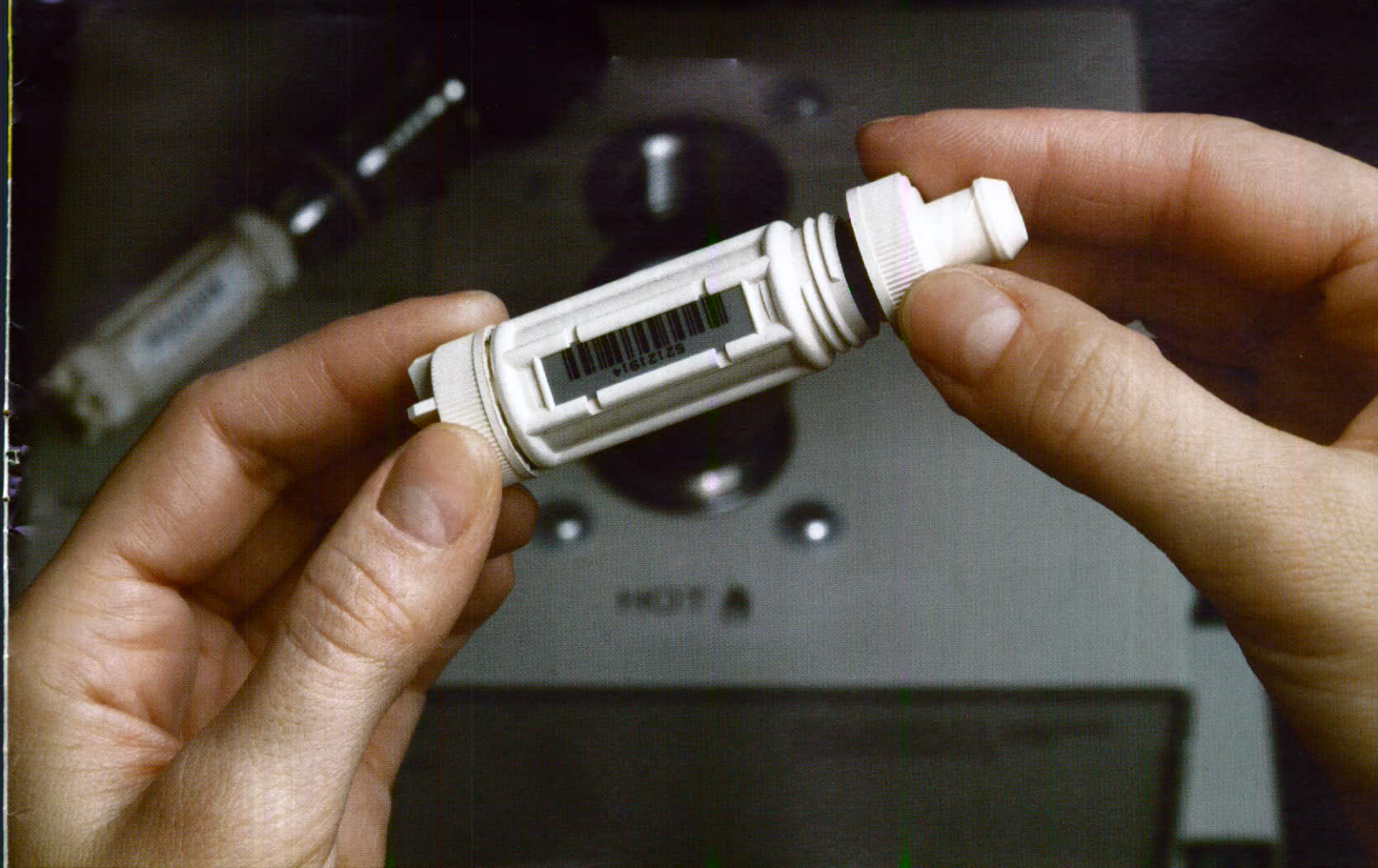
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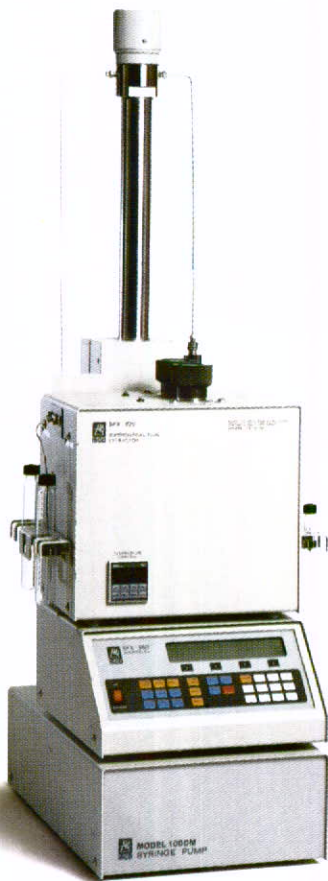
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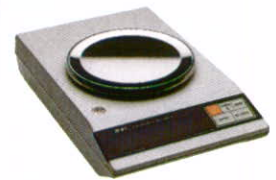
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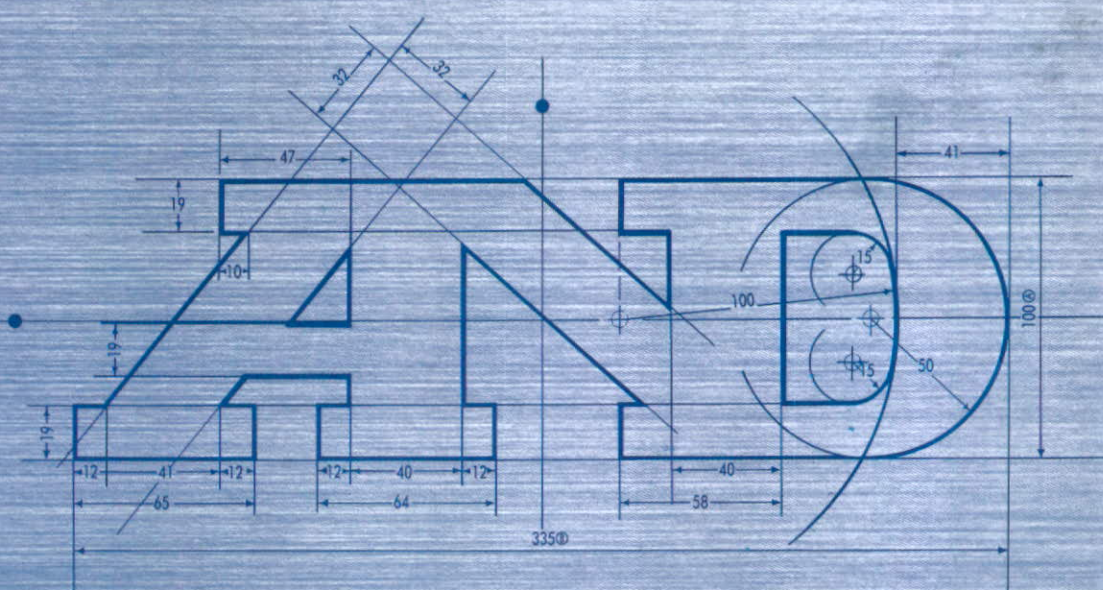
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