



Chemistry

IN NEW ZEALAND

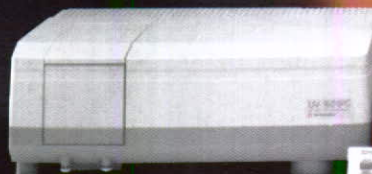
ISSN 0010-5566

FOCUS ON ENVIRONMENTAL CONTROL,
WASTE MANAGEMENT, WATER ANALYSIS



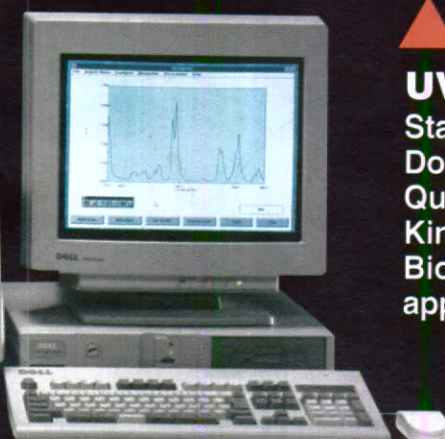
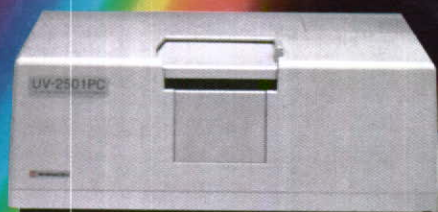
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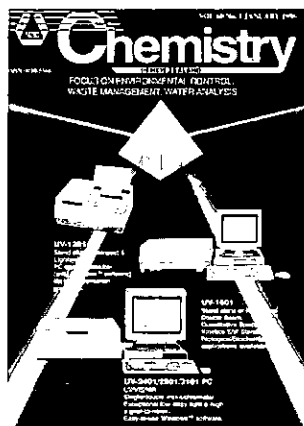
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For further details see the cover story on page 2



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COMING UP ...

March 1996 - Focus on Food and
Beverage Manufacturing

May 1996 - Focus on Forensics, Toxicology,
Chemical Pathology, Clinical Chemistry

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5th of the month of publication

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NEW ZEALAND NATIONAL COMMISSION FOR UNESCO APPOINTMENTS

The following people were recently appointed to the New Zealand National Commission for UNESCO by the Minister of Education, Dr the Hon. Lockwood Smith:

- Ms Roimata Kirikiri
- Professor Sylvia Rumball
- Professor Richard Bedford
- Brother Patrick Lynch
- Mr Patrick McCabe



Professor Sylvia Rumball

These members will be assisted by associate members from the Ministries

of Foreign Affairs and Trade; Education; Research, Science and Technology; Cultural Affairs; the Environment and the Department of Conservation. The new Commission will continue to be chaired by the Hon. Russell Marshall.

Professor Sylvia Rumball is Dean of Science at Massey University and a Fellow of the New Zealand Institute of Chemistry.

THERMOPLASTIC ENGINEERING APPOINTS TECHNICAL ADVISER

Ms Stacey McEwan has recently been appointed as Technical Adviser for Thermoplastic Engineering Limited, Miramar, Wellington.

Thermoplastic Engineering is a market leader in the design, manufacture and installation of laboratory fume management systems. The increasing demand from research institutions and a wide range of industries to meet new stringent health and safety legislation has made it necessary to increase resources in this sector.

Stacey's extensive laboratory experience will be a welcome addition to the company, she was previously employed by Courtauld Coatings and Griffins Foods. Stacey graduated from Massey University in 1992 after completing a Bachelor of Technology (in Bio-Process Engineering).

NEW MATERIALS AND CORROSION CONSULTANT

A new Materials and corrosion consultancy offering quality services in the field of materials technology, metallurgy and corrosion engineering, will open in Auckland in January 1996. "Les Boulton and Associates Limited" will be available to assist companies to maintain their competitive edge by providing up-to-the-minute advice on materials, corrosion and corrosion prevention, thereby avoiding costly failures and the potential for mistakes.

Les Boulton, principal of this new venture, has been a consultant, researcher and lecturer in corrosion control for the past 22 years

and has worked for the DSIR, a private consultancy, and more recently, Industrial Research Limited. Les Boulton and Associates Limited will continue to be associated with IRL in the future and the two companies may work on some joint projects.

Les Boulton has worked on many assignments, both in New Zealand and overseas, for a wide range of clients and industries, including pulp and paper manufacturing, food and chemical processing, petrochemicals, stainless steel fabricators, the marine industry and building services. He has also been associated with other consultancies including engineering consultants, loss adjusters, and legal firms. A particular service offered is the running of in-house seminars for companies whose products are vulnerable to degradation which could lead to product liability claims if corrosion occurs in service.

For further information contact:

Les Boulton, Brooke House, 24 Balfour Road, Parnell, Auckland, P O Box 101261, North Shore Mail Centre. Phone (09) 4783003, Fax (09) 4783713, and mobile phone 021 478300.

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POSSIBLE ICP CONFERENCE

A number of interested parties are investigating the possibility of holding a conference on ICP (Inductively Coupled Plasma Spectrometry) in Hamilton in November 1996. It is envisaged that there would be separate days devoted to ICP-MS (Inductively Coupled Plasma-Mass Spectrometry) and ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrometry) with perhaps a total of three days in all. Anyone with an interest in such a conference should contact:

Dr Peter Robinson

R J Hill Laboratories Ltd.

P O Box 4048 Hamilton

Ph: (07) 8552266, Fax: (07) 8549886, Email: peter@rjhill.co.nz



ENVIRONMENTAL ISSUES



THE MINISTRY FOR THE ENVIRONMENT'S ORGANOCHLORINES PROGRAMME

BULLETIN NUMBER 2

Norman Thom reported on Bulletin No. 1 of the Ministry for the Environment's Organochlorine Programme in *Chemistry in New Zealand* 59, 6 (November 1995). The following is Bulletin No. 2 in full. *Ed.*

Bulletin 1 (July 1995) introduced the Organochlorines Programme with a statement of draft objectives and expected outcomes. This Bulletin provides an update on:

- The Organochlorines Consultative Group;
- Organochlorines in the NEW ZEALAND Environment;
- National Environmental Standards;
- Organochlorine Destruction Technologies.

In the past decade, organochlorine contaminants and wastes have generated a high level of awareness, both nationally and internationally, within industry, Governments and communities. New Zealand's response to these concerns is to determine the status of these contaminants in our country and to set procedures to address them.

The programme focuses on 'dioxins' (PCDDs, polychlorinated dibenzo-*p*-dioxins, and PCDFs, polychlorinated dibenzofurans); PCBs; and the organochlorine pesticides which were widely used in the past within agriculture and industry e.g. DDT, aldrin, dieldrin, chlordane, and pentachlorophenol (PCP) (see Box 1).

ORGANOCHLORINES CONSULTATIVE GROUP

The Ministry for the Environment has convened an Organochlorines Consultative Group as a source of expert advice and to facilitate effective communication and consultation during the programme. The first meeting of the group was held in October 1995. The membership of the group is listed below:

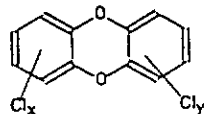
Howard Ellis	Ministry for the Environment (chair)
Dr. Simon Buckland	Ministry for the Environment
Jim Waters	Ministry of Health
Dr. Bill Jolly	MAF Regulatory Authority
Paul Dell	Local Authorities
Mark de Bazin	Timber Industry
Peter Sligh	Pulp and Paper Industry
Dr. Jim Barnett	Agricultural Sector
Bob Moffat	AGCARM, NZCIC
*	Incineration Industry [to be appointed]
Norm Thom	CAE, IPENZ, NZIC, WMINZ
John Hohapata	Adviser on iwi
Jocelyn Keith	National Council of Women
Simon Towle	ECO
Michael Szabo	ECO
*	Technical specialists as required

The Role of the Consultative Group is to:

- advise the MFE (which reports to the Minister for the Environment);
- represent key stakeholders and a diversity of community interests, and contribute individual expertise; advise on programme objectives, directions, priorities, management and progress; guide task-specific working parties; facilitate and coordinate information gathering and dissemination; identify additional resource requirements;

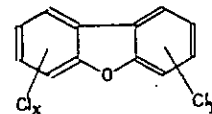
Box 1: Organochlorine Contaminants

(x, y = number of chlorine atoms)



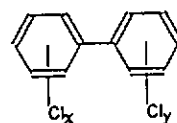
$$(x + y = 1 - 8)$$

PCDDs



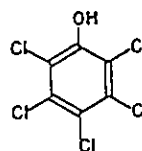
$$(x + y = 1 - 8)$$

PCDFs

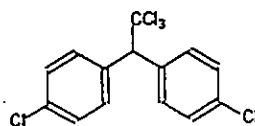


$$(x + y = 1 - 10)$$

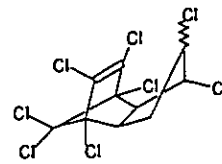
PCBs



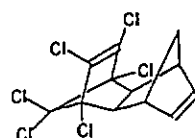
PCP



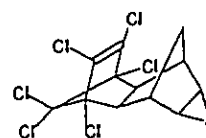
p,p' - DDT



Chlordane



Aldrin



Dieldrin

The Ministry for the Environment has undertaken to:

- work with the Consultative Group to consult effectively with interested parties;
- take account of current policy and environmental, social and economic factors;
- widely disseminate key information using non technical language;
- inform and facilitate an understanding of the issues among interested parties;
- provide suitable opportunities for, allow time for and take account of, comments and submissions.

The Consultative Group is currently reviewing:

- the objectives of the programme;
- the study design for the assessment of the New Zealand environment;
- a protocol for destruction technology trials;
- a communications strategy for the programme

ASSESSMENT OF ORGANOCHLORINE CONTAMINANT LEVELS IN THE NEW ZEALAND ENVIRONMENT: STUDY DESIGN

This study is the first stage of a comprehensive three-year programme to assess the significance of organochlorines in the New Zealand environment. The study design is currently being peer reviewed by New Zealand and international experts.

The key objectives of this study are to:

- *Provide data on the level of key organochlorine contaminants within New Zealand ecosystems.*
- *Enable the level of contamination of the New Zealand environment to be seen in an international context*
- *Support the development and application of national environmental standards or guidelines for these substances*

The first set of environmental samples are timetabled for collection during January 1996 and will continue at intervals over the next twelve months.

To assess the level of organochlorine contamination within New Zealand ecosystems, the approach being proposed is based on the determination of typical or average environmental contaminant concentrations. In developing this study therefore, consideration was given to the following issues:

- the need to be consistent with international studies in terms of sample collection and analytical procedures;
- the environments to be sampled (air, soils, rivers and estuaries) need to be representative of New Zealand with a broad coverage of the country; they will include relatively pristine places (e.g. national parks) and farmland, as well as city, industrial and urban areas.
- data from previous studies will be taken into account.

[A description of this study will be the focus of Bulletin 3; further study documentation is available on request from Simon Buckland, Ministry for the Environment]

NATIONAL ENVIRONMENTAL STANDARDS TO BE DEVELOPED

In implementing the Organochlorines Programme, the Ministry intends to develop a suite of National Environmental Standards (NES) for dioxins:

- these will be promulgated under s.43 of the Resource Management Act 1991; an explicit process of public consultation will be designed.
- they will apply nationally;
- they will be used to determine clean-up criteria for contaminated sites and emission standards for RM Act consents, including consents to operate destruction technologies;
- criteria will be determined for the protection of ecosystems (based on a national protocol) as well as for the protection of human health (based on exposure scenarios for a range of land uses); findings from the assessment of background levels in the environment will be incorporated;
- experience gained in the development of Health and Environmental Guidelines for Selected Timber Treatment Chemicals (which included criteria for PCP and interim criteria for dioxins) will be carried forward;
- criteria and standards established in other countries will be examined for their relevance to New Zealand;

- the current Government policy on the toxicology of dioxins will be re-examined in the light of the US EPA dioxin reassessment, and any other international reviews of dioxin toxicity.

ORGANOCHLORINE DESTRUCTION TECHNOLOGIES

Organochlorine wastes requiring treatment or destruction may arise from a variety of sources, including contaminated soils, sediments, building materials, chemical stockpiles and materials from treatment ponds and waste dumps. The chemical stability of the organochlorine substances of concern, a feature which contributes to their persistence in the environment, also makes them difficult to destroy.

Internationally the most widely employed means to destroy organochlorine chemicals and contaminated materials is high temperature incineration (HTI). However in recent years, public opposition to HTI has increased because of uncertainties over its potential for dioxin emissions. After vigorous local opposition to the proposed use of a cement kiln to destroy stocks of PCBs, New Zealand has exported its major holdings of PCBs for destruction in a dedicated hazardous waste HTI facility overseas, principally in France.

The search for alternative means to safely destroy organochlorine substances, particularly PCBs, has involved considerable research and development worldwide. To help identify destruction technologies appropriate to PCP and dioxin contaminated material, the Ministry for the Environment and the Timber Industry Environmental Council contracted overseas consultants to report on all proven or potential technologies'

Only three technologies were identified for solid wastes:

- High Temperature Incineration (HTI)
- Base Catalysed Dechlorination (BCD)
- The EcoLogic process

High Temperature Incineration

HTI is a collective term applied to a range of proven incineration technologies which achieve high destruction efficiencies. HTI's of possible interest to New Zealand include rotary kilns, "plasmox" (plasma arc heated system), fluidised bed combustion, and controlled air incineration (pyrolysis). A high level of treatment efficiency and reliability can be achieved in modern installations that incorporate appropriate pollution abatement systems. Treatment residues in the form of clinker or slag are normally disposed of in a landfill.

However any proposal to use HTI for the disposal of hazardous waste could be expected to focus public interest and concern on the possibility of dioxin formation and emission from such a facility.

Base Catalysed Dechlorination (BCD)

The BCD technology is a recent innovation developed and patented by the US Environmental Protection Agency (USEPA) Risk Reduction Engineering Laboratory. BCD involves a reaction which sequentially strips chlorine atoms from organochlorine molecules and substitutes them with hydrogen atoms derived from an oil. The reaction, which requires the addition of a proprietary catalyst, takes place over several hours and at elevated temperatures to yield a completely dechlorinated organic molecule and common salt. Commercial BCD units are now operating under license in the USA and in Australia.

ADI Services, a BCD licensee in Australia, have developed a variation of the BCD reaction (called the 'ADOX' reaction) in which the patented BCD catalyst is replaced by an 'accelerator'. In the ADOX reaction the nature of the reaction changes dramatically in that PCP molecules, for example, are decomposed completely to carbon. The reaction, which takes place rapidly, can be applied to much higher concentrations of organochlorines than the conventional BCD process and without the requirement for the addition of oil.

In developing technologies of this type, a significant challenge is to destroy the organochlorine contaminants bound up within soil and other solid materials. One approach is to pretreat the contaminated materials within a thermal desorption unit (TDU). The vapourised contaminants are then condensed and destroyed in the BCD reactor. The Ministry for the Environment, in co-operation with the timber industry corporates, recently contracted the Institute of Environmental Science and Research Ltd (ESR) and ADI Services to undertake treatability trials on a sample of PCP and dioxin contaminated soil. The results to date indicate excellent destruction efficiencies from the combined use of TDU and the ADOX reaction. Further trials are to continue in 1996.

The EcoLogic Process

Another technology has been developed by EcoLogic International Inc (ELI). The technology has been successfully

demonstrated to completely degrade PCP, dioxins and other organochlorine substances as well as polycyclic aromatic hydrocarbons (PAHs).

When applied to the treatment of soil, the contaminants must first be vapourised by a thermal reduction mill (TRM). The unit adopted by EcoLogic comprises a ball mill (to achieve a finely divided soil) operating at high temperatures on a molten tin bath. The vapourised organics are swept into the EcoLogic reactor with a stream of gas (mainly hydrogen and steam). Chemical decomposition reactions take place in the reactor at 900 °C in a hydrogen atmosphere to yield principally methane, carbon dioxide and hydrogen chloride (recovered as hydrochloric acid).

The technology has been successfully audited by both the US EPA during a trial treatment of dioxin contaminated soil, and also by Environment Canada during the treatment of PCB and PAH contaminated harbour sediments. Environmental Solutions International (ESI) and ELI have recently commissioned the first commercial scale EcoLogic plant at Kwinana, south of Perth. This plant is destroying stockpiles of organochlorine pesticides (predominantly DDT) and PCBs. Independent assessment indicates a greater than 99.9999% destruction removal efficiency.

The success of both the BCD/ADOX and the EcoLogic technologies relies on effective materials handling and an efficient TRM pre-treatment phase.

Other Innovative Technologies

- UV light is being used in conjunction with hydrogen peroxide to successfully treat PCP and dioxin contaminated groundwater at the Forestry Corporation of New Zealand's Waipa timber processing plant near Rotorua;
- A number of other technologies can be applied to contaminants dissolved in water but have yet to be seriously considered for the treatment of organochlorine contaminants in soil. These include: "plascon" (in-line plasma arc), high energy electron beam irradiation; gamma irradiation; silver II process; and supercritical water oxidation;

Selected fungal or bacterial species may offer the prospect of bioremediating PCP contaminated soils at a lower cost than thermochemical methods. However whilst bioremediation has been demonstrated to degrade PCP, it does not appear able to degrade the dioxin microcontaminants of PCP.

Technology Performance Criteria

The introduction to New Zealand of a technology to destroy a hazardous waste or achieve site clean-up must address a number of issues:

- The technology must be cost effective. There are a number of aspects to this:

⇒ the technology must be able to meet clean-up or destruction efficiency targets;

⇒ it must be affordable, yet at the same time it must achieve the required clean-up criteria. The development of health and environmental standards are therefore fundamental to the

selection of an appropriate technology;

⇒ the technology must provide value for money. Does it offer a final solution to the contamination problem (e.g. degradation of the contaminants to acceptable end products), or does it transfer the problem to future generations (e.g. containment, immobilisation, land filling)?;

⇒ versatility - is the technology effective on a range of contaminants and materials?;

⇒ what is the nature and extent of pre-treatment required?;

⇒ is the treatment facility small scale and relocatable (i.e. can it be moved from site to site) or would a regional facility be needed?

• The technology must be satisfactory and safe to operate from a number of standpoints:

⇒ occupational, safety and health standards relating to workers and the work place;

⇒ land use consents concerning the siting, installation and operation of the unit;

⇒ the transport of hazardous substances to and from the installation;

⇒ regional council consents for any emissions to air, water or land during its operation;

⇒ consents to satisfactorily dispose of wastes comprising by-products or end products.

• of underlying importance to technical and performance requirements is the reliability and robustness of the operation. In other words the technology must pass a 'warrant of fitness' as tested by certified HAZOP/HAZAN risk assessment procedures.

• Acceptance by the general public on the safety and desirability of any technology is a major consideration. The community will need to be satisfied that the technology is safe.

Any technology to destroy organochlorines will therefore need to undergo a considerable degree of technical and public scrutiny before it is judged to be 'acceptable' and 'safe'.

Full reports available from the Ministry for the Environment.

For further information contact the NZIC representative on the Organochlorines Consultative Group:

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The University of Auckland

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Auckland

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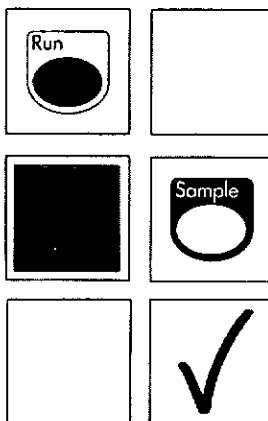
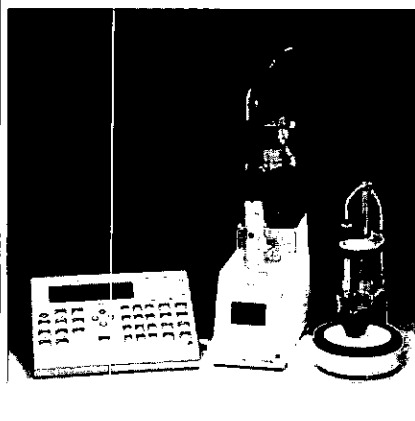
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Hewlett Packard Company now is using the World Wide Web to communicate with biochemists and analytical chemists. By offering a separate analytical directory within HP's Web home page, Access HP, HP now provides scientists with the following:

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In the future, HP plans to add application notes, service and support information, a columns and consumable catalogue, and training and events information.

AMERICAN PHYSICAL SOCIETY SELECTS 1996 RECIPIENT OF HIGH POLYMER PHYSICS PRIZE

The American Physical Society has announced that the winner of its prestigious 1996 High Polymer Physics Prize is Dr Alan N Gent, The Harold A Morton Professor Emeritus of Polymer Engineering and Polymer Physics at The University of Akron. An internationally recognized expert on the physical behavior of polymers, Gent served on the National Research Council panel that evaluated the solid-rocket boosters believed responsible for the 1986 Challenger Space Shuttle accident. For his participation on that panel, he received from astronaut Michael A Baker the "Silver Snoopy," a NASA award recognizing work that enhances the probability of space mission success.

The American Physical Society prize recognizes outstanding accomplishment and excellence of contributions in high polymer physics research. Gent was selected because of his "fundamental contributions to the physics of adhesion and fracture of elastomers," according to the society. The annual award was established by Ford Motor Company in 1960 and includes a certificate citing the recipient's contributions and a US\$5,000 prize.

The award will be presented to Gent at the American Physical Society general meeting in St. Louis, Missouri, USA in March 1996. More than 41,000 physicists around the world are members of the American Physical Society, which was founded in 1899 for the advancement and diffusion of the knowledge of physics.

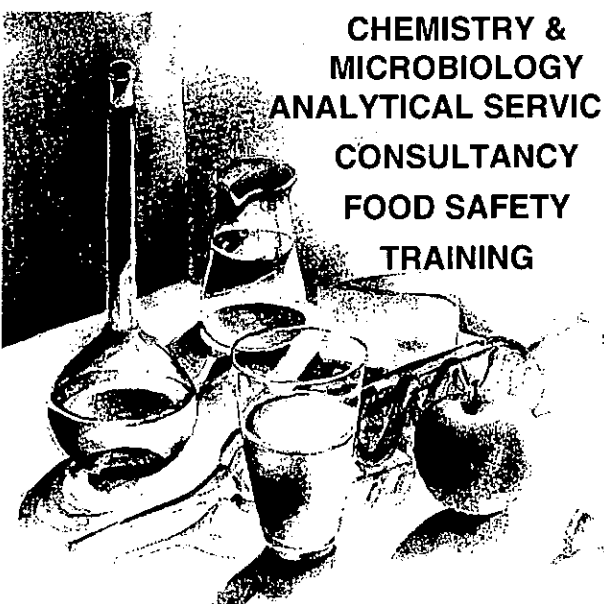
Gent left his native England in 1961 to join The University of Akron, Ohio, USA. He spent more than three decades at The University of Akron, during which time his abilities and achievements as a researcher in the area of deformation and fracture of polymers brought honours and accolades to him and the institution. He has been honoured by the American Society for Testing and Materials, the Plastics and Rubber Institute, the Society of the Plastics Industry and the Society of Rheology.

Most of Gent's major awards were received in the last five years. In 1990, the American Chemical Society's Rubber Division awarded Gent its highest honour, the Charles Goodyear Medal. The following year, Gent was named to the prestigious National Academy of Engineering, and received the "Silver Snoopy" award from NASA. In early 1993, he was inducted into the Ohio Science, Technology and Industry Hall of Fame in Columbus, Ohio, USA.

Gent retired from The University of Akron and received emeritus status in June 1995. He continues collaborative research at The University of Akron with Dr Gary Hamed, Professor of Polymer Science and Biomedical Engineering. Gent also is a science adviser to The Goodyear Tire and Rubber Company.

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LETTER FROM AUSTRALIA

One of the Grand Old Men of Australian chemistry is Arthur Birch, Emeritus Professor at the Australian National University in Canberra. Arthur has always taken an interest in the history of his beloved organic chemistry and owns a fine collection of old books, and it is always a delight to read what he has to say about the things that interest him. In the *Notes and Records of the Royal Society of London* for 1993 (volume 47, number 2, pages 277-296) he writes about the tropinone synthesis of Sir Robert Robinson and reflects on just how Robinson came to the first biomimetic synthesis. In 1917 Robinson published the results of his work with a 'soup' containing succindialdehyde, acetonedicarboxylic acid and methylamine. From this mixture, at physiological pH and in the cold, Robinson isolated tropinone, a substance with the same nitrogen-bridged seven membered ring structure as cocaine.

Birch is exceptionally well qualified to write about Robinson because their careers intersected in a number of ways. Birch worked in Robinson's laboratory at Oxford for ten years and, upon the older man's death, wrote (in 1976) an obituary and assessment of his scientific career. In between these two biological markers, Birch was professor of organic chemistry at Sydney from 1952 to 1955, occupying a chair whose first incumbent was Robinson (1913-1915).

When Birch took up his appointment he looked in the store for traces of his illustrious predecessor, and 'found some obvious relics of his scientific interests: a large quantity of morphine, some steroidal alkaloids and, astonishingly to me, a large bottle of calcium acetonedicarboxylate, the key intermediate in his 1917 tropinone synthesis'. A recent check showed that the bottle was no longer in the store, and also failed to locate any publication from the Sydney department which mentioned its use.

Birch asks himself 'Did Robinson initiate the tropinone synthesis in Sydney?' Persistent asking in 1952 finally led Birch to the department's oldest inhabitant, Julius W Hogarth, who had been Robinson's personal assistant and who recalled that 'RR wished to condense succindialdehyde with acetonedicarboxylic ester using diethylamine as catalyst. But, there was none of that in the store: the only amine available was methylamine so he used that instead'. It is only in recent years that a wide range of chemicals has become readily available in Australia, and so Hogarth's story rings true, and Birch observed that earlier 'synthetic programs tended to be organized around what was already in store ... (or) ... which grew in the local bush'.

Birch goes on to explore the ideas that Robinson drew on in designing his synthesis, notably that of Thiele who has used diethylamine as catalyst for the preparation of benzocycloheptadienones. Following that logic, the nitrogen bridge would be formed last, whereas with methylamine as catalyst it was possible to form from the dialdehyde an intermediate having a five membered ring - a 1,5-dihydroxypyrrolidine, dubbed a pseudobase - with the three-carbon unit being added in the second step. Birch also discusses the work of Winterstein and Trier, and of Willstätter, that Robinson was able to draw on.

Birch is mildly critical of 'historians of science who were not themselves active participants in creative science' and says that

much of his history of scientific ideas he located in the experimental sections of papers and in the footnotes. The detailed discussion contains a number of Birch gems, including the comment that Robinson, in contrast to senior German professors, liked to work at the bench and 'in 1914 largely destroyed by fire a Sydney laboratory, he told me, through a melted rubber gas-tube to a Bunsen burner beneath an overheated sand-bath.

'Robinson's pregnant ideas in this field did not emerge in mystical fashion', Birch concludes, as he reflects that 'developments in a rational scientific field can nevertheless be subconscious' and consigns to legend the idea that it was Robinson's biogenetic ideas that led to his synthesis of tropinone. Forty years in preparation, Birch's paper is a great read, but published in a place that most organic chemists would be unlikely to come across it. They would be even less likely to stumble on the 1976 assessment of Robinson's career because it was published in the *Journal and Proceedings of the Royal Society of New South Wales*. I can recommend it, also, to the reflective practitioner.

Professor Ian D Rae
Deputy Vice-Chancellor
Victoria University of Technology
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PESTICIDE RESIDUES

Finding Out More And More About Less And Less

Patrick T Holland, Food and Biological Chemistry Laboratory,

Horticulture and Food Research Institute, Ruakura Research Centre, Private Bag 3123 Hamilton

Introduction

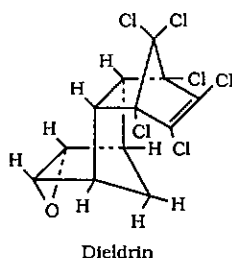
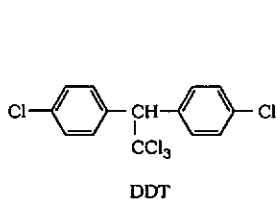
Pesticide residues have been a catch cry of environmental and consumer groups since the mid-1960s when Rachel Carson drew the public's attention to the deleterious ecological effects of organochlorine insecticides, which were in widespread use at that time. The complex role of pesticides in modern society with the associated problem of pesticide residues has undoubtedly contributed to the disenchantment with chemistry that is prevalent in Western society, often fuelled by misleading propaganda from special interest groups on residue issues.

Pesticide residues are closely regulated on foods in most countries using Maximum Residue Limits (MRLs) which are based on recognised safe and reasonable use patterns. Increasing attention is also being given to environmental issues, particularly to sensitive resources such as estuaries and water. The proposed moving of pesticide regulation in New Zealand from under the wing of the Ministry of Agriculture and Fisheries to the Ministry for the Environment (under an authority to be established under the Resource Management Act, 1991) recognises this need to take a very broad view.

Knowledge on residues has depended on advances in analytical chemistry, particularly Gas Chromatography (GC), combined Gas Chromatography-Mass Spectrometry (GC-MS) and High Performance Liquid Chromatography (HPLC). This article outlines some issues regarding pesticide residues in New Zealand with particular reference to the role of the analytical chemist.

Organochlorine Insecticides (OCs)

DDT (1,1-diparachlorophenyl-2,2,2-trichloroethane) was widely used in New Zealand pastoral agriculture, and dieldrin or lindane to a lesser extent. In the 1960s we followed other countries and banned widespread use of OCs. In our pragmatic fashion, this was largely based on concerns about meat products, in which residues were coming under regulation in export markets.



Chlordane was used in the treatment of plywood until recently which has led to the contamination of some industrial sites with flow on effects to adjacent areas such as the Manukau harbour. Dieldrin had a number of minor registered uses until recently.

The OCs are resistant to microbial degradation and have a propensity to concentrate in lipid-rich tissues. These properties lead directly to their most undesirable characteristics - the environmental persistence, bioconcentration, and biomagnification through the food chain of their residues. They share these characteristics with two other notorious classes of contaminants, the polychlorinated biphenyls (PCBs - electrical insulating fluids) and polychlorinated dibenzodioxins (PCDDs - by-products of manufacture of some organochlorines and of incineration). Pentachlorophenol (PCP), another timber treatment chemical that has been poorly regulated, is a major source for PCDDs in New Zealand.

While residues of DDT, and its primary metabolite DDE (1,1-diparachlorophenyl-2,2,2-dichloroethene), have declined to low levels in the soils of most parts of New Zealand (less than 0.1 mg/kg), levels of 1-5 mg/kg are not uncommon on farms in Canterbury, where DDT use was high and where dry land conditions have lead to very slow degradation rates. High soil residue levels are an intractable problem, which can result in excessive residues in meat or dairy products. Reducing them will depend on enhancing bioactivity in the soils including the use of irrigation.

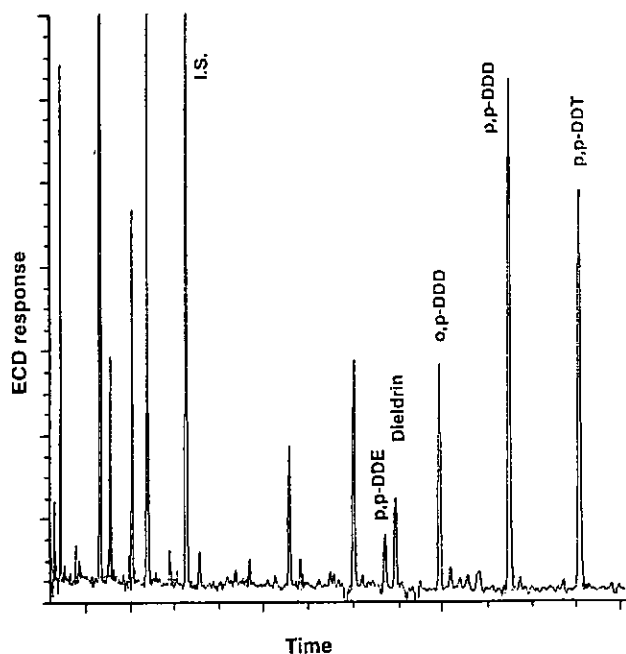


Figure 1: Electron capture detector gas chromatogram of an extract of a marine sediment containing 29 µg/kg total DDT + metabolites and 2 µg/kg dieldrin (I.S. = internal standard)

OC residues are detectable in most of our estuarine areas and are bioconcentrated in shellfish and other biota in these ecosystems. The levels are not particularly high by international standards except in localised, highly contaminated sites adjacent to industrial areas such as the Mangere Inlet in Auckland (1). The gas chromatogram of an extract of a marine sediment (Figure 1) illustrates the power of modern analytical techniques.

The high resolution capillary column and optimised electron-capture detector (ECD) enable detection limits of 1 µg/kg dry weight (1 ppb). The chromatogram reveals the presence of DDT, DDD, DDE, and dieldrin each in the 2-20 µg/kg range. Concentrations of OCs in organisms dwelling in this sediment can be expected to be 10-20 times higher - the start of biomagnification up the food chain which can lead to damaging levels in marine mammals.

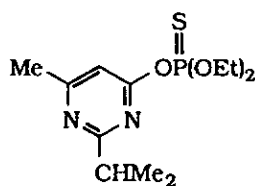
Humans also carry body burdens of OC residues accumulated mainly through diet. The significance of these residues is a subject of intense interest. At typical levels (less than 1 mg/kg on a fat basis) they are generally thought to be relatively inert and non-carcinogenic. However, current biomedical research is pursuing the theory that some OCs and other xenobiotics (compounds that are foreign to the body) mimic estrogens, leading to increased susceptibility to carcinomas in fatty tissues such as the breast.

Organophosphorus (OP) and carbamate insecticides

The banning of OCs encouraged the development of a wide range of OP and related carbamate insecticides, which act by inhibition of insect nervous-system cholinesterase. Early products such as parathion also had very high mammalian toxicities (LD50 rat <5 mg/kg body weight), which made them very hazardous to use. However, more recent OPs are much less toxic (e.g. pirimiphos methyl; LD50 rat 2000 mg/kg), and are widely used in agriculture.



pirimiphos-methyl



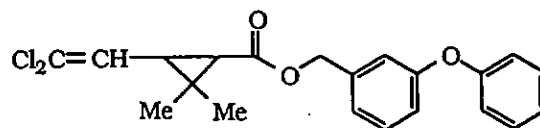
diazinon

OPs are readily deactivated and degraded in mammals and by microorganisms, and therefore do not accumulate. Despite widespread use for over 30 years, and close scrutiny by modern techniques, residues from agricultural uses are virtually never found in wider environmental samples. Ecological ill-effects are believed to be limited to reductions in the populations of some beneficial insects in sprayed areas. Sensitive analysis for residues can be carried out by GC with detectors selective for phosphorus (Flame Photometric Detector (FPD), or Nitrogen-Phosphorus Detector (NPD)).

Synthetic Pyrethroid Insecticides (SPs)

SPs are structural analogues of the natural pyrethrins and have a mode of action on insect nerve junctions similar to the OCs. Permethrin is one of the most commonly used. They have good biodegradability due to the ester linkage and are used at very low rates (5-50 g per hectare); consequently environmental residues are uncommon but may be expected where industrial effluents accumulate. Their principle disadvantage is the very broad insecticidal activity which tends to eliminate many beneficials. Residue analysis (generally GC-ECD) is

complicated by the range of isomers (R,S or cis/trans) that may be present.



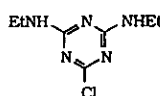
permethrin

Fungicides

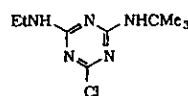
Fungicides are widely used on crops in New Zealand as the climate encourages growth of plant pathogens. A diverse range of chemical classes has been developed with the emphasis in recent years being on biodegradability. As with modern insecticides, residues are rarely found in the wider environment. An exception is copper, which accumulates in soils under crops treated with cupric oxide or the hydroxide (Bordeaux mix) - a conundrum for 'organic' proponents and a serious problem in Kenya, where decades of heavy use have led to phytotoxic levels in the soils of coffee plantations.

Herbicides

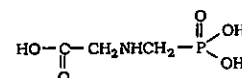
This is the most important sector of the New Zealand pesticide market and contains an ever increasing diversity of chemicals. While many herbicides are of limited environmental significance, due to rapid degradation or strong adsorption e.g. glyphosate, some herbicides designed for longer-term control have more mobile and persistent residues with the potential too contaminate the wider environment. A number of herbicides have been implicated in contamination of ground water resources in North America and Europe. Recently, low-level residues of atrazine, terbutylazine and simazine have been detected in some wells in South Canterbury.



simazine



terbutylazine



glyphosate

Figure 2 shows a region of the GC trace for a water extract prepared by solid-phase extraction (SPE) using C₁₈-silica impregnated filter disks. The NPD allows selective detection of the triazine residues at levels below 0.05 µg/L. HPLC is also very suitable for trace analysis of herbicide residues in water.

The sulfonyl-urea class of herbicides has come into prominence recently owing to their very high activity against many weed species, and their very low mammalian toxicity. They act by specific inhibition of an enzyme in the pathway for biosynthesis of valine and isoleucine in plants. Residue analysis can be carried out by HPLC, but has been difficult due to the very low rates of application. Recently we have developed conditions under which diazomethane can be used to produce the thermally stable dimethyl derivatives of sulfonyl-ureas (Figure 3) so residue analysis can be carried out by GC (2). Figure 4 shows the GC-MS selected-ion chromatogram for detection of several sulfonyl ureas in water at 0.1 µg/L. The method has also been applied to field and laboratory experiments that showed metsulfuron residues in soil degrade with a half-life of about 20 days.

Residues in the Diet

Although modern insecticides and fungicides are readily degraded in the environment by soil microorganisms, residues on treated crops, such as fruit or vegetables, often do not dissipate quickly. Residues still present at harvest are regulated through MRLs, which in New Zealand are set in the Food Regulations.

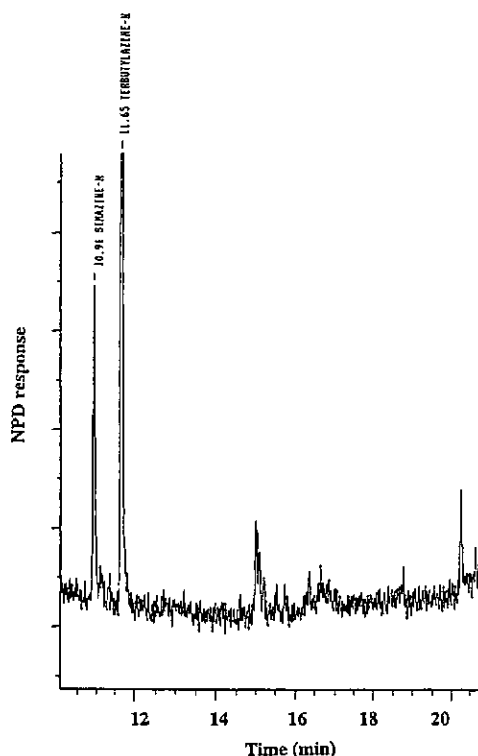


Figure 2: Nitrogen-phosphorus detector gas chromatogram of an extract of a ground water sample containing simazine (0.10 µg/L) and terbutylazine (0.51 µg/L).

A combined Department of Health/Ministry of Agriculture and Fisheries survey of fruit and vegetables was carried out in 1991/92 (3). Multi-residue analytical techniques were used, which can detect a wide range of residues. Sub-samples of crops were extracted with ethyl acetate and cleaned by gel-permeation chromatography to remove lipids and pigments. High-Resolution Gas Chromatography (HRGC) with effluent splitting to ECD and NPD allowed detection of over 80 pesticides. Figure 5 shows chromatograms for standards and an tomato sample containing residues of pirimiphos-methyl and permethrin. The large peak on both channels is the internal standard, carbophenothion. A similar method has been developed for multi-residue analysis of wine and juices, which uses SPE to concentrate residues (4).

Over 52% of the 740 samples tested in the survey had no significant residues and 43% had residues below set MRLs. 5% of samples had residues that either exceeded a set MRL or for which there was no set MRL for that pesticide on that particular crop. Most of the MRL violations were from moderate residues due to unregistered uses of a pesticide approved for other crops rather than uses of banned products or levels exceeding set MRLs. The results were in broad agreement with residue monitoring in other countries and confirmed Department of Health rankings, which put microbial contamination of food as a much more serious health problem than residues.

Most pesticides lack systemic action, and therefore residues are found mainly on exterior surfaces, where they are amenable to removal in operations such as trimming, washing or peeling that most crops undergo before consumption. Further losses can arise during cooking. These factors all greatly reduce the exposure of consumers to pesticides in the diet. A review of the extensive literature on the effects of storage and processing on residues in food has recently been carried out by the IUPAC Commission on Agrochemicals (5).

Export Surveillance

While environmental and dietary concerns are of increasing importance to pesticide policy, the issue of residues in our export agricultural products continues to dominate thinking in the agricultural sector. Very large financial and technical inputs are made by the major commodity groups (meat, dairy, pip fruit and kiwifruit) into residue testing to ensure consignments meet the requirements of various overseas markets. Multi-residue techniques are supplemented by ELISA format immunoassays (6). Residues found in meat and dairy products are usually confined to low levels of DDE/DDT but a wide range of fungicide and insecticide residues may be present on horticultural crops.

Meeting quarantine and MRL standards while producing blemish-free fruit can be a difficult exercise for growers, particularly as requirements vary between countries. Producer boards setting recommended spray programmes for growers need good insight into the residual properties of the pesticides. HortResearch has developed a predictive computer model, which extrapolates field data into residue estimates for particular spray scenarios (pesticides, dates of application, rates, date of harvest). The program is based on the following simple two-stage first-order model with three (pesticide-dependent) parameters for the quantity of pesticide remaining on a fruit at time t after spraying:

$$D_t = D_0(e^{-k_1t} + k_3e^{-k_2t})/(1-k_3)$$

The initial quantity of pesticide deposited (D_0) is determined by the rate of application and size of the fruit. The first exponential term accommodates initial rapid volatilisation of pesticide, while the second term covers slower losses of more firmly retained residues. A residue concentration is then calculated which allows for fruit growth over time t . This model has proved able to provide remarkably accurate predictions of residue levels on apples or kiwifruit. Figure 6 shows calculated and predicted residues for chlorpyrifos on kiwifruit through a growing season when six spray applications were made.

Spray Drift

This is a controversial issue affecting horticulture in two ways. Firstly, through crop damage due to drift from pastoral or forestry spraying of herbicides, and secondly, through the ire of neighbours subjected to drift from orchard spraying. Many crops are extremely sensitive to the 'hormone' herbicides such as 2,4-D or triclopyr. Analysis of affected foliage has revealed that residues as low as 2 µg/kg can indicate damaging exposure to drift. Reliable detection of residues at these levels requires GC-MS.

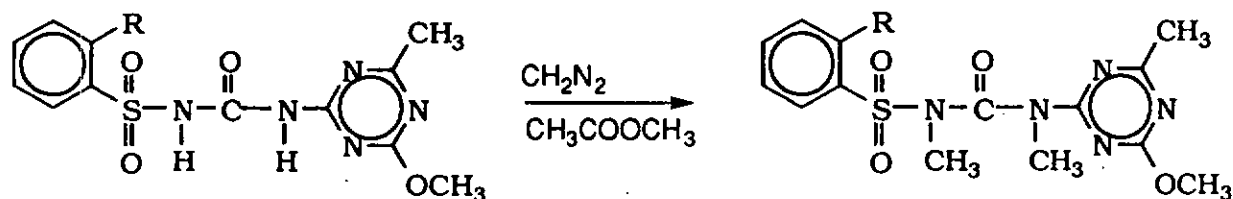


Figure 3: Derivatisation of residues of sulfonyl urea herbicides with diazomethane to form thermostable dimethyl derivatives

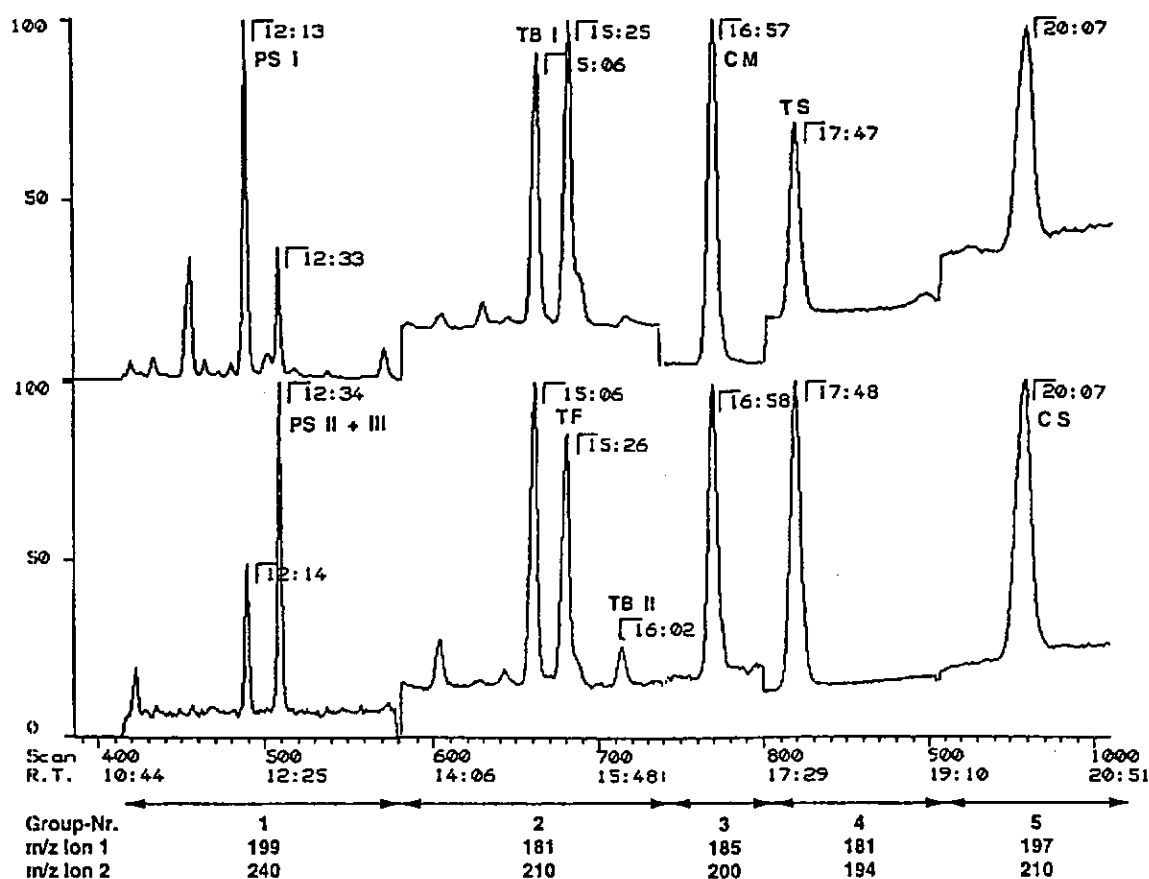


Figure 4: Reconstructed ion chromatograms of the confirmatory analysis by GC-SIM-MS of a water sample spiked with cinosulfuron (CS), chlorimuron-ethyl (CM), primisulfuron-methyl (PS), thifensulfuron-methyl (TF), triasulfuron (TS) and tribenuron-methyl (TB) 0.1 µg/L each

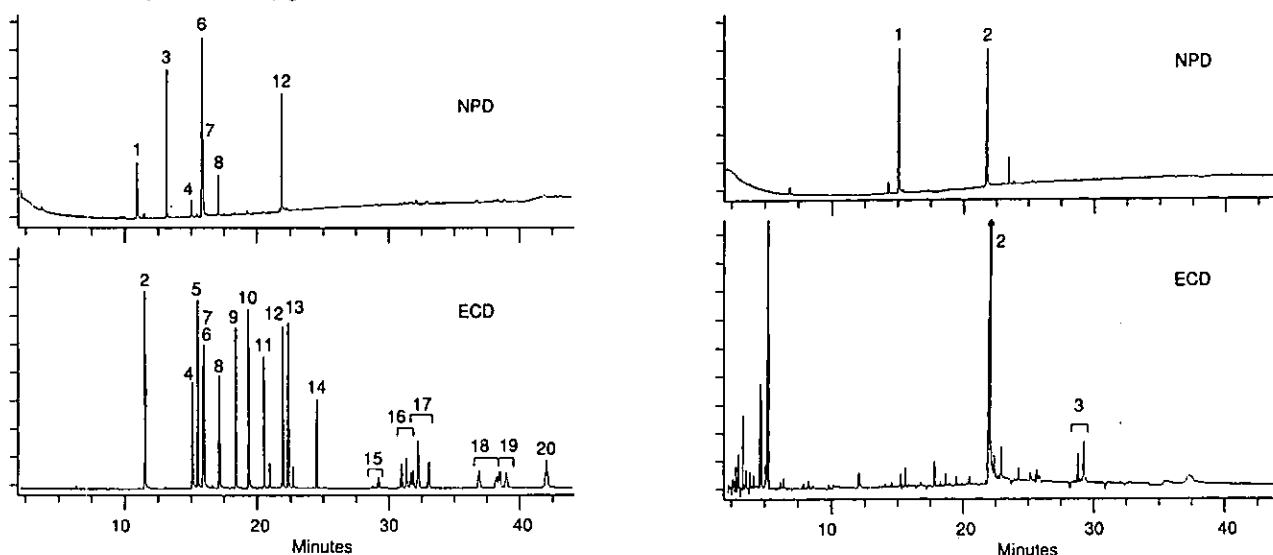


Figure 5a: Mixed pesticide standard (1 ng each) by HRGC using column effluent splitting to ECD and NPD: 1, simazine; 2, lindane; 3, pirimicarb; 4, bromacil; 5, aldrin; 6, triadimefol; 7, parathion-ethyl; 8, penconazole; 9, endosulfan-a; 10, dieldrin; 11, endosulfan-b; 12, carbophenothion; 13, p,p-DDT; 14, dicofol; 15, permethrin; 16, cyfluthrin; 17, cypermethrin; 18, fenvalerate; 19, fluvalinate; and 20, deltamethrin

Figure 5b: HRGC analysis of extract of tomatoes with incurred residues: 1, pirimiphos-methyl 0.17 mg/kg; 2, carbophenothion internal standard 0.4 mg/kg; and 3, permethrin 0.03 mg/kg

Pesticides from orchard spraying are detectable outside sprayed blocks. Deposited droplets can be extracted off filter papers and very fine particles or vapour-phase samples can be trapped in air samplers on polystyrene resin beads (XAD-4). Experiments have been conducted in kiwifruit orchards (7). Typical levels of deposited drift were 30-300 µg/m² at about 25 metres downwind from the shelter, which is less than 1% of deposits in the sprayed crop. Levels rapidly dropped with distance and were non-detectable beyond 50-100 m. Aerial drift levels were typically 0.5-5 µg/m³ with diazinon showing higher levels than the other less volatile pesticides. These drift levels are low by any objective health standards but may still cause

concern to close neighbours who see drift as an unwarranted intrusion from the orchard.

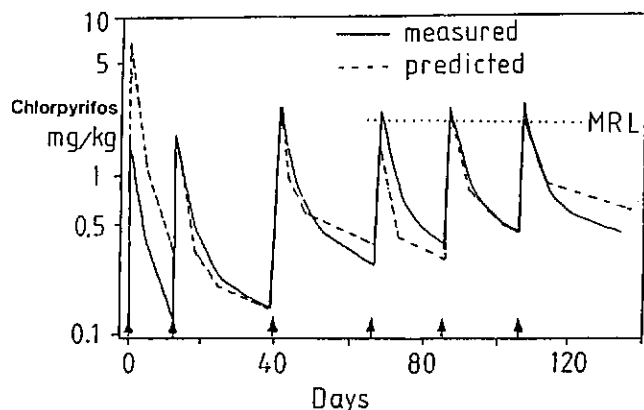
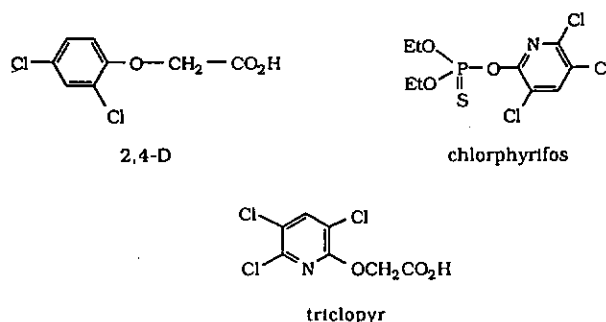


Figure 6: Predicted versus found residues for chlorpyrifos on kiwifruit following six sprays. Arrows indicate the time of application of the pesticide

Conclusions

The severe regulatory pressures that have operated for some years has meant that most registered uses of current pesticides are very safe in regard to both human and environmental health. Despite many scares and individual incidents, the overall risk/benefit analysis is actually rather good, particularly if one takes as a base the multiple and manifest problems caused by the OCs. However pressures are continuing to mount worldwide for even stricter standards and, as a small country highly dependent on agricultural exports, we must try to keep ahead of these trends. Residues in export commodities will be of even greater significance to New Zealand post-GATT as countries turn to non-tariff barriers. Thus analytical techniques for low level residues will remain a focus for research, development and regulation of pesticides.

Local communities are also demanding more say in agricultural practices and insisting on extremely high environmental standards. The recent collections of unwanted pesticides from farms by some local councils have contributed to decreasing the risks. All pesticides have the potential for misuse and community groups have a role in highlighting problems and assisting with education. Chemists must play their part in this process, or risk further alienation.

Acknowledgements

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NEW PRODUCTS

HP APPLICATION NOTE EXPLAINS HOW TO REPLACE CLASS A VOLUMETRIC GLASSWARE WITH THE HP PREPSTATION

Hewlett Packard Company has published an application note (Literature 5963-5259E) entitled "A Validation Study: Performing Dilutions with an HP PrepStation Compared to Manual Preparation Using Class A Volumetric Glassware".

The publication describes an extensive validation study that demonstrates how the HP PrepStation is equivalent to manual dilutions using Class A glassware in the range of dilution factors from two to two hundred. With validation established, the publication concludes that those laboratories using Class A glassware can move to automated instrumentation to increase productivity and decrease operating costs with only simpler equivalency testing required for validation of particular assays.

The 16-page, black and white application note includes experimental conditions, results, and statistical analyses for the validation study. It also discusses the benefits obtained by the authors when using the HP PrepStation for automated dilutions.

The application note is available without charge from any Medtec Products Ltd sales office.

Contact: Peter Hermans, Medtec Products Ltd
P O Box 34241 Northcote, Auckland
Ph: (09) 4806763, Fax: (09) 4806386
circle number 21 on the reader reply card

THE PLATE COUNTER REVOLUTION CONTINUES... TOTAL PERFORMANCE FOR BETA, GAMMA AND LUMINESCENCE COUNTING

Wallac Oy, of Finland, have continued the success of the Microbeta Plus with the latest plate counter, the Trilux.

Based on true liquid scintillation counting technology with coincidence counting, Trilux has the superior features of a top quality beta counter including single button operation, and single and dual label DPM results. However, Trilux is more than just a beta counter as without any modification, it can be used for gamma labels such as iodine and chromium as well as for the modern luminescence applications.

The additional capabilities include the ability for up to 4 mL tubes to be counted, improved luminescence performance and updated software. Existing customers can also upgrade their Microbeta Plus with the new larger tube accessory and the other special cassettes for a variety of applications.

The Trilux allows the user to use any kind of label (beta, gamma, luminescence) quantitatively on filters, microtitre plates or in tubes. So many assays now use microtitre plates which save reagents, sample, cocktail and isotope avoiding the need to transfer to a scintillation vial for counting - you can count the plate directly in the Trilux. Reducing your assay costs, saving you time, and minimising waste.

Solid scintillators further reduce costs in waste disposal and even microtitre plates with the scintillator incorporated in the plastic walls of the plate (Scintistrip) are available from Wallac.

Wallac provide a complete list of application notes for most assays and the list continues to grow. Please ask Sci Tech for further information on how the Trilux can improve your laboratory's counting efficiency.

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HEWLETT PACKARD AND MEDTEC PRODUCTS LTD INTRODUCE A MODULAR HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) SYSTEM

Medtec Products Ltd and Hewlett Packard have introduced a modular high-performance liquid-chromatography (HPLC) system that combines advanced validation- and certification-support capabilities with innovative design, manufacturing and support features. The result is an HPLC that enables laboratories to comply more easily with regulatory standards while reducing their ownership costs.

The new HP 1100 Series HPLC systems and modules are designed to meet the needs of the highly trained research chemist as well as the less-skilled technician. The product family consists of an isocratic pump, a binary pump, a quaternary pump, a vacuum degasser, a manual injector, an autosampler, a thermostatted column compartment, a variable-wavelength detector, a diode-array detector, a control module and an HP ChemStation for HPLC.

Decreased operating costs, increased reliability, easy-of-use, and automated functionality make HP's next-generation HPLC well-suited for industrial laboratories that use non-chemists to perform routine testing and QA/QC analyses. For example, in a typical QA/QC laboratory in which the HP 1100 Series HPLC system is controlled using a hand-held unit, the laboratory manager or trained technician can develop a method and store it on an insertable PC card. A less-skilled operator then can plug the PC card into the instrument to set up the method automatically. In addition to virtually eliminating errors, this approach makes it easy for laboratories to standardise HPLC analyses. For example, an international pharmaceutical company can distribute validated methods and procedures to geographically distant laboratories by e-mail, thereby providing consistent, error-free method loading every time.

Laboratories that perform more sophisticated analyses also will appreciate the improved performance of the HP 1100 Series. The new systems have the following added functionality:

- a new binary high-pressure gradient pump that yields excellent performance even at very low flow rates;
- an on-line vacuum degasser that enables cost-effective and highly efficient solvent degassing;
- a new autosampler (with sample-preparation features) that

With the new HP 1100 Series HPLC, now your lab can do *more* with *less*.



You can comply *more* easily with regulatory standards, spend *less* on cost of ownership.

The new HP 1100 Series HPLC is designed to help you meet a major laboratory challenge: tougher regulatory and quality standards, combined with ever-tighter budgets.

These systems ship from the factory with an extensive array of built-in validation capabilities. And, through innovative design, maintenance and support features, the HP

1100 Series can substantially lower your cost of ownership.

To help your laboratory do more with less, the HP 1100 Series comes with numerous features designed to enhance uptime. It incorporates field-proven components; software for on-site and remote diagnostics; multimedia maintenance procedures; and an exceptionally easy-to-repair design.

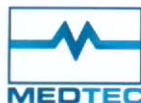
To boost productivity, the HP 1100 Series offers an intuitive PC graphical user interface and a new hand-held controller, for easy method

entry—plus a whole range of automation features.

For extensive regulatory support, enhanced productivity, new levels of performance and functionality, access to HP's top-rated support services—and low ownership costs, choose the new HP 1100 Series.



**HEWLETT®
PACKARD**



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NEW PRODUCTS

- provides improved precision;
- a new thermostatted column compartment that allows operation at temperatures from 10 °C below ambient up to 80 °C;
- a variable-wavelength detector with new optical design that provides improved baseline stability and signal/noise; and
- a new diode-array detector that provides extended wavelength detection and improved signal-to-noise ratio; from 190 nm to 950 nm.

In addition to being able to support methods and applications that currently run on the HP 1050 and HP 1090, the HP 1100 Series has been optimized to handle a variety of new applications. HP will publish these applications in application notes and solution guides that specify exact analysis conditions, performance data, and chromatograms.

Advanced Validation Capabilities Speed System and Method Verification:

Analytical laboratories serving the pharmaceutical, biopharmaceutical, environmental, food and chemical industries will find that the HP 1100 Series can help generate validated results quickly and economically. In addition to offering the extensive set of field-testing validation tools embodied in HP's previous HPLC systems, the HP 1100 Series offers a number of new capabilities including the following:

- software for operational qualification and performance verification (OQ/PV) performs complete system check-out in less than four hours;
- SOPs in electronic format can be adopted directly or customised by users;
- an improved electronic logbook tracks instrument performance, maintenance activities and unusual events or errors;
- improved software for automated method validation demonstrates method integrity;
- improved suitability-testing software verifies the performance qualification (PQ) of the system;
- control charts of critical performance characteristics let users know if instrument and method performance is being maintained; and
- instrument serial numbers, instrument logbooks, instrument conditions and column parameters are stored with raw data for traceability and data integrity in binary files.

Design Innovations Lower Ownership Costs

In addition to leveraging existing, proven HPLC technology, HP took advantage of a number of design innovations to make the HP 1100 Series systems considerably less expensive to purchase, operate and maintain. By adopting a chassis and housing design called E-PAC - which makes use of fewer highly integrated parts and more environmentally sensitive packaging materials - HP has been able to reduce the base price of the HP 1100 Series modules by as much as 20 percent.

Further cost reductions are achieved through faster and easier start-up. There are no complicated gas connections, and only one cable is required to interconnect the modules. Because

graphical step-by-step instructions are provided on paper and on multimedia CD-ROM, customers can install systems on their own.

The HP 1100 Series is controlled either by an HP ChemStation with an intuitive graphical user interface (GUI) or a hand-held system control module that is based on HP's unique 200LX palmtop PC technology. The HP ChemStation provides instrument control, data acquisition, and data evaluation capabilities for multiple techniques. Therefore, anyone who already is familiar with an HP ChemStation for gas chromatography (GC) or capillary electrophoresis (CE) will be able to use the HPLC ChemStation with a minimum of additional training. On-line tutorials guide the user through activities such as method and sequence setup. The single hand-held controller can be used to control a complete system. The operator can set method and sequence parameters using a miniaturized keypad and icon-based LED display. Extensive on-line, context-specific information makes operation easy.

An all-electric design eliminates the need for expensive helium degassing or circulating fluids to cool the column oven. Pumps work with 2 mm and 1 mm id columns at low flow rates to reduce mobile phase usage. The system can be programmed to enter "sleep" mode when not in use, thereby saving energy and extending the life of the components.

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EDWARDS BIOLINE INCUBATOR SHAKERS

Designed and manufactured in Australia, these benchtop shaker incubators offer users reliability and quality only found previously in much more expensive brands.

The shaking mechanics employ a three-point drive system utilizing twelve permanently lubricated bearings with the addition of an electronic speed management circuit for smooth start and constant speed operation, irrespective of changes in line voltage or work load.

Flask capacity is up to 64 x 50 mL or 5 x 2000 mL flasks. Options include cooling, flask clips and utility trays.

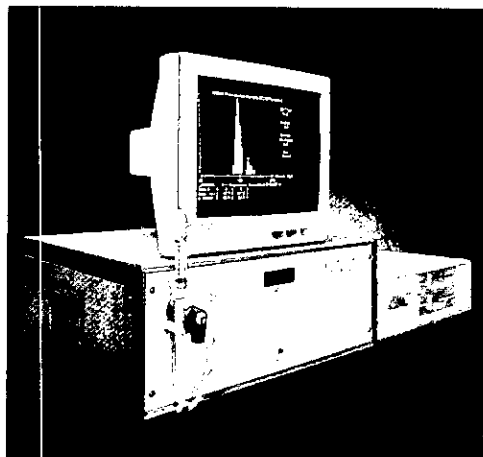
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AUTOMATIC HIGH RESOLUTION PARTICLE SIZE ANALYSIS

Particle Sizing Systems offer optical sensing single-particle sizers to suit most research or quality control applications. The Nicomp Accusizer Model 770 has a fast pulse counter, fast direct-memory access and fast multi-channel analyser to handle up to 20,000 particles per second. SPOS (single-particle optical

NEW PRODUCTS

sensing) is the method used which enables particles in a liquid or gas suspension to be sized individually as they pass through a narrow "photozone" of uniform illumination. Particles entering the photozone are detected using either the technique of light scattering (for particles smaller than approximately 20 microns) or light blockage (for diameters larger than about one micron). In either case a signal pulse results, with a height which increases monotonically with particle diameter. The system computer constructs the true particle size distribution at high speed by converting the pulse heights to diameters using a standard calibration curve.



There is a choice of sensors providing particle size ranges from 0.5 to 2500 microns. The automatic diluter option allows continuous exponential dilution of concentrated particle suspensions and has both variable-volume dilution chamber and variable-speed electromagnetic stirrer. The system is PC controlled (386/486) and the software provides real-time display of size distribution during analysis.

The Nicomp Submicron Model 370 provides the user with a particle size range from 0.002 to 5 microns. The Nicomp Accusizer Model 377 is a combined system providing the widest possible particle sizer on the market today with a range of 0.005 to 2500 microns.

Accessories include autosamplers, high-power and very high-power laser options, and multi-angle options.

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NEW UV/VISIBLE SPECTROPHOTOMETERS FROM JASCO

Jasco have introduced a number of new models of UV/Visible spectrophotometer.

The Model V-530 double beam spectrophotometer is a general-purpose instrument. The software that controls the unit facilitates simplicity and ease-of-operation while maintaining high performance. The size of the optical bench is 484 (W) x

477 (D) x 205 (H) mm, which saves bench space in a crowded modern laboratory. Double beam technology and high-quality concave grating are offered. Reduction of stray light to 0.04% allows for highly precise measurement in a wide photometric range. High-volume optical throughput and high-speed response detectors allow the model to scan at speeds of up to 4000 nm/min with minimal tracking error. A full complement of hardware accessories, such as cell holders and flow cell units, and software packages such as kinetic analysis and protein-nucleic acid quantitative analysis applications are available. A validation software package is also available.

The V-530iRM and V-530PC are well suited for the needs of individual operators. The V-530iRM offers data acquisition and processing by means of an easy-to-use intelligent Remote Module (iRM) equipped with a wide back-lit graphic liquid crystal display (320 x 40 dots). The V-530iRM can be upgraded to full PC control. The V-530PC is controlled by an MS-DOS-based PC with the same fully integrated PC software package that is supplied with advanced V-series models. The software utilizes high-speed computers to perform data acquisition, manipulation, and analysis by means of simple and easy key strokes.

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NEW RFA 300 RAPID FLOW ANALYSER FROM ASTORIA PACIFIC INTERNATIONAL

The RFA 300 Rapid Flow Analyser brings the latest advances in segmented continuous flow analysis to the chemistry laboratory for non-environmental applications. By utilisation of narrow bore tubing and glass reaction coils, superior results are obtained.



Features include:

- Up to 300 samples per hour
- Automation of complicated chemistries
- Modular design allows easy expansion of the system
- Low flow volumes means lower running costs
- FASPAC software allows for automatic recalibration, eliminating the need for operator intervention
- Advanced design and manufacturing plus solid state electronics gives greater reliability and superior performance.

The result is an automated wet chemistry system of high reliability, engineered to exceptionally high standards.

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NEW PRODUCTS

AFFORDABLE HPLC AUTOMATION

Alltech have introduced the 570 Autosampler to automate any HPLC, protein chromatograph or ion chromatography system. Available in metal-free and cooled versions, the 570 is ideal for quality control laboratories where precision, accuracy and costs are equally important. If you are processing 10 or more samples per day without an autosampler you are wasting expensive technician time and probably accepting wider margins of error than you need to. The Alltech 570 Autosampler costs less than one third of a technician's salary and works quietly and reliably with the same accuracy every time freeing your highly trained staff for more productive tasks.



Contact: Alltech Associates Inc.

P O Box 100352 North Shore Mail Centre, Auckland

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ARE YOU UP TO TEMPERATURE?

YSI has remained a leader in precision temperature measurement for over 50 years. The new 4600 Series Precision Thermometers from YSI, take metrology level temperature measurement to the laboratory, to the manufacturing floor, and to the field.



Three versions are available which address a broad range of temperature measurement applications:

The Full Spectrum of Water Quality Measurement

THE NEW YSI WATER ANALYSIS SYSTEMS

The safest and easiest way to measure:

- * Chlorine
- * Nitrate
- * Iron
- * Copper
- * Hardness
- * Ammonia
- * Nitrite
- * Magnesium
- * Phosphate
- * many others



John Morris Scientific Ltd

P O Box 6348 Wellesley Street, Auckland

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Freephone: 0800 651700

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NEW PRODUCTS

** Model 4600 - Flexible and Precise*

This is a general purpose, highly accurate digital thermometer using standard YSI 400 Series Interchangeable Probes ideal for applications where system accuracy and flexibility are important. Features include:

- Wide measurement range: -40 to +150 °C
- Thermometer accuracy: ± 0.015 °C, range 0 to 50 °C
- Probe styles available: stainless steel tubular, air/gas, vinyl tip, surface temperature, 1 mm general purpose, 22 gauge needle.

** Model 4610 - Extreme Precision With Interchangeable Probes*
Offers extremely high system accuracy by combining the 4610 thermometer with YSI's unique extremely high accuracy interchangeable probes. Probes are designed and calibrated for use only with the 4610 thermometer. The 4610 is the right choice when interchangeable probes are needed for field replacement and flexibility in measurement applications. Features:

- Temperature range: 0 to +70 °C
- Extreme system accuracy: ± 0.05 °C, range +20 to +50 °C
- Interchangeable probes in three styles: 1 mm general purpose, vinyl tip general purpose, stainless steel tubular.

** Model 4600S - Secondary Temperature Standard - Customer Specified Calibration*

Calibrated for maximum accuracy over a user-defined range. To accomplish this METROLOGY LEVEL system accuracy, the user selects a single measurement point or multiple points (up to four) where extreme accuracy is required. YSI then calibrates the instrument along with the desired YSI 400 Series Probe at the required temperature to meet your requirements.

- Wide measurement range: -40 to +150 °C
- System accuracy: ± 0.025 °C at calibrated point within 0 to +50 °C range.

All models are powered by a 9 V battery and are fully portable. Typical applications range from analytical and scientific instrumentation companies for use in the field service of high precision instruments, industrial process oriented industries i.e., food processing, pharmaceutical, pulp and paper and biotechnology, metrology laboratories, research laboratories, and power generation facilities.

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Ph: (09) 3663999 Fax: (09) 3663060 Freephone: 0800 651700
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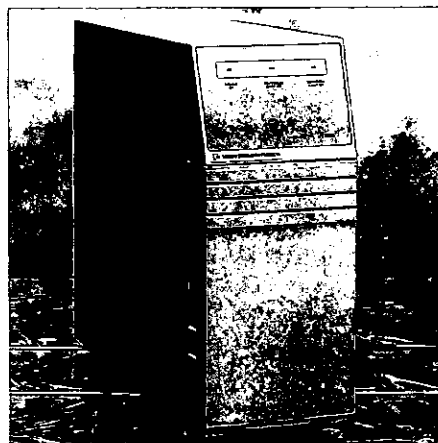
NEW Spectra SYSTEM UV 3000 SCANNING UV/VISIBLE DETECTOR

Using revolutionary Low Inertia Scanning (LIS) technology, developed by NASA, and Thermo Separation Product's patented direct optics design, this new instrument provides multidimensional capabilities similar to a diode array detector.

Features include:

- Unsurpassed optical performance
- Higher sensitivity
- Lower drift

- Improved linearity
- Virtual elimination of refractive index effects



The result is a multi-spectral UV/Visible detector which is less expensive and provides better performance than diode array detectors.

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NEW SEPTUM VALVE FROM SGE

SGE's new VLLMA5/2000 Septum Valve is the ideal solution for processing 5 mL to 2000 mL volumes of gases for analysis. The valve gives easy access to the sample while the septum prevents any losses or contamination. Easily spike or add internal standard to 5 and 25 mL Purge and Trap Samples or to a 2000 mL Jumbo Syringe, close the valve and swap the septum for a removable needle for injection. SGE's New Septum Valve part number 031911.

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FOOD APPLICATIONS FOR CAPILLARY GC

SGE capillary column recommendations for food applications are a useful guide for the food chemist and are detailed in a single page quick reference sheet including chromatogram references and part numbers available from Alltech, the chromatography specialists.

Several applications specify the new SGE BPX columns with silphenylene chemistry. These revolutionary columns for high temperature work will be available from 1 March 1995 for a limited time at 1/2 price, only from Alltech.

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NEW PRODUCTS

SGE μ -FLOW HPLC PUMP/SAMPLE SPLITTER

Save money and increase sensitivity

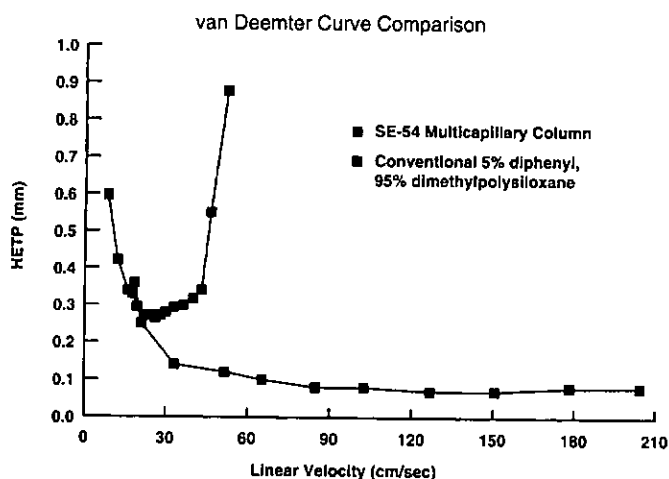
SGE has used new flow split technology in the design of the μ -flow HPLC splitter (part number 206360). This device is designed to allow a conventional HPLC piston pump to deliver micro volume flows suitable for 0.3 mm, 0.5 mm, 1 mm and 2 mm id HPLC columns. Waste solvent can then be recycled. The unit also allows accurate and reproducible splitting of conventional injection volumes to volumes suitable for micro columns. Request data sheet PD-0096-H.

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A MULTICAPILLARY IS MORE EFFICIENT

High flow rates dramatically reduce run times on Alltech's new MULTICAPILLARY columns, without sacrificing efficiency or resolution. A plot of height equivalent to a theoretical plate (HETP) at various flow rates, (below) shows how standard capillaries recede from the optimum (minimum) HETP value very quickly while MULTICAPILLARY offers a wide (30 to 210 cm/s⁻¹) flow rate window at high efficiency. MULTICAPILLARY columns provide short retention times with a high degree of separation and excellent resolution. Temperature programming is rarely necessary with MULTICAPILLARY columns because high flow rates elute strongly retained compounds under isothermal conditions.

A comparison of an SE-54 MULTICAPILLARY van Deemter curve performed at 95 °C versus a standard 5% diphenyl, 95% dimethylpolysiloxane column performed at 130 °C.

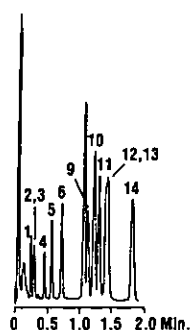


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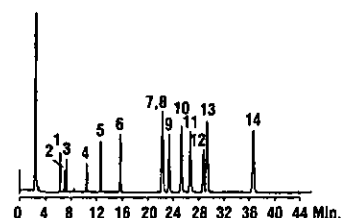
MULTICAPILLARY IMPROVES PESTICIDE SCREENING DRAMATICALLY

On a standard capillary column, a typical chlorinated pesticide analysis takes approximately 37 minutes. Using a MULTICAPILLARY column, the same analysis at the same temperature takes just 2 minutes, without sacrificing analytical results. Sample throughput jumps 6.5x from 4.6 to 30 samples per hour. Improve your productivity with Alltech's MULTICAPILLARY column currently available in SE-30, SE-54 and Carbowax 20M chemistries.

Pesticide Mixture on MULTICAPILLARY



Pesticide Mixture on Standard Capillary



Peak ID:	1. α -BHC	7. p,p'-DDE
	2. Lindane	8. Dieldrin
	3. β -BHC	9. o,p'-DDD
	4. Heptachlor	10. Endrin
	5. Aldrin	11. o,p'-DDT
	6. Heptachlor epoxide	12. p,p'-DDD
		13. β -Endosulfan
		14. p,p'-DDT

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LONG LIFE HPLC COLUMNS

Alltech's ALLTIMA HPLC columns are longer lived because the bonded phase is polymerically attached to the base silica. Polymeric bonding provides column stability when exposed to harsh mobile phases e.g. extremes of pH. The chromatograms below show the excellent stability of an ALLTIMA column. ALLTIMA C18 is available in two different carbon loads to give you a choice of selectivity in a longer life column. The ALLTIMA C18-LL (Low Load) material offers significantly shorter retention times for faster separation of later eluting compounds.

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NEW PRODUCTS

SHELL VIALS FOR WATERS AUTOSAMPLERS

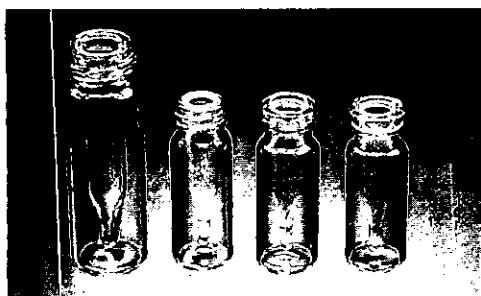
Alltech Polypropylene Limited Volume Shell Vials are the one vial alternative to expensive and tricky-to-assemble glass vials with springs and inserts. Alltech Polypropylene Limited Volume Shell Vials eliminate the most frequent cause of bent needles in Waters autosamplers - trapping of the needle between the insert and the vial. Colorful caps simplify sample identification too. Alltech Polypropylene Limited Volume Shell Vials for both 48- and 96-place Waters' carousels are available.



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NO MORE BENT NEEDLES

Alltech's New Step Vial System has self-centering inserts with a wider opening. The larger id prevents missed injections and bent needles. Automatic alignment of the flanged insert assures maximum sample withdrawal. There are no springs to worry about as the Alltech Step Vial System fully supports the insert. Step Vial is compatible with robotic autosamplers and available in clear, amber, screw top, solid crimp and snap-ring crimp top 12 x 32 mm and 15 x 45 mm vials from Alltech.

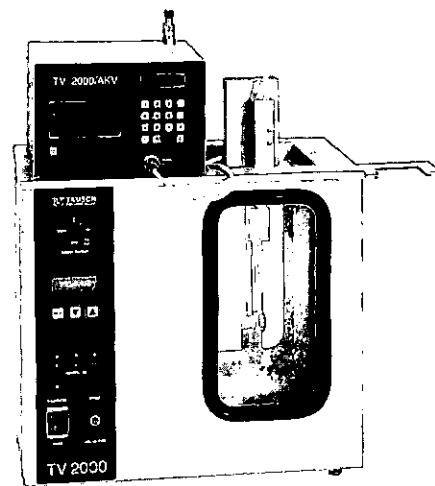


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SEMI-AUTOMATED VISCOSITY BATH

A.i. Scientific releases the new TV2000/AKV Viscometer Bath from Tamson. It utilises a unique opto-electronic sensing technique to measure the viscosity of light and dark fluids in the 0.3 to 10,000 cSt range. The microprocessor control unit will calculate and print the (average) value in cSt of up to 9 repeat tests and store up to 25 tube constants. Insulated stainless steel construction ensures a temperature stability and uniformity

of ± 0.01 K. Compliant with ASTM D445 and IP71, the TV2000/AKV features auto-calibration upon start-up as well as automatic safety shut-downs with error reporting. A.i. Scientific is providing support for all Tamson Viscometer Baths.



Contact: Kevin Moloney, A.i. Scientific
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SHIMADZU NOW REPRESENT O I ANALYTICAL

O I Analytical, leading manufacturers of purge and trap, head space samplers and specialised gas chromatography (GC) detectors - is now represented in New Zealand by Shimadzu. The O I Analytical range complements Shimadzu's state-of-the-art range of gas chromatographs.

Sample Introduction

- Purge & Trap Systems
- Head Space Samplers

Specialised GC Detectors

- Photoionisation (PID), Halogen Specific (XSD)
- Electrolytic Conductivity (ELCD)
- Dual PID/ELCD and PID/FID

Sample Preparation

- Analytical Microwave Systems
- Liquid/ Liquid and Liquid/Solid Extraction Systems
- Gel Permeation Chromatography Clean-up

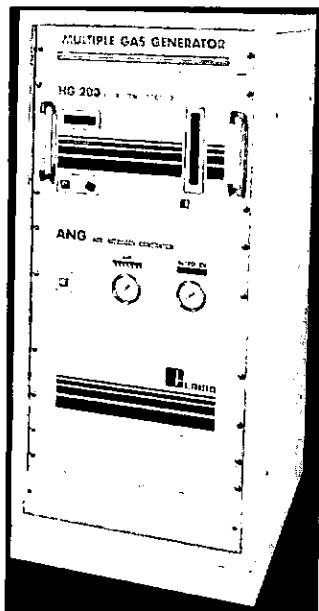
Contact: Shimadzu New Zealand
P O Box 47027 Auckland 8
Ph: 0800 735 725, Fax: (09) 836 0668
circle number 40 on the reader reply card

HYDROGEN AND PURE AIR GENERATOR FOR GC-FIDs

Now available is a single gas generation unit providing ultra-high purity hydrogen and pure air that improves FID sensitivity and baseline stability. Developed specifically for gas chromatography as a viable alternative to multiple gas cylinders.

NEW PRODUCTS

Optional flow rates range from 185 mL/min to 600 mL/min for hydrogen and pure air flow rates of 1000 mL/min to 2500 mL/min. The small footprint unit takes up less space than two gas cylinders. The unit is safe to use in a laboratory and provides a continual supply of high purity gases at the correct pressure at the point of use. Units are also available with optional nitrogen generator suitable for use with GC-ECDs.



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ON-LINE VOCs IN AIR

Perkin-Elmer has developed, in collaboration with the USEPA Atmospheric Research and Exposure Assessment Laboratory, a robust, on-line system for monitoring of VOCs in air. It is applicable to site emission measurements, urban air quality testing and workplace air monitoring.

This system, which is based upon the Model ATD 400 thermal desorber, completely eliminates liquid cryogen from the determination of non-methane organic air pollutants.

An electrically cooled adsorbent trap is used to focus analytes to capillary GC analysis, while sophisticated dual or single column capillary chromatography provides optimum resolution of complex target analyte mixtures without subambient GC oven cooling.

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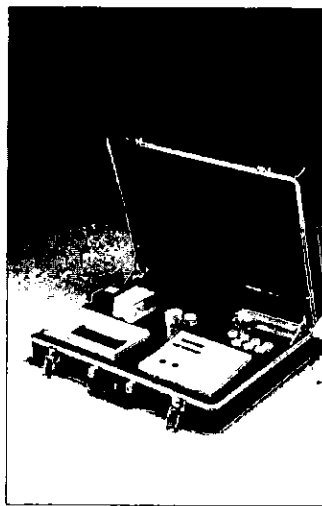
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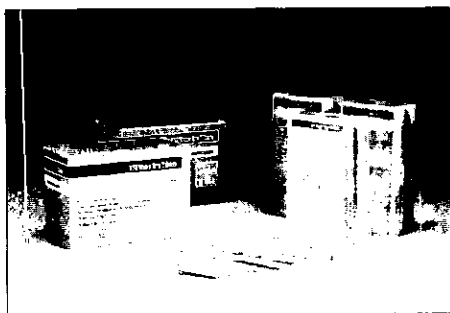
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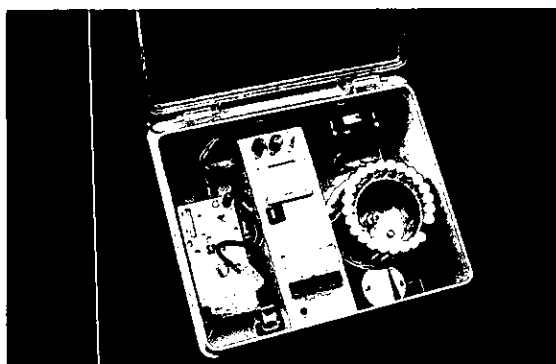
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Maintaining Quality In An Environmental Testing Laboratory

Peter Robinson PhD FNZIC
Environmental Division Manager
R J Hill Laboratories Ltd, P O Box 4048 Hamilton

Samples may be collected from environmental sites for a variety of reasons. Testing may need to be carried out for Resource Consent applications, compliance monitoring, pre-purchase reports, spill or pollution responses or for site cleanup purposes.

Because of the variety of sites which may be involved, any laboratory which chemically tests samples for clients working in the environmental area is likely to be presented with samples from a wide range of matrices. These may be relatively clean, as in drinking water or from some groundwater bores, through moderately dirty, such as stormwater run-offs and tip leachates, to pure effluents, waste waters from industry and sewage. They may also contain varying levels of salinity, high hardness, low pH or considerable concentrations of toxic species such as cyanide, sulphide, arsenic or organics such as PCP, PAHs, PCBs or pesticides.

Chemical testing laboratories maintain quality by analysing "blanks", quality control samples, spiked samples, replicates and Certified Reference Materials as appropriate to the test being carried out. All of these are useful for confirming the validity of the testing procedures, but, usually, only by analysing spiked samples can the contribution of any matrix interferences be determined. The experience of the technicians and analysts, as well as a thorough knowledge of the testing method and instrumentation, can also be valuable in recognising samples likely to present problems, and it is essential that the laboratory have alternative methods available to deal with any samples which have matrix interferences affecting determination of the analyte in question.

No one method is suitable for analysing all samples for a particular analyte, and it is very useful for the laboratory to have some background information about the samples, such as type of site, expected levels of components, i.e. high or low, and any specific details such as sewage (microbiological hazard) or gold mining effluent (possible high cyanide).

Some examples of matrix interferences and methods used at R J Hill Laboratories Ltd to deal with them so as to supply reliable data to clients are given below.

Organonitrogen pesticide analysis

Following extraction and pre-concentration, samples are analysed using a Gas Chromatograph (GC) fitted with two columns, one to an Electron Capture Detector (ECD), the other to a Nitrogen-Phosphorus Detector (NPD). Naturally occurring nitrogenous compounds may give a response on the NPD which can represent a false positive for a pesticide. Many of the pesticides also give a signal on the ECD which, because it has

a column of different polarity connected, can be used for confirmation. Further confirmation can be obtained by analysing the sample using a Gas Chromatograph-Mass Spectral Detector (GC-MSD). Analysis for organochlorine pesticides and polycyclic biphenyls (PCBs) using a GC-ECD combination is also prone to false positives, and we have found it essential to have the GC-MSD for use in checking these results.

Low Level Toxic Metals

The sample is usually digested or extracted using nitric acid and then analysed by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). As ICP-MS cannot cope with high dissolved salts, we routinely screen all samples for conductivity before analysis. This will detect samples containing very saline matrices for example, and these are then analysed by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS).

There are many polyatomic interferences in ICP-MS, for example Ar-C on chromium, Ar-Cl on arsenic, Ca-O on nickel. It is essential that these interferences are recognised and other techniques used to analyse the samples if necessary so that reliable results are provided. We use GFAAS or Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) for this purpose.

Summary

In order for an analytical chemistry laboratory to provide reliable results on the wide range of matrices encountered with environmental samples, the laboratory must have a range of instrumental techniques available along with the technical expertise to recognise and deal with problem matrices as they arise.

R J Hill Laboratories Ltd has invested heavily in instrumentation in the last few years and is now perhaps the best equipped environmental testing laboratory in New Zealand, having ICP-MS, ICP-OES, GFAAS, GC-MS, GC-ECD/NPD, and a variety of other instrumentation available. We also have a commitment to employing well qualified staff who are given appropriate training in these techniques. Taken together, this allows us to provide our clients with reliable results at a competitive price, and with an acceptable turnaround time.

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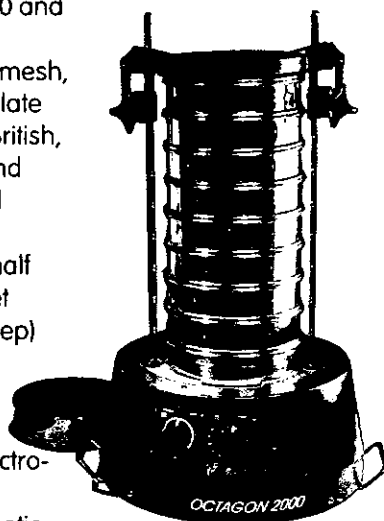
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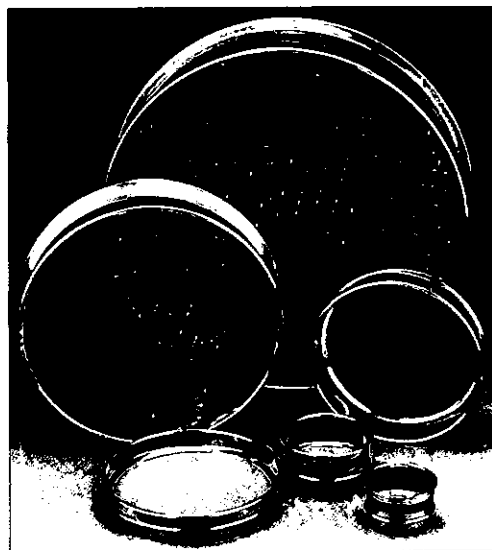
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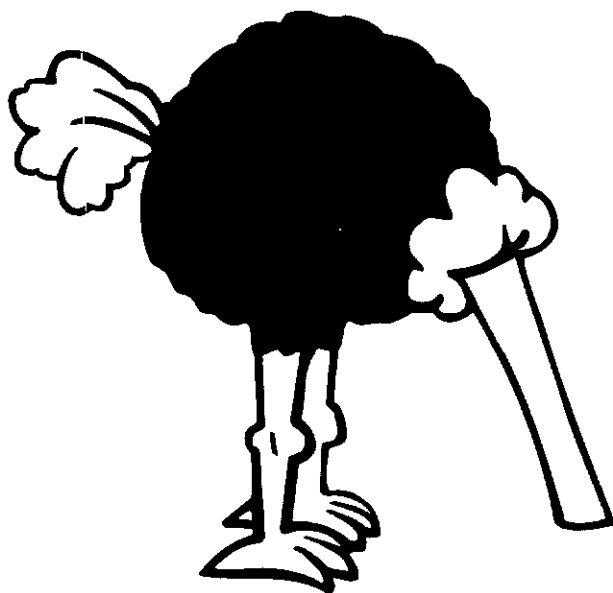
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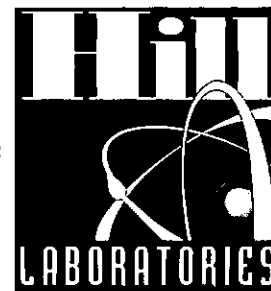


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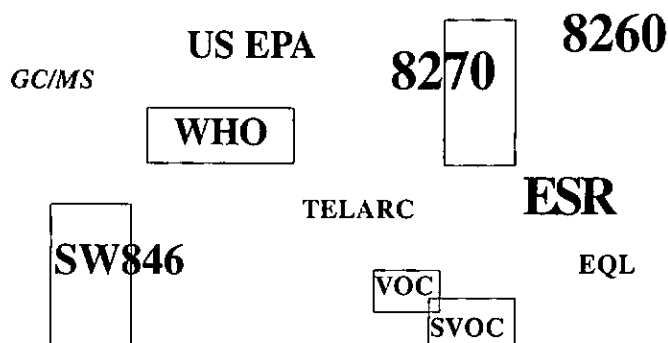
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The Language of Modern Environmental Science?

Bob Symons and Stuart Glen

ESR: Environmental, P O Box 30-547, Lower Hutt



Introduction

Communication in today's world is built around jargon and interpreting a plethora of acronyms can be a very daunting task. At the Institute of Environmental Science & Research (ESR) we can assist in easing this process as it applies to environmental chemistry by explaining some of the associated nuances. We currently hold TELARC (Testing Laboratory Registration Council of New Zealand) registration and WHO (World Health Organisation) accreditation for the analysis of an extensive array of environmental pollutants using techniques based upon United States Environmental Protection Agency (USEPA) methodologies. These methods are largely taken from "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods" Nov 1986; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington DC, 20460. SW-846, 3rd Edition: Proposed Update I, II and IIA". September 1994 or more commonly known as SW846.

Two of the current topical and therefore frequently requested assays are USEPA Methods 8270 "Semi-volatile Organic Compounds (SVOC) By Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique" and Method 8260 "Volatile Organic Compounds (VOC) By Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique."

What exactly are Methods 8270 and 8260 and what do they offer?

There is a perceived confusion among or by many in the environmental arena about the application of USEPA Methods 8260 and 8270 to particular environmental problems. The following offers a brief outline of both of these methods as detailed in SW846.

Method 8260 (VOC) relies upon the use of Method 5030 for the sample preparation and extraction of volatile organics by a purge-and-trap procedure. Method 5030 can be used for most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits are

approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a capillary column. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. Water samples can be analysed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system. This method also describes the preparation of water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

The estimated quantitation limit (EQL) of Method 8260 for an individual compound is approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water. EQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

Method 8270 (SVOC) has only recently been offered by laboratories in New Zealand. There are 262 semi-volatile priority pollutants which are able to be determined using the techniques described in Method 8270. This does not mean, however, that all of these compounds require analysis in any one sample. The list of compounds offered will vary from laboratory to laboratory, and will often be defined by the matrix or the potential contamination. ESR is able to offer a large proportion of these compounds due to the high level of staff expertise and instrumentation.

Method 8270 is primarily used to screen a large number of potential contaminants, and report the approximate concentrations of each contaminant detected. The cost of analysis varies greatly due to the scope of SVOCs offered, and the technique used for quantification. **Method 8270 can only be performed by GC/MS.** Using GC/MS technology, non-target as well as target compounds can be determined. This is not possible with the more traditional GC/ECD type technology. Due to the increasingly complex nature of the components, straight GC analysis provides a low level of acceptability in determining individual compounds. By using GC alone it is necessary to make assumptions that the peak being measured is actually the desired compound. GC/MS on the other hand is able to provide mass spectral data for each component, and therefore provides a much greater degree of certainty. There is no guarantee of a correct answer without GC/MS analysis.

Analysis of samples for semi-volatiles requires the use of suitable extraction procedures followed by appropriate clean-up techniques. Water, soil/sediment, sludge, and waste samples requiring analysis for base/neutral and acid extractables and/or organochlorine pesticides must undergo solvent extraction prior to analysis. The SW-846 manual contains five methods that may be used for this purpose: Method 3510; Method 3520;

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Method 3540; Method 3550 and more recently Method 3545. The method that should be used on a particular sample is highly dependent upon the physical characteristics of that sample. Therefore, review of these five methods must be done prior to choosing one in particular. Appropriate surrogate standards and, if necessary, matrix spiking solutions are added to the sample prior to extraction for all five methods.

Method 3510: Separatory Funnel Liquid-Liquid Extraction

Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. Method 3520 should be used if an emulsion forms between the solvent-sample phases, which can not be broken up by mechanical techniques.

Method 3520: Continuous Liquid-Liquid Extraction

Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. The limitations of Method 3510 concerning solvent-sample phase separation are avoided with this procedure.

Method 3540: Soxhlet Extraction

This is a procedure for extracting non-volatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes.

Method 3550: Sonication Extraction

This method is applicable to the extraction of non-volatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes using the technique of sonication. Two procedures are detailed depending upon the expected concentration of organics in the sample.

Method 3545: Accelerated Solvent Extraction (ASE)

This method is applicable to the extraction of non-volatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The method uses elevated temperature (100 °C) and pressure (1500-2000 psi) to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure. This method is applicable to solid samples only, and is most effective on dry materials with small particle size.

Direct injection of extracts from any of the above extraction techniques into a gas chromatograph can cause extraneous peaks, deterioration of peak resolution and column efficiency, loss of detector sensitivity, and can greatly shorten the lifetime of expensive columns. The following techniques have been applied to extract purification: partitioning between immiscible solvents; adsorption chromatography; gel permeation chromatography; chemical destruction of interfering substances with acid, alkali, or oxidising agents; and distillation. These techniques are used individually or in various combinations, depending on the extent and nature of the co-extractives.

It is an unusual situation, e.g., with some water samples, when extracts can be directly determined without further treatment. Soil and waste extracts often require a combination of clean-up methods. For example, when analysing for organochlorine pesticides and PCBs, it may be necessary to use gel permeation chromatography (GPC), to eliminate the high boiling material and a micro alumina or Florisil column to eliminate interferences with the analyte peaks.

The estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 1 mg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples. EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

The USEPA generally requires using approved methods for sampling and analysis operations fulfilling regulatory requirements, but the mere approval of these methods does not guarantee adequate results. Inaccuracies can result from many causes, including unanticipated matrix effects, equipment malfunctions, and operator error. Therefore, the quality control component of each method is indispensable.

At ESR the data acquired from quality control procedures are used to estimate and evaluate the information content of analytical data and to determine the necessity or the effect of corrective action procedures. The means used to estimate information content include precision, accuracy, detection limit, and other quantifiable and qualitative indicators.

Summary

Method 8260 and 8270 are screening methods for a large range of potential contaminants, and are well suited to such applications as contaminated site investigation, leachate, groundwater and drinking water.

With the utilisation of GC/MS technology a much wider range of contaminants can be efficiently and cost effectively determined. GC/MS technology often allows non-target compounds to be determined.

GC/MS technology offers advantages over more traditional straight GC techniques as it provides a far greater degree of certainty by providing mass spectral data for each component rather than (often) the single peak obtained by traditional GC alone. GC/MS may be more expensive, but the quality of data generated far outweighs the cost of analysis.

The range of contaminants analysed varies greatly from laboratory to laboratory due to the number of contaminants which are able to be determined by an individual laboratory, the technology used or available, the sample matrix, and the type and scope of the investigation.

Method 8260 and 8270 have associated sample preparation methodologies under SW846 which form an integral part of the precision and accuracy of the final result.

References

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods" Nov 1986; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington DC, 20460. SW-846, 3rd Edition: Proposed Update I, II and IIa". September 1994.

* * * * *

CADMIUM ACCUMULATION IN NEW ZEALAND PASTURE SOILS

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Introduction

The toxicity of cadmium (Cd) to both human beings and animals has been known for many years. Humans ingest Cd from foods and inhale it from tobacco smoke and other industrial emissions. The Cd concentrations in food products reflect primarily the amounts of Cd taken up by plants from soil and the subsequent intake by grazing animals. Recently there has been growing concern about the accumulation of Cd in New Zealand pasture soils due to the continuous use of phosphate fertilizers in which Cd occurs as a contaminant (Bramley, 1990; Roberts *et al.*, 1994).

Health authorities in many parts of the world are becoming more concerned about the effects of Cd on human health. The Cd accumulating in the offal (mainly kidney and liver) of grazing animals not only makes it unsuitable for consumption but also threatens the access of New Zealand offal products to overseas markets. There is an urgency in every country to ensure that the Cd content of food stuffs produced generally complies with standards and compares well with other countries. Effective action in the long-term will depend on gaining an understanding of the causes of the accumulations and a proper appreciation of the issues for public health.

Entry of fertilizer-derived Cd into the food chain depends on the amount of Cd input through fertilizer addition, the properties of the fertilizer and the soil (especially soil pH), the rates of uptake by plant species and the rate of absorption by the grazing animals. This article focuses on the extent of Cd accumulation in New Zealand soils due to past fertilizer use practices and the possible methods to minimise the uptake of Cd by the pasture species.

Cadmium in nature

The concentration of Cd in igneous rocks, sandstones and limestones is generally low (<0.2 mg Cd kg⁻¹). Although the concentration of Cd in phosphorites and rocks derived from lacustrine sediments and marine black shales is considered to be high (11-25 mg Cd kg⁻¹) (Peterson and Alloway 1979), none of these three occur to any great extent in New Zealand.

In nature, Cd occurs mainly in association with zinc ores, of which sphalerite (zinc sulfide) forms the main commercial source of Cd. Prior to the development of industry, Cd entered agriculture systems at a slow rate as a result of the weathering of rocks and volcanic activity. Now Cd is released into the atmosphere through various industrial process which include: smelting of metals, combustion of coal, oil and wood, and incineration of wastes. The use of phosphate fertilizers and the disposal of sewage sludges directly add Cd to soil.

Cadmium in phosphate fertilizers

The Cd content of some of the most commonly used phosphate fertilizers is presented in Table 1. The Cd in most fertilizers originates mainly from the phosphate rocks used for manufacturing phosphate fertilizers (Williams and David, 1973). The Cd in phosphate rocks is concentrated in the first instance through biogenic processes, particularly by the action of microorganisms, under marine conditions. Cadmium subsequently substitutes for Ca which has similar ionic size in the apatite lattice.

Table 1. Phosphorus (P) and cadmium (Cd) contents of various phosphate sources and the number of years required to exceed the threshold concentration of cadmium (3mg Cd kg⁻¹) in soils due to fertilizer application.

Phosphate fertilizer	P content (gkg ⁻¹)	Cd content (mgkg ⁻¹)	Years required to exceed the threshold limit **
Single superphosphate	98	32	166
Triple superphosphate	190	70	152
Diammonium phosphate	200	10	1125
Reactive Phosphate Rocks			
North Carolina	132	54	135
Sechura	131	12	614
Egyptian	130	10	732
Gafsa	134	70	107

** At an annual fertilizer application rate of 40 kg P per hectare

The Cd in superphosphate is water soluble and the concentration in the water extracts of superphosphate is proportional to the Ca extracted indicating that Cd is associated with the Ca in the phosphate, presumably mainly as Cd(H₂PO₄)₂ and CdSO₄. High analysis phosphate fertilizers, such as triple superphosphate, partially acidulated phosphate rocks and ammonium phosphates which are manufactured using phosphoric acids generally contain lower Cd content relative to P, reflecting the losses of Cd during the manufacture of phosphoric acids.

Cadmium in soils

In soils Cd occurs in various forms which include: free ions in solution, soluble and insoluble inorganic and organometallic complexes, ions absorbed onto iron, aluminium and manganese hydrous oxides, precipitates such as sulfides, phosphates and carbonates, and minerals, primarily biotite and riebeckite (Peterson and Alloway, 1979).

The important factors which affect the activity of Cd in soils and its availability for plant uptake include soil pH, the amount of Cd present, the metal sorption capacity of the soils, the presence of other micro elements (e.g. Zn) and macro elements (e.g. P), soil temperature, moisture and aeration (Chaney and Hornick, 1977). The affinity of Cd for soil surfaces is dependent on the pH and the type of surfaces, with the affinity of Cd increasing with pH and decreasing with concentration of Cd added.

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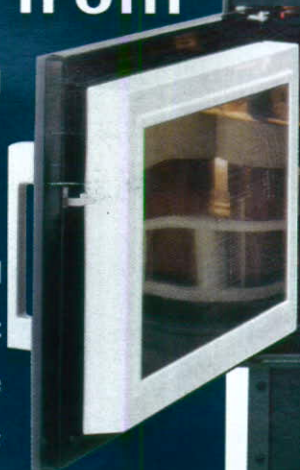
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Comparison between virgin or native (i.e. unfertilized) and agricultural soil has often been used to indicate contamination of soil through agricultural practices. Roberts *et al.*, (1994) conducted a survey of 398 sites through out New Zealand with 312 farms sites and 86 native sites to a depth of 75 mm (Table 2). They obtained evidence for the enrichment of Cd in pastoral soils and there was a highly significant correlation between total soil P and total Cd across all sites which implicates the use of P fertiliser in soil Cd enrichment. Similar results were also obtained for a range of Australian soils (Table 2). This is not surprising considering the long history of superphosphate use, manufactured from Nauru Island phosphate rock, in both New Zealand and Australia. Nauru superphosphates typically contained 34-69 mg Cd kg⁻¹ (Rothbaum *et al.*, 1986).

Table 2. Cadmium content in unfertilized and fertilized soil samples of various soil groups in Australia and New Zealand (Roberts *et al.*, 1994)

Soil Type	Cadmium Content (mgkg ⁻¹)	
	Unfertilized	Fertilized
Australia		
Red brown earth	0.055	0.188
Red podzolic	0.024	0.085
Krasnozom	0.030	0.303
Alluvial	0.144	0.270
Podzol	0.033	0.342
New Zealand		
Alluvial	0.13	0.16
Brown grey loam	0.19	0.49
Gley	0.24	0.42
Peat	0.22	0.69
Yellow brown earth	0.16	0.22
Yellow brown loam	0.23	0.70
Yellow brown peat	0.31	0.75
Yellow grey earth	0.13	0.12

Although many countries have formulated a threshold level for Cd and other heavy metal accumulation due to the use of sewage sludge, there has been no threshold limit for heavy metal accumulation through fertilizer use. Based on the threshold level for sewage application (3 mg Cd kg⁻¹ soil), the number of years taken to exceed the threshold level in soil through various sources of phosphate fertilizer is presented in Table 1. This indicates that although fertilizer addition represents the major source of Cd input to soils, at the normal annual rate of fertilizer input (40 kg P per hectare) to pasture soils, the rate of Cd accumulation is considered to be very slow.

Cadmium uptake by plants

Although Cd is a non-essential element for both plants and animals, increasing Cd concentration in soil leads to an increase in Cd uptake. Plant species differ in their ability to extract soil Cd and in general weed species are shown to accumulate more Cd. A nationwide survey by Roberts *et al.*, (1994) indicated significantly higher Cd concentration in weeds (0.28 mg Cd kg⁻¹) than either grasses and legumes (0.06-0.1 mg Cd kg⁻¹).

They also observed that while the concentration of Cd in pasture species did not vary between the native and pastoral sites, the Cd concentration in weeds was higher in the pastoral sites than the native sites. In Australia the highest Cd concentration is obtained in cape weed (1.57 mg Cd kg⁻¹) which is a common component of pastures in Australia and New Zealand. Williams and David (1973) have shown that the concentration of Cd in legumes is several times higher (0.155 mg Cd kg⁻¹) than the associated grass (0.031 mg Cd kg⁻¹), although this difference was not apparent in the New Zealand study (Roberts *et al.*, 1994). The increased concentration of Cd in legumes may be related to the acidifying effect of legumes.

Cadmium in animals and humans

Most of the Cd accumulated in grazing animals is derived from pasture intake. Bramley (1990) has estimated that annually approximately 55 mg of Cd is ingested per sheep and approximately 275 mg of Cd by cattle through the intake of herbage. Work in New Zealand has indicated that cattle and sheep may ingest 1-10% and >30% respectively of their dry matter intake in the form of soil. In areas where soil contains high Cd, for example as a result of sewage sludge and fertilizer application, soil ingestion is expected to play a significant role in Cd uptake by the farm animals. Roberts *et al.*, (1994) have shown that even though sheep ingest 36-46 kg per year, this only contributes 5 to 8% of the total Cd intake for the lax and hard grazed flocks respectively.

Ruminants do not have a homeostatic control mechanism for regulating Cd absorption or excretion which is affected by the level of dietary Cd content. Although intestinal uptake of Cd has been estimated to account for over 90% of the total Cd absorbed, most of the Cd ingested is discarded. About 80 to 90% of the total ingested Cd is excreted in the faeces and only 0.05% excreted in the urine. Most dietary Cd is bound to metallothionein and is absorbed intact into the circulation. Over 50% of the Cd ultimately accumulates in the liver and kidney. In animals, kidney and liver Cd accounts for 50-70% of the total Cd with kidney having a higher Cd concentration than the liver. Other organs such as the pancreas, spleen, heart, brain and testis, together with muscle and fat accumulate small amounts of Cd. In blood Cd is associated with albumin-like protein which is transported to the kidney where it is filtered through the glomerulus and reabsorbed by the proximal tubules (Friberg *et al.*, 1985).

In animals, the effects of prolonged exposure to abnormal levels of Cd include testicular necrosis, placenta destruction, abortion, teratogenic malformations, renal damage, osteomalacia, immunosuppression, pulmonary oedema and emphysema. In humans, severe Cd exposure can result emphysema, bronchitis, ulceration of nasal mucosa, renal dysfunction, liver necrosis and anaemia, hypertension, skeletal deformities, prostrate and lung cancer and teratogenesis (Chowdhary and Chandra, 1987). Different countries have set guidelines on the maximum permissible levels for Cd in various meat products (Table 3). In an earlier survey of cattle, pigs and sheep in New Zealand, the mean Cd concentration in kidney cortex, liver and muscle was less than 0.4, 0.1 and 0.05 mg Cd kg⁻¹ wet weight respectively (Solly *et al.*, 1981). These values are well below both the concentration measured in other countries (Table 4) and the maximum permissible concentration stipulated by many countries (Table 3).

More recent testing of animal offal however, has indicated that some 22-28% of sheep and 14-20% of cattle between 1988-1991 had kidney Cd contents greater than the permissible level of 1 mg Cd kg⁻¹ (Roberts *et al.*, 1993). In general, older animals had higher kidney Cd contents as these animals had longer exposure to Cd in their environment and hence greater opportunity to consume and retain Cd. Feral deer and sheep isolated from human intervention also showed age related retention of Cd in kidney tissue as a result of exposure to naturally occurring Cd in the environment.

Table 3. Maximum permissible limits for cadmium in muscle, liver and kidney of sheep and cattle in different countries

Country	Maximum permissible limit (mg Cd kg ⁻¹ fresh weight)		
	Muscle	Liver	Kidney
The Netherlands	0.05	1.0	3.0
Australia	0.2	1.25	2.5
Hungary	0.5	0.5	0.5
New Zealand	1.0	1.0	1.0
Denmark	0.1	-	0.5

The average annual consumption of red meat per New Zealander (70 kg adult) is estimated to be 80 kg. Based on a median Cd content of 0.01 mg kg⁻¹ wet weight, the annual intake of Cd is expected to be 0.8 mg which is equivalent to a daily intake of 2.19 µg Cd. This is considerably less than the maximum safe level of 70 µg Cd per day (Peters, 1988) from all sources in the diet. Although offal, such as liver and kidney contains more Cd than the other meat, the small quantity of offal consumed by the New Zealand population is unlikely to contribute significantly to the total Cd intake. However in sections of the population and in pets where offal forms a larger portion of the diet, Cd intake from this source could be a major concern.

Table 4. Cadmium concentration in liver and kidney samples of cattle and sheep in various countries

Country	Animal	Cd Concentration (mg Cd kg ⁻¹ fresh weight)		Reference
		Liver	Kidney	
Australia	Cattle	0.18	0.30	Langlands <i>et al.</i> (1988)
	Sheep	0.30	0.96	
New Zealand	Cattle	0.10	0.25	Solley <i>et al.</i> (1981)
	Sheep	0.10	0.25	
Netherlands	Cattle	0.11	0.36	Vos <i>et al.</i> (1987)
USA	Cattle	0.21	0.55	Mussman (1975)

The "population critical concentration" of Cd at which 10% of the human population would exhibit signs of renal impairment is about 160 mg Cd kg⁻¹ wet weight (Friberg, 1984). From the above calculations it can be shown that it is unlikely that Cd input from meat will exceed the critical concentration in the lifetime of a New Zealander.

Reducing Cd accumulation

Accumulation of Cd in soil and its subsequent uptake by pasture plants can be minimised by the following practices:

Use of low Cd containing phosphate fertilizers: This is achieved by either selective use of phosphate rocks with low Cd or treating the phosphate rocks to remove Cd. Superphosphate fertilizer manufacturers in New Zealand and Australia are introducing voluntary controls on the Cd content of phosphate fertilizers. The fertilizer industry aims to lower the Cd content in phosphate fertilizers from the current level of 340 mg Cd kg⁻¹ P to 280 mg Cd kg⁻¹ P by the year 2000. The Cd content as determined by the rock phosphate source is the most difficult to control because supplies of rock phosphate with low Cd contents are limited and sources with higher Cd contents continue to be used in many countries for practical reasons. A number of phosphate rocks are low in Cd and these can be used for the manufacture of superphosphates. Alternatively since Cd has a low boiling point (BP=789 °C), it can be removed by calcining the phosphate rocks. Phosphoric acid used in the food industry is manufactured mostly only after the removal of Cd through calcination of the phosphate rocks. Calcination of phosphate rock may not be a practical option in the fertilizer industry because it is expensive and calcination decreases the reactivity of phosphate rocks making them unsuitable for direct application as a source of phosphorus.

Immobilization of soil Cd: Low soil pH and soils of low cation exchange capacity induce a greater uptake of soluble Cd by plants. The Cd in soils can be immobilised by increasing the soil pH through the addition of liming materials. The Ca added through liming may serve to depress Cd uptake by competing for exchange sites at the root surface.

Reduced Cd intake by animals: While the weed content of well-fertilized pasture is generally low, the elevated weed Cd concentration for pasture sites indicates that the occurrence of weed species in the sward should be discouraged. This may be achieved through appropriate grazing management practices and fertilizer policies which encourage vigorous grass and legume sward hence suppressing weed establishment and growth.

In some countries, to encourage selective grazing of weeds, pastures are sprayed with herbicides at a rate that does not kill pasture species, but increases the palatability and accessibility of the weed species to sheep at high stocking rates; this is known as spray/graze management. Since the Cd concentration in most weed species is generally higher than that in pasture species, this practice may increase the uptake by animals of both Cd and herbicides.

Sheep and cattle grazed at high stocking rates have higher Cd burdens than those more moderately stocked which is attributed mainly to more soil ingestion in the former situation. If this is the case, reducing Cd intake in grazing animals will need to focus both on minimising the Cd content of pasture and the soil ingestion of grazing animal.

Conclusions

Phosphate fertilizers form the primary source of cadmium accumulation in New Zealand pasture soils. Although there has been an increase in Cd concentration in fertilized soils over native soils, the estimated rate of accumulation of Cd through fertilizer application is slow. The accumulation of Cd is generally higher in weed species than in pasture species. Intake of Cd by grazing animals is likely to result in the accumulation of Cd in the liver and kidney and thereby renders them unsuitable for consumption. Accumulation of Cd in soils and its subsequent uptake by plants can be minimised by using phosphate fertilizers that are low in Cd and immobilizing soil Cd through liming.

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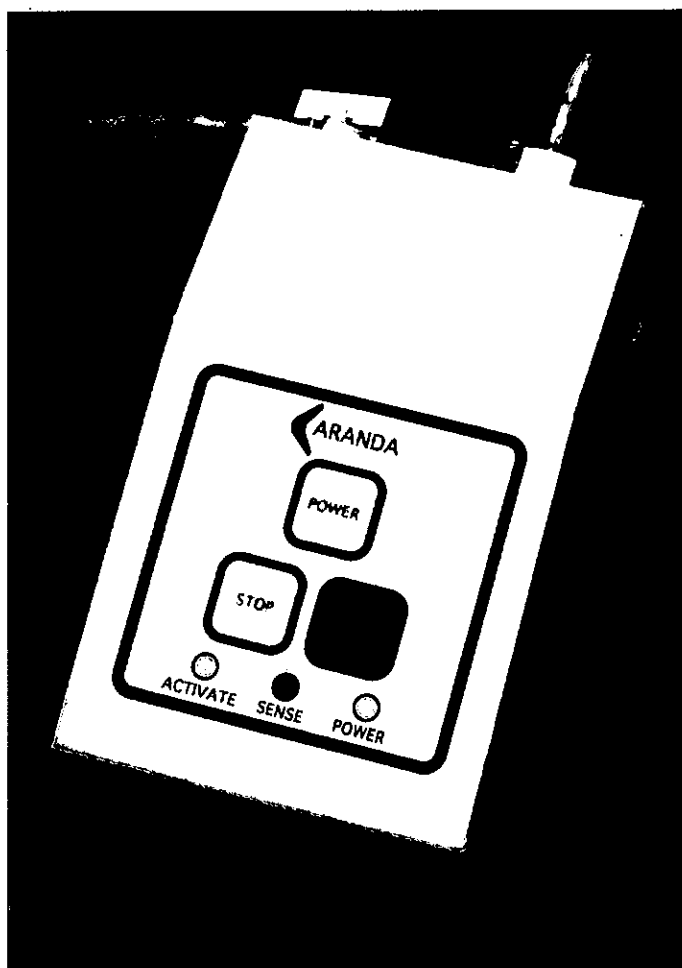
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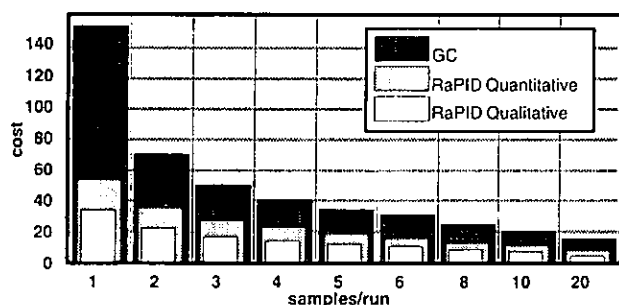
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Key Words:

- immunoassay • magnetic particles • antibodies
- pesticides • toxic organics

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Figure A. Cost Per Sample Comparison Between RaPID Assay and GC (\$/US)



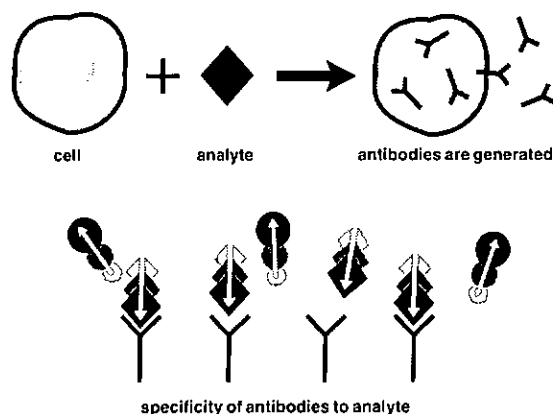
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Theory and Procedure

An immunoassay is generated using an *antibody* that very selectively recognizes a specific *antigen*. The antibodies are formed on exposure of specialized cells to a specific antigen (analyte) of interest. These cells then produce large quantities of antibodies specific for that analyte. When the antibody is exposed to a mixture of analytes, such as pesticides or toxic

organics in a soil or food extract or water sample, it will recognize and bind to only the specific analyte that induced its original formation (Figure B).

Figure B. Generation and Specificity of Antibodies



RaPID Assay uses these analyte-specific antibodies that are attached to solid support (magnetized particles) to facilitate analyte binding. Note that the antibody-antigen complex cannot be broken apart (except by extreme changes in buffer pH or ionic strength), and thus should be considered a permanent bond within the assay system. For maximum sensitivity of the immunoassay, RaPID assays are developed in a competitive format. This means that the analyte in the sample and an enzyme-labeled analyte *conjugate* compete for the antibody binding sites. A colour solution is used to react with the enzyme conjugate generating colour that can be read with a spectrophotometer at 450 nm. This developed colour is inversely proportional to the amount of analyte in the sample.

The enzyme-labeled analyte is commonly called the "conjugate" because the analyte is conjugated to an enzyme. The enzyme functions as a "label" to indicate the specific amount of analyte bound to the antibody. This type of immunoassay is commonly called enzyme-linked immunosorbent assay, ELISA or EIA. The enzyme is coupled or "linked" to the analyte. The antibody attached to the magnetized particle acts as the "immunosorbent", adsorbing the analyte through its specific binding sites.

RaPID Assay utilizes magnetized particles as the solid support to assist in capturing analytes. Use of these particles provides important benefits:

- Added as part of the reaction mixture in the assay, the magnetized particles with attached antibodies are dispersed evenly throughout the sample. The proximity to the analytes provides optimum antigen-antibody kinetics.

- The particles are specially coated, providing an even surface for uniform attachment of the antibodies and leaving no available surface for attachment of non-specific contaminants. Their small size (about 1 micron in diameter) provides a large total surface area to which numerous antibodies can be bound.

- The magnetized particles allow for controlled washes using a magnetic rack. After the sample, enzyme-conjugate, and antibody-coated particles react, the magnetic rack pulls and holds the particles, with analytes attached, to the bottom of the reaction tube. The particles can then be easily and effectively washed to remove all unbound matter, without any loss in sensitivity.

Assay Steps (Figure C)

1. The sample, enzyme-conjugate, and antibody-bound particles are combined.

2. The analyte in the sample and the enzyme-conjugate compete for antibody binding sites; each has an equally strong affinity for the sites. Therefore, a high concentration of analyte in the sample will occupy more antibody binding sites, leaving fewer sites available for the conjugate. Conversely, a low concentration of analyte in the sample will occupy fewer antibody binding sites, leaving more sites available for the conjugate.

3. Following the reaction period, the magnetic rack is applied and the particles are washed.

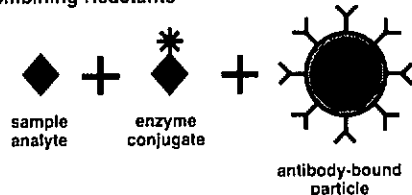
4. Next, a colour solution is added. This solution provides a substrate for the enzyme (horseradish peroxidase, HRP) and a chromogen (tetramethylbenzidine, TMB) that will turn blue as the enzymatic reaction progresses. The amount of colour development in the solution is directly correlated to the amount of enzyme-labelled conjugate bound to the antibody and is inversely proportional to the amount of analyte in the sample.

5. A stop solution (dilute sulfuric acid, H_2SO_4) is added at a predetermined time of the colour incubation. The stop solution maintains optimization of the enzyme kinetics. The stop solution also will change the developed blue colour to yellow. Because this is a competitive assay, darker yellow indicates less analyte in the sample, and a lighter yellow indicates higher levels of analyte in the sample.

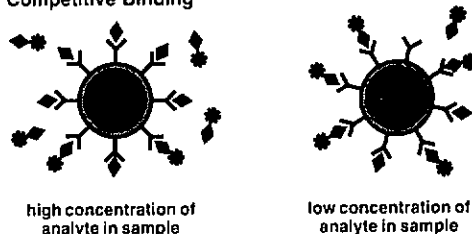
6. The reaction tubes containing the developed color solution are read in a spectrophotometer with absorbance at 450 nm. A standard curve is plotted, and the actual concentration of the specific analyte present in the test sample is determined.

Figure C. The Assay Steps

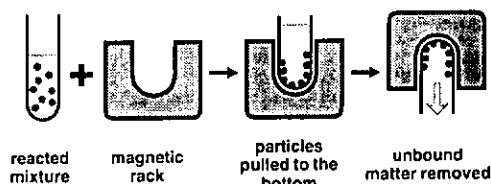
Step 1: Combining Reactants



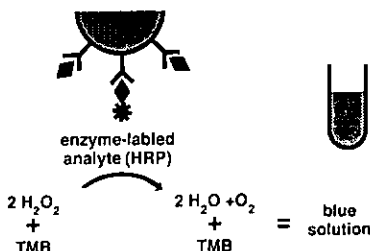
Step 2: Competitive Binding



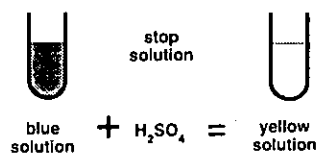
Step 3: Collection and Washing of Particles



Step 4: The Color Reaction

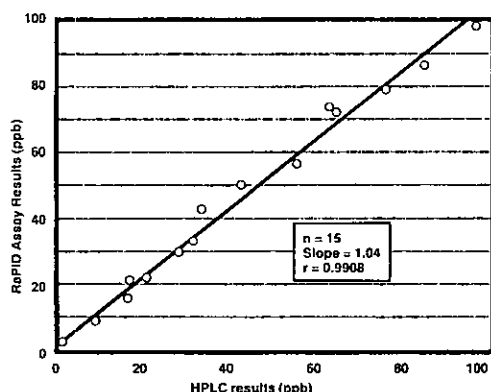


Step 5: Adding the Stop Solution



enzyme label = horseradish peroxidase (HRP)
 substrate = hydrogen peroxide (H_2O_2)
 chromogen = tetramethylbenzidine (TMB)
 stop solution = dilute sulfuric acid (H_2SO_4)

Figure D. Benomyl/Carbendazim Correlation: RaPID Assay vs. HPLC



Correlation With Conventional Methods

All RaPID Assays are compared to conventional methods prior to commercialization. Figure D displays the correlation between Benomyl/Carbendazim concentrations as determined by RaPID Assay and HPLC in 15 samples. These samples were chosen at concentrations within the limits of the HPLC method, and were diluted into the low detection range of the RaPID Assay. The correlation coefficient (r) is 0.9908 with a slope of 1.04.

Methods and Certification

Immunoassay techniques have been approved in US Environmental Protection Agency (US EPA) SW-846 series

4000 methods. RaPID Assays have been certified by the California Regulatory Notice and are an AOAC recognised method.

Any reliable method can be used for non-mandatory site work. A list of EPA validated kits is available; any kit not on the list can be used if the appropriate validation data is generated and documented. A guidance document for validation is available from the US EPA Office of Solid Waste.

Conclusion

RaPID Assays are an analytical system capable of detecting very low levels of toxic organics and pesticides with a high degree of selectivity. Each kit is available in either 20 or 80 sample sizes and provides all necessary solutions for the assay, three-point calibration curves, and controls.

For further information including ordering information contact:
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P O Box 31-180 Milford, Auckland
Ph: (09) 4791442, Fax: (09) 4791442
circle number 51 on the reader reply card

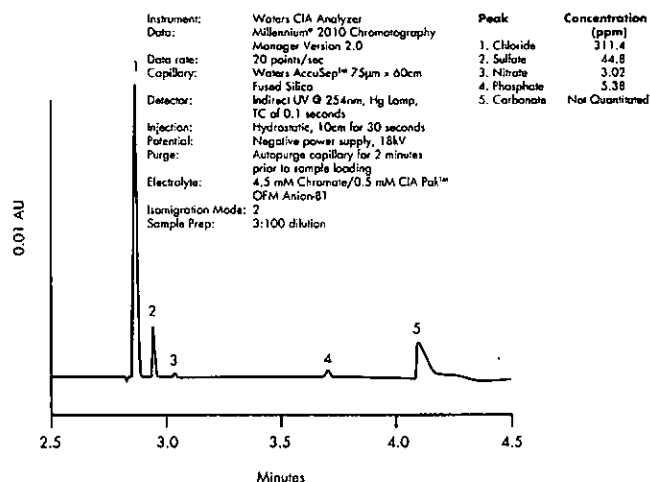
Using Capillary Ion Analysis to Monitor Wastewater

Waters Corporation, 34 Maple Street, Milford MA 01757, USA

Capillary Ion Analysis (CIA) is a technique developed by Waters for analyzing low molecular weight inorganic and organic ions by capillary electrophoresis.¹⁻⁷ CIA is fast, with typical analyses completed in five minutes or less. Since there is no chromatography column involved in CIA, sample preparation is much simpler than with ion chromatography. The unique selectivity of CIA enhances the separation and identification of anions and cations in water.

The advantages of CIA are demonstrated in this study of anions and cations in wastewater discharged into a stream from a meat processing plant in Northern Kentucky. Samples of stream water and wastewater, collected upstream from the point of discharge, at the point of discharge, and at various points downstream from the plant, were analysed to monitor dilution of the wastewater as it mixed with the stream water.

Figure 1: Rapid Anion Analysis of Wastewater by CIA - Fall Sampling



With CIA there are no interferences from cations, organic acids or neutral compounds.

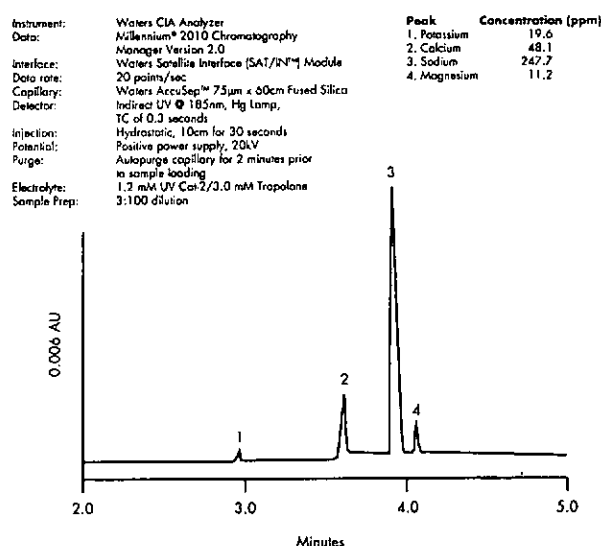
Sampling Strategy

The wastewater from the meat processing plant discharges from a pipe into a creek, which flows into the Licking River, and eventually feeds into the Ohio River. No other industrial site in this area discharges wastewater into the same creek. Approximately 200-300 yards below the discharge site, water from a nearby lake drains into the creek via a spillway.

Water samples were taken at different points along the creek during summer, fall and winter to investigate seasonal concentration loading and dilution rates. Water samples were taken upstream from the discharge tube, directly from the tube, and at various points downstream as well as from the lake water and at the point where the lake water and creek water mix. The sample of the mix was not available in the summer because the lake was low and no water was flowing over the spillway.

Figure 1 shows the results of CIA analysis, or electropherogram, of the anions in a water sample taken 200 feet downstream from the discharge pipe. Figure 2 shows the cation analysis of the same sample. Both samples were analysed with Waters CIA Analyzer. To convert from anion to cation analysis usually takes less than fifteen minutes, a much faster and easier task than switching between comparable applications in ion chromatography. Summary results for the fall sampling are shown in Table I along with percent relative standard deviations (%RSD) for triplicate analyses of the water samples.

Figure 2: Rapid Cation Analysis of Wastewater by CIA - Fall Sampling



Increased sensitivities for cations was achieved by detection at 185nm.

CIA is Fast. Sample Preparation is Simple.

CIA is a very rapid technique with analysis times being very brief. Sample preparation procedures are much simpler, and therefore faster than what is often required for ion

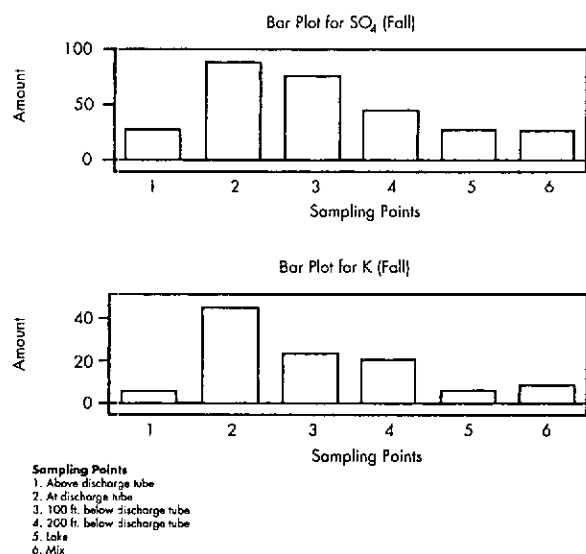
Table 1: Summary of Results for Fall Sampling*

	Above Discharge	At Discharge	100 Ft. Below	200 Ft. Below	Lake	Mix
Cl ⁻	17.7 (0.63)	1112.5 (0.11)	511.7 (0.17)	311.2 (0.22)	6.8 (1.0)	12.2 (1.1)
SO ₄ ⁻²	25.9 (0.34)	87.3 (0.64)	74.8 (0.54)	45.2 (0.60)	27.2 (0.56)	27.0 (0.42)
NO ₃ ⁻	ND	11.7 (1.05)	4.8 (1.21)	3.08 (1.12)	ND	ND
HPO ₄ ⁻²	ND	20.2 (0.40)	9.6 (1.28)	5.4 (0.92)	ND	ND
K ⁺	5.5 (1.73)	43.8 (1.22)	22.8 (1.62)	19.4 (2.10)	5.9 (1.89)	6.8 (2.21)
Ca ⁺	39.2 (0.98)	42.4 (1.52)	53.6 (1.36)	48.5 (1.22)	43.9 (1.18)	40.0 (1.33)
Na ⁺	14.1 (1.02)	790.3 (0.47)	336.1 (0.82)	246.7 (0.34)	7.1 (1.35)	11.2 (1.20)
Mg ⁺	11.2 (1.14)	12.8 (1.32)	14.5 (1.62)	11.4 (1.45)	8.6 (1.78)	8.4 (1.56)

*Amounts in ppm (µg/mL), N=3, %RSD in parenthesis, ND=none detected

chromatography. This is because there is no column packing to contaminate or equilibrate. Separation in CIA is based on a different mechanism than ion chromatography. It takes place in a capillary filled with an easily replaced electrolyte solution. The separation occurs when an electric field is applied to the electrolyte. For anion analysis, inorganic anions, organic acids and neutral compounds migrate at different speeds toward the detector, while cations move in the opposite direction. This eliminates the possibility of one group of compounds interfering with another. Sample preparation in this study consisted of merely diluting and filtering the samples prior to injection.

Figure 3: Summary Bar Plots for a) Sulfate and b) Potassium - Samples Collected in Fall (amounts are in ppm)



As shown in Table I, Na⁺ and Cl⁻ levels increase dramatically at the point of discharge compared to the background level of these ions in a sample taken upstream from the discharge tube. The main ions of interest are chloride, sodium, potassium, sulfate, phosphate and nitrate since they are typically byproducts of meat processing. Magnesium and calcium remain fairly constant. This is due to leaching of these ions from exposed limestone rock in the area along the creek.

Elevated levels of ions measured at the discharge point are rapidly diluted downstream but typically do not reach normal background levels until mixed with the lake run-off. Summary bar charts of the concentration-sampling point profiles for each ion were produced by Waters Millennium 2010 Chromatography Manager. Example reports are shown in Figure 3. Using Waters Capillary Ion Analyzer System with the Millennium Chromatography Manager provides a reliable means of monitoring ions in complex samples with minimal

sample preparation, rapid analysis times and versatile processing and presentation of the results.

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OBITUARY: DR JOHN ROGERS: 1919-1995

John Rogers was a man with a great zest for life which was expressed in his keen interest in people and a very wide range of subjects and activities of which the New Zealand Institute of Chemistry was of great importance.

Although he was a distinguished chemist who published some twenty five papers and made a significant contribution to the science of agriculture, his vision was not limited to ivory towers. He had a lively interest in commercial and industrial matters, was an enthusiastic and proficient gardener and worked tirelessly for a number of voluntary organisations. His sound common sense always took him straight to the heart of a problem when he did not flinch from his chosen course of action. This sometimes brought him into conflict with others but he did not lose their respect.

John was born at Walton-on-Thames in England and moved to Christchurch at an early age where he completed his primary schooling and went on to Christchurch Boy's High School before entering the then Canterbury University College in 1937, where he was Lizzie Rathbone Scholar, Sir George Grey Scholar and won the Hayden Chemistry prize. After graduating MSc with First Class Honours in 1940, he, like many others in that time of war went to work in the CSIRO laboratories at Fisherman's Bend in Melbourne until 1945.

During his time there he married Molly whom he had met, through their common interest in tramping, while they were students at Canterbury College. Molly's journey from Christchurch to Melbourne for the wedding was extraordinary for those wartime days with the Auckland to Sydney stage by Short Empire flying boat requiring the Prime Minister's approval.

Following his time in Australia, John worked for the Soil Bureau in Wellington before taking up a position as Senior Lecturer in the School of Mines at the University of Otago, in the course of which he spent a year at MIT as holder of Fulbright and Nuffield Scholarships.

While the family, which now included two sons and a daughter, lived in Dunedin they continued their pursuit of outdoor pleasures with camping, climbing and tramping. Then came two years sponsored by Consolidated Zinc and the Ernest Oppenheimer Foundation working in the Department of Colloid Science at Cambridge University, leading to a PhD.

John's next move was to Canada to work for two years as a research officer with International Nickel.

1959 saw the family back in Dunedin where John took charge of the Geological Survey's high pressure/high temperature research at the University of Otago.

Seven years later John was Director of the New Zealand Fertiliser Research Association's station at Otara in South Auckland where he remained until his retirement in 1980. John was a strong supporter of the Research Association idea which gave tangible government input to the technical base of New

Zealand industry and, through his vision and energy, he was able greatly to increase financial support from the fertiliser industry and thereby completely change the focus and scope of the work at Otara. New equipment was acquired, engineers and professionals of other disciplines were added to the staff and the work was taken out of the laboratory into the field with meetings and demonstrations throughout the country. It was a complete transformation.

After retiring from the Research Association, John visited Pakistan to advise the government on establishing a fertiliser research laboratory.

John joined the New Zealand Institute of Chemistry in 1946 and was elected an Honorary Fellow in 1986. He was the Institute's first Easterfield Medallist and he served it in many ways at branch and national level over more than forty years. He was on both the Otago and Auckland branch committees in positions including secretary, chairman and council delegate. He also served as Conference Secretary in 1948, as Conference Chairman in 1965 and in 1988-89 was Second Vice President.

During his eight years as Honorary General Secretary, John played a large part in establishing the Chemical Education Trust, the AC Kennett and the NZIC/RACI Visiting Speaker awards and the Chemists' Support Package to assist chemists seeking employment.

On his travels overseas, while working or in retirement, he always took the opportunity to make contact on behalf of the Institute with kindred bodies in Australia, Britain, Germany, Malaysia, Singapore, South Africa and Thailand for example.

It remained a disappointment to John that the Institute has not been strengthened by more formal links with the RACI of which he had also been a member.

Other scientific bodies to which John contributed his energy and ideas were the New Zealand Geochemical Group (founding Chairman and Life Member), Australasian Institute of Mining and Metallurgy (New Zealand Councillor for five years), American Institute of Mining, Metallurgical and Petroleum Engineers, The Auckland Institute and Museum (Councillor for many years) and New Zealand Institute of Agricultural Science.

John's knowledge of and love for horticulture showed in his own garden, which regularly produced prize-winning blooms, and his membership of such organisations as the Auckland Lily Society, Friends of Auckland Regional Botanic Gardens and sometime Chairman of the Auckland Horticultural Council. For many years, he was a fellow of the Royal New Zealand Institute of Horticulture.

With John's death, the New Zealand Institute of Chemistry has lost a devoted and forceful advocate, supporter and friend who will long be remembered for his wide range of interests in his humanity.

Raymond Hopgood

NZIC NEWS



FROM THE PRESIDENT...

May I take this opportunity to wish you all a prosperous and successful 1996.

Dr John Rogers

I should like to extend my personal and the NZIC's condolences to Mrs Molly Rogers on the death of her husband, Dr. John Rogers recently. John will be remembered as an ardent supporter and long time member of the NZIC. Of late John had been concerned about the Institute's future. So much so that he wrote to the Institute in 1995 expressing those heartfelt concerns. John's words were heeded. Bill Denny began a process to produce a strategic plan from those concerns. In this indirect, but tangible way, John will have contributed to the reshaping of the Institute he was so proud of.

At John's funeral in the Anglican Church in Papatoetoe, Auckland I was impressed by the strength of the representation of the NZIC. I noted two past presidents; Drs Llywellyn and Wright, the current Executive; myself, Alan Turner and Denis Karl, numerous Auckland representatives and many others including Ian Devereaux who delivered the eulogy.

Audit

The audit of the Secretariat has been completed by Deloitte Touche Tohmatsu. The report was in the hands of the Standing Committee on 22/11/95 as required by the draft strategic plan. It will be given to the full Council at its meeting in early March. The audit was conducted over a two week period with input from the Executive Officer and myself. The audit was designed to identify the tasks carried out by the Secretariat. At this stage no attempt was made to determine either the quality or quantity aspects of each task. The initial reaction to the study would suggest that the Secretariat follows faithfully the duties prescribed for it at its inception. Further analysis will be required. For those who are interested the audit cost circa \$2,500 to undertake.

CASANZ

I spent a few days in Melbourne in late November. I attended a two day seminar entitled "Managing SO₂ in Australia". It was run by CASANZ, the Clean Air Society of Australia and New Zealand. The various papers presented discussed the effects of SO₂ on human health, plant life and other aspects of the environment. The only disappointing feature was the lack of New Zealand interest. Three out of fifty was the attendance on a subject very relevant to New Zealand.

RACE

I also had the pleasure of having a working breakfast with Dr Susan Cumming, Executive Director of the RACI. It was interesting to compare notes and respective challenges. Dr Cumming was envious of our Journal and RACI is now coming to terms with its own Journal. A change of format and printing techniques were looking positive. A second item of common interest was the balancing of the Secretariat resources. The RACI's office consists of a fulltime Executive Director and 5 or 6 helpers. This compares to the NZIC's 1/2 person and a helper. Because of the availability of resources the RACI was now looking after the interests of the Institute of Physics and the Chemical Engineers. Thus increasing its revenue and its services base. The NZIC through its strategic planning process was considering the opposite. Placing itself under the wing of another body, in this case the Royal Society, for the very same reasons, strength and resources.

A third item of interest was that Dr Cumming had initiated an audit of the RACI's Secretariat for much the same reasons that we have undertaken one of our Secretariat. Results were not to hand but should make interesting reading in the near future. The study was carried out by Deloitte.

Finally Dr Cumming and her staff produced a superb annual report for the RACI. It was high quality, done in-house, desk top style.

Challenge

In early December I visited a major chemical process plant in Northland. Our party was shown around the complex by one of its chemists, Ian Hamilton. Yes, he did belong to the NZIC. His eight colleagues, however, did not! We discussed at some length the ills of the NZIC. He agreed that change was needed.

However he said there was little of real relevance for him, as an industrial chemist, in the Journal. I would like to challenge Ian

as I was so challenged nine years ago. You have a process on site which is pure chemistry. You have a sophisticated analytical instrument which delivers a multiplicity of determinands within minutes as opposed to the traditional wet chemistry techniques which took days to complete. Write an article for the Journal on the process and analytical techniques. I am certain it could be written in such a way as to not impact on either technological or commercial sensitivity.

How many other chemists are there in New Zealand, like Ian, who treat their work very much as a norm? Yet to others it would be abnormal and fascinating. Come on Ian!

Elections

I read with interest Dr Jim Coxon's letter to the editor in the November 1995 issue of *Chemistry in New Zealand*. The thrust of Jim Coxon's letter was that the elected members of the Council should be emplaced by an elective process and not by rotation through the Branches. The proposition being that the best persons may not assume high office within the Institute.

Rule 16.2 of the Institute's Constitution allows for such to happen. Each year the Executive Officer calls for nominations for President, Vice Presidents, Honorary General Secretary, and Honorary General Treasurer. It is simply tradition that dictates the rotation process. If the practice was to be changed, and I

am neither for nor against the proposition, I would strongly recommend that the person so elected to be President be prepared to stand for a two year term. Again allowed for in Rule 16.5.

Waikato

Finally I should like to report on the Waikato Branch's final committee meeting for 1995. It was unusual in a number of respects. It was held over lunch at a suitable venue in Hamilton. Two Past Presidents were in attendance; Drs Llywellyn and Wright. Executive Officer, Alan Turner was present from Wellington. Institute issues were discussed at length. The discussions were all the more philosophical because of the presence of three genuine students. This was the third such working lunch held by my "home" Branch. I find them both supportive and informative.

We talk of changing the Institute. Making it more responsive to the members needs. I think a great deal can be achieved locally with enthusiastic chairpersons like the Waikato's Dr Peter Robinson. People who are prepared to be innovative at the grass roots level. We have said a great deal of our Secretariat. Invite our Executive Officer to your next meeting. He is remarkably human and he has an excellent presentation on trace chemicals in ethanol.

CHEMISTRY IS ALIVE!

Yes, very much so.



A few weeks ago Tim Foy, Head of Department of Science at Huntly College in the Waikato, invited me to the College. He had been running a 'hands on science' programme during the week for potential new entrants to the College. Groups from all the local primary schools had been taken through a series of twelve experiments. The visiting students had been supervised by Huntly College sixth formers. The theme was very much science and fun.

Tim Foy

Tim Foy uses the Institute. He took a party of 15 through the "Chem13" examination last year. He has in the past taken students to demonstrations from visiting Australian 'Magic Show' chemists.



Above: Group from Kimihia Primary School, Huntly. College sixth former Karl Aaron (second from right) was the demonstrator for this experiment.

We are looking to the future of the Institute. To realign it for the year 2000 and beyond. Tim Foy is important. Just as the other hundreds of his ilk are around New Zealand. ChemNZ is an outstanding success. That may be part of the answer. Aim for the grassroots. The NZIC has to develop a series of products that satisfy its members needs.

Nath Pritchard

Nath Pritchard, President NZIC

MANAWATU BRANCH NEWS

The Manawatu Branch AGM was held at Wharerata Staff Club on 6 December 1995. Twenty two members came to the meeting followed by dinner and Branch Chairman Mike Boland's speech "Of cows and genes and "active foods and aromatic rings".

In his annual report Mike Boland reviewed another successful year for the branch. He congratulated Sir Neil Waters on his knighthood and Sylvia Rumball on receiving the New Zealand Science and Technology Award and on her appointment as Dean of Science at Massey University. He noted the retirement of three members; Sir Neil Waters, Lady Joyce Waters, and Professor Geoff Malcolm, and wished them well for the future.

The following Officers were elected for the new term:

Chairman	Dave Harding
Treasurer	Alastair MacGibbon
Secretary	Grant Boston
Branch Editor	Harry Percival
Branch Committee:	Ces Johnson
	Mike Boland
	Gill Norris
	Mark Patchett
	Mark Smales
	Alan Furness
	Tony Burrell
	Clyde Smith
	Kath Fletcher (Hawkes Bay Representative)

Tony Wright was elected as a Trustee of the Manawatu Chemical Education Trust.

The meeting then discussed the "Future Options for the NZIC" document circulated to all members earlier in the year. The following resolution was passed unanimously:

"The Manawatu Branch of the NZIC supports the recommendations proposed in principle by the Strategic Review Committee and urges the Council to proceed with these reforms with expedition."

After a fine dinner, Mike Boland spoke on the role chemistry has in developing the cows of the future.

The next branch meeting is scheduled for early March when we will travel to Hawkes Bay and investigate the role of chemistry in the production of fine wine - see you there!

Grant Boston

BOOK REVIEWS

DICTIONARY OF ORGANIC COMPOUNDS 6th EDITION

J Buckingham and F MacDonald, Executive Editors
Chapman & Hall, 2-6 Boundary Row, London SE1 8HK, UK
ISBN 0 412 54090 8 (Nine volume set)

The last (5th) edition of the Dictionary of Organic Compounds was published in 1982, and the present sixth edition (DOC 6) has been keenly awaited, since it is a leading reference work for almost everyone who works with, or seeks information about, organic molecules. The present dictionary contains ca. 11,000 pages in 9 volumes, 3 of which are indexes, viz. name index, molecular formula index, and CAS registry number index.

The new edition has almost 30% more entries than the previous edition, and 50% of all entries are new giving a total 62,000 entries which cover approximately 170,000 compounds. Each entry gives the name of the compound, alternative chemical names and trade names, its CAS registry number, a structure diagram, molecular formula and mass, physical properties, toxicity and other hazard data, common derivatives, and literature citations. Each entry is also given its own dictionary number. Many (ca. 80%) of the entries have been revised and increased data is given, including upgraded hazard and toxicity data. These extra entries and the revised data has meant that a reduction in the size of the print has been necessary and the material is now presented in CPI 8 (point size) format. For anyone with poor eyesight one would not want it any smaller. The printed dictionary is attractively presented and is well-bound, as indeed it needs to be for volumes which are intended for heavy use.

The cost of the dictionary is not cheap; at \$NZ7,782.00 as at 30/11/95, but in my opinion well worth the price. The dictionary is also available in a CD-ROM version which allows access by text searching, i.e. from words in the text, CAS number, melting point, and also by structure and substructure searching. Appropriate software is provided. The CD-ROM version is to be updated bi-annually. At 30/11/95, a year's single user version subscription was \$NZ8,380.00 with a renewal cost of \$NZ2,275.00, or a one year's network subscription was \$NZ20,951.00 with a \$NZ5,687.00 renewal fee, although bona fide academic institutions qualify for a significant reduction in price. A special package which includes both the 9-volume printed version plus a year's user subscription costs \$NZ13,747.00 and the package with a network version \$NZ25,000.00. Again academic institutions qualify for a sizeable discount (39% for the later version).

Earlier editions of the dictionary were referred to as "Heilbron's Dictionary", or more usually just as "Heilbron" and the reference work has been in use for over 60 years. It is an essential compilation for any institution where organic compounds are used and is recommended for every library serving the needs of such people.

R C Cambie, Chemistry Department, University of Auckland

SI CHEMICAL DATA

Gordon Aylward and Tristan Findly, 3rd Edition
John Wiley and Sons, Milton, Queensland, Australian, 1994

As my first edition copy at home is falling apart from use and my second edition copy, always on my desk at work, is looking worn it was very timely to receive a copy of the third edition. I believe all chemistry undergraduates including first year students should possess a copy of this data book, and teachers should make sure students use it as part of their training. A look through the contents would be a valuable education experience for first year students.

There are both changes and new material in this 3rd edition. The most immediately obvious and probably the most significant addition is the assignment of a hazard code number to all compounds in both the tables of properties of inorganic and organic compounds. At the foot of each page of these tables the codes are explained in a condensed form and on pages 154-177 there is an appendix with expansions of these condensed codes. This information is invaluable, especially as we now teach under new laws on hazards and safety, and is an excellent source of information for students. Changes reflect advances in technology; gone are the log tables at the end of the book (modern students would not know how to use them!) and under decimal fractions and multiples come zepto, z, zetta, Z, yocto, y, and yotta, Y-10⁻²¹, 10²¹, 10⁻²⁴ and 10²⁴ respectively. To the table of fundamental constants are added the unified atomic mass unit, the Hartree energy, the molar volume of an ideal gas at 1

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Telephone (04) 528-0718. Fax (04) 528-0493



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bar and 0 °C and the standard acceleration of gravity. Symbols for thermodynamic data take the new format recommended by IUPAC, (e.g. ΔH_f becomes $\Delta_f H$).

Under the table of properties of organic compounds the list of common names has gone, but they appear for each compound in the table. The old table has been renamed 6A, *properties of organic functional groups*, and properties of seven carbohydrates are added, and a new 6B, *properties of amino acids (constituents of proteins)* appears with pK_a and pI values.

There is a new table on bond dissociation enthalpies to go with the average bond energies; the term *latent heats* is gone (but not the information); the table of *first ionisation enthalpies of the elements* has been reformatted as *periodic trends in first ionisation enthalpies*; *stepwise* stability constants are now *cumulative*; and there are new tables on boiling temperature elevation and freezing temperature depression constants, on critical constants of selected substances, on infrared absorption frequencies and on the electronic configuration of the elements. Inside the back cover are hints on first aid.

Is there anything else which might have been usefully added to the third edition? Yes, tables of 1H and ^{13}C chemical shifts in nmr and half-lives and emission class of some important radioactive isotopes would have been very useful from the viewpoint of a teacher of undergraduates.

Aylward and Findlay should be congratulated in making an excellent publication even better and up-to-date. I would recommend every teacher of chemistry have a copy on his or her desk. I can think of no better instantly available source of vital and common information for chemists.

J E Packer

Associate-Professor of Chemistry, University of Auckland

PACIFICHEM '95

Pacificchem '95 held in Honolulu, December 17-22 1995 was a huge success. It proved to be the world's largest-ever chemical meeting with 633 presentations from almost 8000 registrants over the five day meeting. The NZIC delegate on the organising committee, Professor Brian Halton (Victoria University) will present a more comprehensive report on the meeting in the next issue of *Chemistry in New Zealand*.

MICROBETA TRILUX WORKSHOP APRIL 15 and 16 1996

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"PERSPECTIVES IN MARINE NATURAL PRODUCTS 1996" Conference Centre, University of Auckland, 11-12 July 1996

This is a two-day symposium organised by the Department of Chemistry, University of Auckland and sponsored by the Auckland Branch, and the Fats and Oils Group, of the NZIC. It aims to promote interest in marine natural products and to show the importance of chemistry in this sphere. It follows similar successful symposia held in Auckland in 1982, 1987, and 1991.

The programme will consist of 11 invited lectures of 1 hour, and contributed papers of 20 minutes. The convenor welcomes additional contributions for the 20 minute lectures.

The provisional list of invited lecturers is:

- Dr J Blunt, Chemistry Department, University of Canterbury, Christchurch, New Zealand.
- Dr P Murphy, Australian Institute for Marine Science, Townsville, Australia.
- Dr R Capon, Organic Chemistry Department, University of Melbourne, Melbourne, Australia.
- Dr J Volkman, Department of Oceanography, CSIRO, Hobart, Australia.
- Dr B Bowden, Department of Chemistry and Biochemistry, James Cook University, Townsville, Australia.
- Dr M Garson, Organic Chemistry Department, University of Queensland, Brisbane, Australia.
- Dr J Coll, University of Central Queensland, Rockhampton, Australia.
- Dr T Molinski, Department of Chemistry, University of California, Davis, USA.
- Dr P Northcote, Department of Chemistry, Victoria University, Wellington, New Zealand.
- Professor R Anderson, Department of Chemistry, University of British Columbia, Vancouver, British Columbia, Canada.
- Professor DJ Faulkner, Scripps Institute of Oceanography, La Jolla, California, USA.

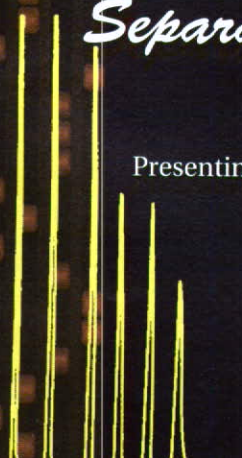
The social programme includes a seafood lunch on 11 July and a happy hour on the 12 July at the end of the symposium. Visitors from out of town can be accommodated at O'Rourke Hall on request to the Convenor, and parking in the lower car park can be arranged for a minimal fee.

As a consequence of generous sponsorship, the registration fee has been set at only \$100 and in order to encourage as many students as possible to attend, a fee of \$50 has been set for bona fide students. Registration forms are available from the Convenor, Professor R C Cambie, Department of Chemistry, University of Auckland, Private Bag 92019, Auckland. Telephone +64-9-3737599.

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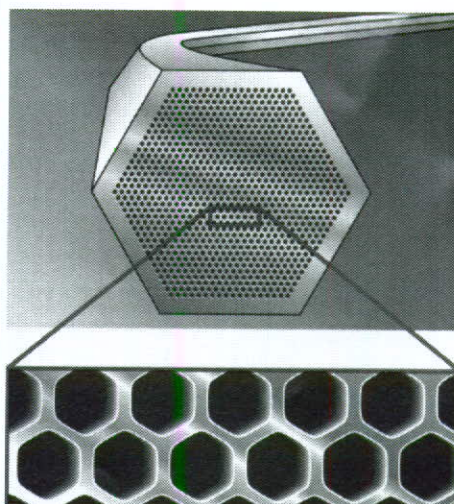


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