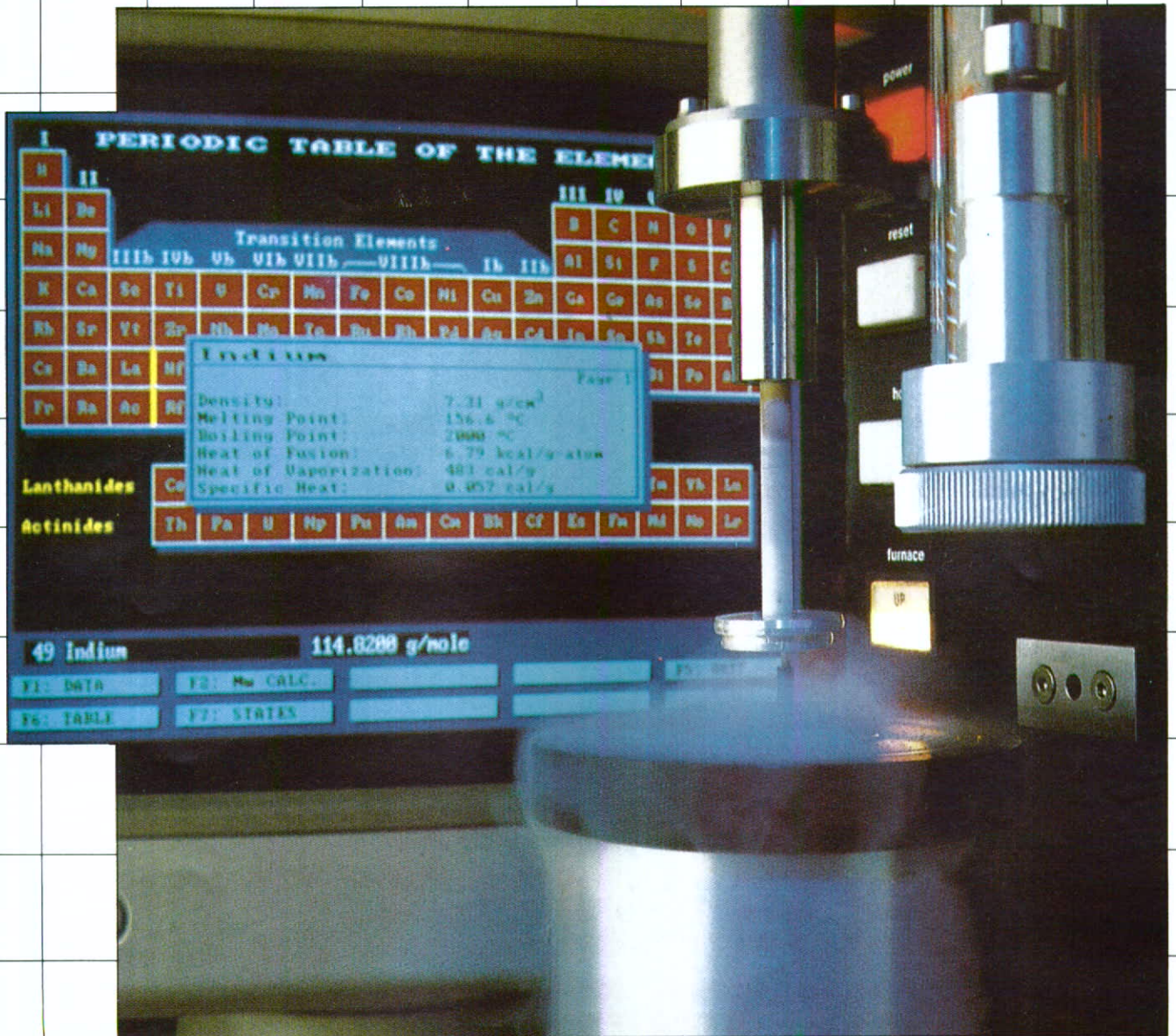




# Chemistry

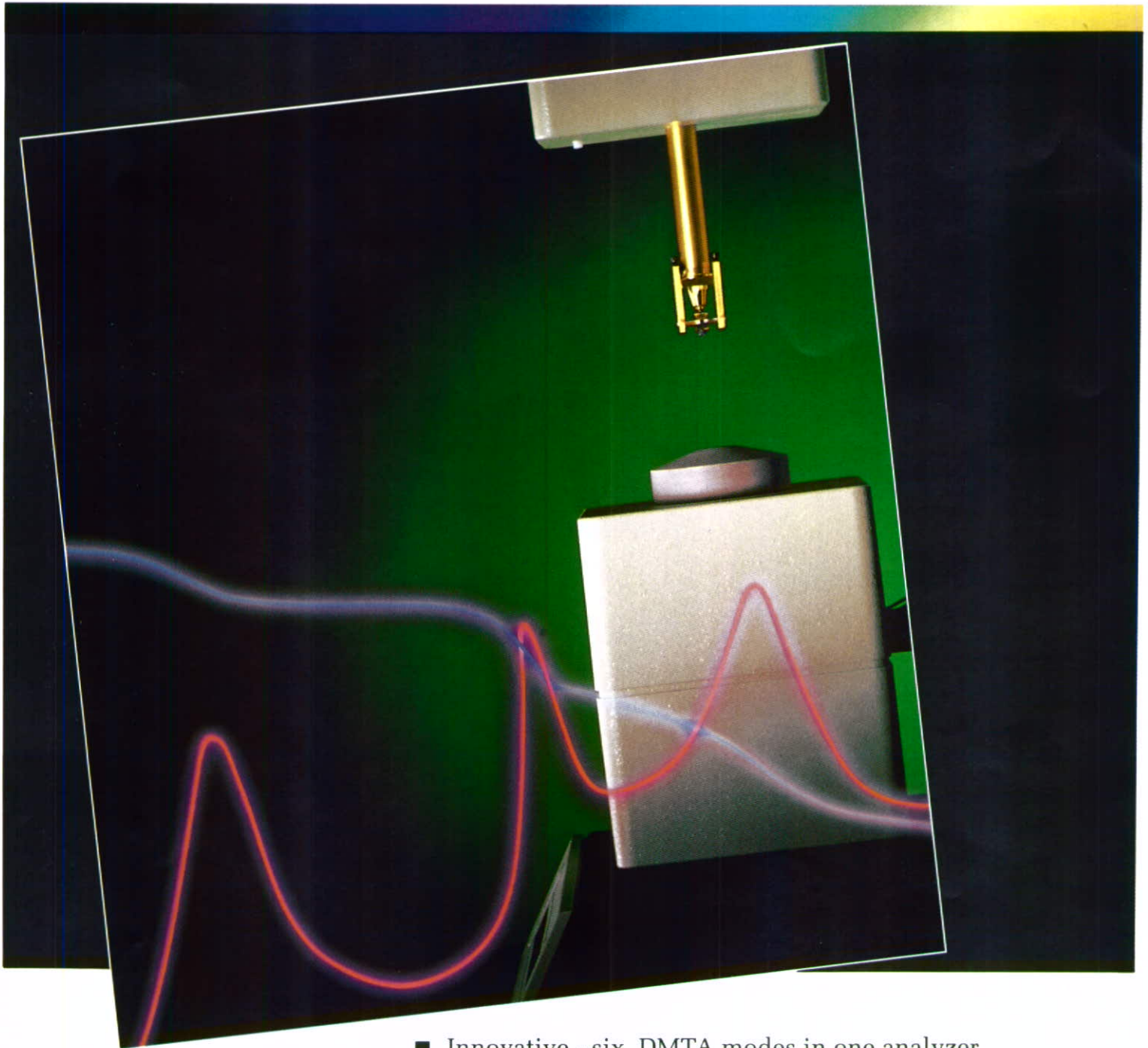
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In this issue we are doing something a little different.


The Otago Branch decided for their contribution to the Journal, that members would be interested in seeing what a wide range of interesting and challenging chemistry is being explored by students and their supervisors at Otago University.

We have arranged, in no special order, a series of very short summaries which collectively demonstrate that there is a wealth of interesting exciting challenging and useful chemical research taking place on an ongoing basis in New Zealand's universities.

The editor looks forward to receiving the considered contributions from other Branches for publication in your Magazine.

EDITOR


PACIFIC POLYMER FEDERATION




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3rd PACIFIC  
POLYMER CONFERENCE

13-17 December 1993  
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Organized by the Polymer Division of  
The Royal Australian Chemical Institute for  
the Pacific Polymer Federation.

**Pacific Polymer Federation**  
The Conference organising committee has posted the 1st brochure calling for papers. Information is available from Neil Edmonds Auckland Institute of Technology Private Bag 92006 Auckland Phone (09) 307 9999 Fax (09) 307 9973

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### Cover Story

When Polymer Laboratories acquired Stanton Redcroft and Omnitherm Corporation, the industry's finest thermal analysis instruments were brought together in the Thermal Sciences Division of Polymer Laboratories. Exceptional quality and precise temperature control are the bases upon which we build our instruments.

The first Simultaneous TGA-DSC/DTA Thermal Analyzer, - The first multi-module thermal analysis system which has been expanded to the first seven-module system, PL-ETA. - The patented PL-DMTA with torsion capability. Polymer Laboratories provides everything from differential scanning

calorimeters to dynamic mechanical thermal analyzers. For further information contact: Sci Tech, Science and Technology (NZ) Ltd. Auckland 270-3332, Wellington 566-6096, Christchurch 383-1146, Dunedin 477-7860.

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# THERMAL ANALYSIS

## INSTRUMENTATION AND APPLICATIONS

Bruce Fraser - *Douglas Scientific*

**Thermal Analysis is defined as "a group of techniques to measure a certain physical property of a substance (and/or its reaction products) as the function of temperature by changing the temperature of the substance according to a controlled program". Physical properties include mass, temperature, enthalpy, dimension, dynamic characteristics, and others depending on the physical properties to be measured.**

Physical property	Defined technique	Physical property	Defined technique
Mass	Thermogravimetry (TG)	Dimensions	Thermal expansion measurement
	Isotonic mass change method	Dynamic characteristics	Thermomechanical analysis (TMA)
	Evolved gas detection (EGD)		Dynamic thermomechanical measurement
	Evolved gas analysis (EGA)	Acoustic characteristics	Thermoacoustic emission measurement
	Emanation thermal analysis		Thermoacoustic measurement
Temperature	Thermal particle analysis	Optical characteristics	Thermooptical measurement
	Heating curve method	Electric characteristics	Thermoelectric measurement
	Differential thermal analysis (DTA)	Magnetic characteristics	Thermomagnetic measurement
Enthalpy	Differential scanning calorimetry (DSC)		

(1) The Society of Calorimetry and Thermal Analysis, Japan (ed.): *Foundation and Application of Thermal Analysis*, p. 1(1985).

Table 1. Classification of Techniques of Thermal Analysis

Thermal analysis has been applied to solve problems on numerous materials including glass, ceramics, metals, foods, pharmaceuticals and polymeric materials. The purposes of the application may be classified into the following three categories.

- a) Characterisation of materials
- b) Evaluation of chemical reactions
- c) Measurement of thermophysical properties

Temperature dependence of physical properties is influenced by various factors such as the molecular structure of the substance, molecular configuration and presence of other molecules. Consequently the measurement of temperature dependence of a physical property gives us information on the macroscopic state of the substance.

Thermal analysis is basically carried out by changing the temperature at a constant rate. Generally chemical reactions are carried out at constant temperature. In thermal analysis, the temperature is scanned at a constant rate from low to high temperature and so thermal analysis quickly gives us information about the temperature range where the reaction occurs. Applying kinetic theory we can estimate the reaction rate at a constant temperature from the measured temperature programmed thermogram.

Melting point, softening temperature, specific heat capacity, thermal expansion coefficient etc are important thermophysical properties. Generally in order to obtain a thermophysical property value a standardised measuring method should be employed. Recently thermal analysis test methods have been standardised and so it is possible to measure thermophysical properties by thermal analysis.

**2. Instrumentation** Of the four main modes of thermal analysis detailed below, differential scanning calorimetry is by far the most popular and widely employed

### 2-1 Differential Scanning Calorimetry (DSC):

DSC instrumentation is available in two operating principles: heat flux type and from one manufacturer, heat compensation type.

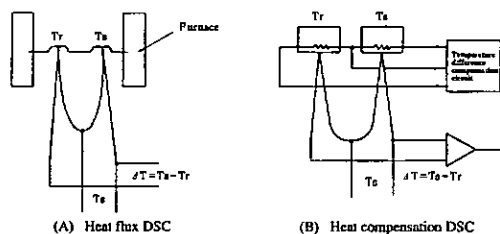


Fig 1. Principle of DSC

In the heat flux type DSC a sample and a reference substance are put on a metal plate (generally constantan is used as material for the thermocouple) connected to a furnace which is a heat sink, and the temperature difference of the sample and reference substance is measured. Since the difference of heat flowing into the sample and reference substance is proportional to the temperature difference of the sample and the reference substance, the heat flow ( $\text{mJ s}^{-1}$ ) is indirectly obtained by measuring the temperature difference. As the furnace has a uniform temperature distribution, noise due to temperature fluctuations in the furnace and due to changes in convection are extremely small resulting a stable baseline and hence sensitivity of measurement is very high. In heat compensation type DSC, two microheaters are used to heat the sample and reference substance individually and the supply of power to the microheaters is controlled to keep a zero temperature difference between the sample and reference cells. This difference in the supply of electric power ( $\text{mJ s}^{-1}$ ) is recorded. Since the heat capacities of the holder and microheaters are small, response to changes between sample and reference are rapid.

Either method gives high precision in the measurement of heat or specific heat and the data can be handled quantitatively.

Figure 2 shows a typical DSC curve. The sample is PEEK (poly ether ether ketone) film, weighing 8.18mg. The ordinate axis is in mW (mJ s<sup>-1</sup>) and the downward direction indicates absorption of heat, and the upward direction, release of heat. The abscissa is the temperature axis. In this example then, glass transition is found at 142.4°C, the low temperature crystallisation at 176.1°C and melting at 340.8°C. The heat of crystallisation is 97.5mJ and the heat of fusion is 150.2mJ. These calorific values are obtained from the peak area.

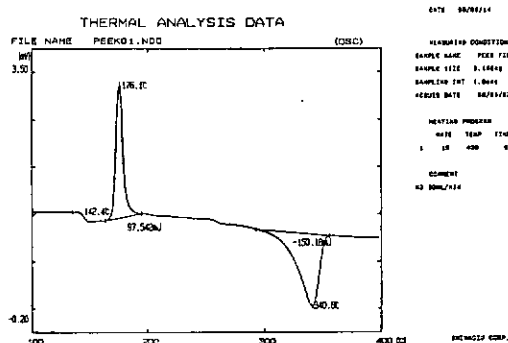


Fig 2. Typical DSC Curve

**2-2 Thermogravimetric Analysis (TG):** TG is a technique for measuring the change of mass in during a heating or cooling process or at constant temperature, and practical examples of application include evaporation, sublimation, decomposition, oxidation, reduction and adsorption and desorption of gas. TG is measured by a thermobalance which is generally one of three types: suspension type, top balance type and horizontal balance type. See Fig 3.

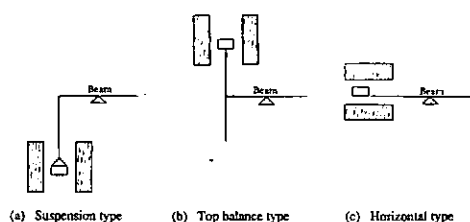


Fig 3. Thermobalance Methods

The suspension type has superior vibration resistance due to the low mass of the balance mechanism, baseline drift is small and measurement sensitivity is high. However, sample temperature cannot be measured directly.

The top balance design allows simultaneous DTA/TG measurement and sample temperature is directly measurable. However as it is less resistant to external vibration, the TG curve is likely to be much noisier. This configuration is also more susceptible to baseline drift.

The horizontal configuration is more tolerant of high gas flows and direct temperature measurement is easy. However the balance beam is expanded by heating and the balance sensitivity varies with both temperature and positioning of the sample.

Figure 4. is the TG and D-TG (differentiated) curve for the decomposition of calcium oxalate, the steps being: loss of water, loss of CO and loss of CO<sub>2</sub>. In addition, the D-TG curve shows a small peak at the beginning

of the second step of weight loss. This is due to the evolution of a small amount of CO<sub>2</sub> and it cannot be seen from the TG curve. The height of the D-TG peaks indicate the rate of weight loss.

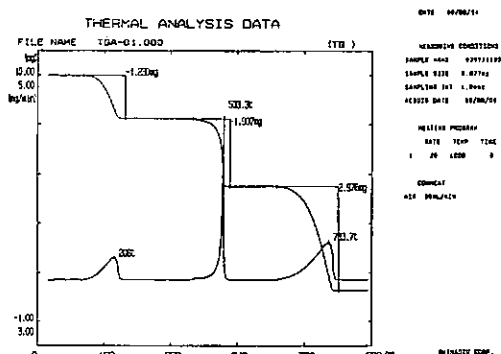


Fig 4. Typical TG, D-TG Curves

**2-3 Differential Thermal Analysis (DTA):** DTA is similar in principle to heat flux type DSC, the major differences being engineering ones as the upper temperature limit is around 1100°C (cf: around 700-750°C for DSC). Some DTA instruments are optionally available in high temperature options capable of operation up to 1500°C.

**2-4 Thermomechanical Analysis (TMA):** TMA is a method for the determination of the mechanical properties of a sample under compressive or tensile load in a thermally controlled environment. The applied load may be static or dynamic and the analysis reveals strain deformation of the sample along the stress axis. Additionally thermal expansion or contraction can be observed against time and/or temperature under constant load or no-load conditions. In both the stress-strain and expansion/contraction measurements the results are plotted and reported as dimensional change (usually in micrometers or percentage) against the load and/or time/temperature. TMA can give practical data for an enormous range of materials and finds use in areas such as the development, manufacture and QC of materials including polymers, glass, ceramics, metals and mixed or hybrid materials. Fig 5 (see below) shows thermally generated shrink-stress of plastic kitchen wrap. The plot of changing load against temperature is the shrink stress curve.

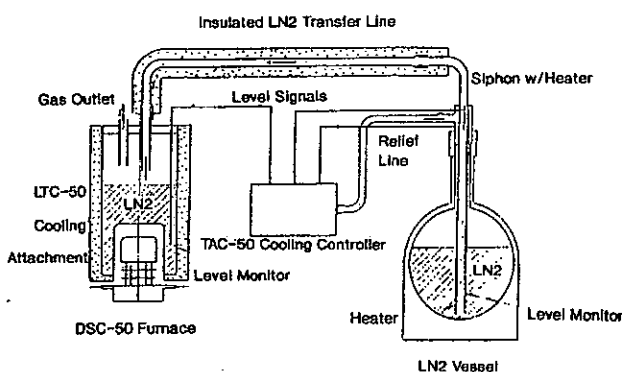
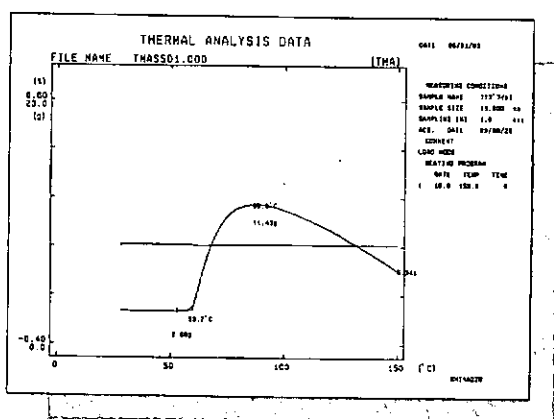


Fig 5. 11-1 Application of TMA-50

**2-5 Control systems and data processing:** A wide variety of data processing and control options now exist for modern thermal analysers with virtually all

systems offering PC control and data processing. Most systems run under DOS, some offer UNIX based systems and thermal software packages are now beginning to appear running under Windows which has emerged as a very popular platform for scientific software. Controllers vary from single to multi-instrument control and vary in their ability to process previously acquired thermograms whilst control of running instruments is proceeding as a background activity. In addition some instruments offer stand-alone control with more basic data output to a chart recorder.

**2-6 Accessories:** Generally a wide range of accessories exist to deal with a variety of analytical and sample requirements. Examples include: Devices for atmosphere control within the thermal analyser, passive and active cooling devices for subambient operation (Fig 6.), interface for coupled operation with FTIR allowing analysis of evolved gases.



Initial load was 2.68g with the onset of shrinkage at ca. 60°C. A maximum stress of 11.45g was seen at 86

Fig 6. "figure 2. Auto-cooling system diagram.

### 3. APPLICATIONS

A large number of applications exist, the following are intended as an illustrative sample. Melt and characterisation of polymers: Glass transition measurements: Screening for crystalline polymorphism: Measurement of water in starch gel: Measurement of absorbed water and water of crystallisation: Evaluation of effects of flame retardant treatment: Measurements of clay and ceramic materials: Interactions between components in pharmaceuticals: Oxidation of polymers: Kinetics of dehydration: Specific heat measurements: Measurement of thermal expansion coefficient: Measurement of softening temperature of plastic film: Melting of fats: Oxidation of edible oils: Retrogradation of starch: Investigation of freeze drying conditions: Purity measurement with DSC: Thermostability testing.

#### Conclusion:

Developments in hardware design, temperature control, detector performance reliability and ease of use over the last few years have seen thermal analysis move to increasingly common use in a wide range of applications such as polymers food, pharmaceutical, and materials science. The techniques can quickly provide useful answers either not obtainable or only obtainable with greater difficulty using alternative analytical techniques.

## PACIFICHEM '95

Planning for Pacificchem '95 has begun! At a ceremony in Honolulu on August 8 the American Chemical Society (ACS), the Canadian Society for Chemistry (CSC), the Chemical Society of Japan (CSJ), the New Zealand Institute of Chemistry (NZIC), and the Royal Australian Chemical Institute (RACI) signed a formal agreement to co-sponsor "The 1995 International Congress of Pacific Basin Societies" (Pacificchem '95) to be held in Honolulu from December 15-22. This represents the first congress of this nature in which NZIC (and RACI) is a formal co-sponsor. Members should not become alarmed at possible costs to NZIC as responsibility for the congress on the three large societies. Our efforts in assisting with the congress planning are recognised with a financial incentive based upon the number of New Zealanders that attend.



Agreement Signed for Pacificchem '95: Seated (left to right): Joe Dixon (ACS), Larry Weiler (CSC), Hitoshi Ohtaki (CSJ) Standing: Graham Johnston (RACI) and Brian Halton (NZIC)

At a three-day planning agreement was reached on ten broad subject areas for the congress. These are:

- 01 AGROCHEMISTRY: including agriculture, cellulose, carbohydrate, pulp and paper chemistry.
- 02 ANALYTICAL CHEMISTRY: including clinical, electrochemical and trace analysis.
- 03 BIOSCIENCE AND TECHNOLOGY: including microbial and pharmaceutical chemistry.
- 04 CHEMICAL ECONOMICS AND BUSINESS:
- 05 CHEMICAL EDUCATION:
- 06 ENVIRONMENTAL SCIENCE AND TECHNOLOGY:
- 07 INORGANIC CHEMISTRY: including nuclear and geochemistry.
- 08 MACROMOLECULAR CHEMISTRY:
- 09 ORGANIC AND MEDICINAL CHEMISTRY:
- 10 PHYSICAL CHEMISTRY

Each of these areas will consist of thematic symposia and anyone interested in organising a symposium of their choice can submit a proposal to do so. The Congress requires that every symposium have at least THREE co-organisers, each one from a different Pacific Basin Country, with the main proponent acting as the co-ordinator for the arrangements. Anyone requesting approval to organise a symposium can obtain the requisite proposal form and further information from the NZIC Pacificchem representative whose address appears below. The deadline for submission is April 15, 1993 (notification of acceptance, deferral or rejection will be made by July 15, 1993) in order to allow for about 70% of the conference theme to be set by the

Go to page 90

# RESEARCH ACTIVITIES AT THE UNIVERSITY OF OTAGO

## ANALYTICAL CHEMISTRY CONSULTING AND RESEARCH CENTRE - CHEMSEARCH

### Chemistry Department, University of Otago

The Centre operates within the Chemistry Department and uses the facilities and staff of the Department. The Director of the Centre is Dr. R.G. Cunningham with Dr. M.R. Reid as the Manager who handles most of the initial enquiries and carries out or supervises the analyses.

The recent installation of an inductively coupled plasma emission spectrometer will enable multiple metal and some non-metal analyses on a wide range of samples to be carried out. Trace metal analyses in water samples, soil and biological materials can be carried out on a single sample and are generally very cost effective. For ultra trace metal analyses the well established techniques available along with the refurbished class 100A clean room will continue to be used.

While not directly part of the Consulting and

Research Centre the Campbell Microanalytical Laboratory continues to provide elemental analyses of the highest standards. The range of analyses offered is more extensive than many other laboratories can conduct and this, along with our reputation for reliability has resulted in increasing business from Australia. With the availability of the ICP spectrometer we shall be able to develop methods for routine analyses of metals and non-metals such as sulfur, phosphorus, arsenic etc. in organometallic compounds. At present such analyses are often impossible due to interferences.

In addition the centre carries out a variety of chemical consulting and analytical work for businesses and public bodies. much of this work is non routine and provides interest along with a significant research component at times.

### CLAY - A VERSATILE RESOURCE

MELVILLE CARR

Chemistry Department Otago University

When we think of clay we may picture dirty shoes, slippery walking tracks, making pottery and impervious layers in the garden. We would be unlikely to think of clay as a major storehouse of nutrients in soils, drilling mud for deep holes, decolourising and purifying agent and as a catalyst or even of shape-selective catalyst. In addition clays have been used as fillers in polymers and rubber, as the base for face powders (talcum), in blackboard "chalk", and as essential fillers in glossy paper. This great variety of uses is a consequence of the unique features of clay mineral structures and their chemical compositions.

Clays have been widely used in pottery by man for at least 7000 years because they are found all over the earth's surface and can be easily shaped, become rigid on drying and hard and strong on firing. Clays contain aluminosilicate minerals derived from the primary earth rocks through weathering processes. The crust of the earth consists essentially of silicon (29%), oxygen (47%), aluminium (8%), together with iron (5%), calcium and magnesium (6%), and sodium and potassium (5%). Hence the minerals of the crust are mainly aluminosilicates of calcium, magnesium, sodium, potassium and iron or oxides of silicon and iron. It has been estimated that the sediments (shales, mudstones, siltstones, etc.) on the continents consist of about 50% clay minerals.

Clay minerals are hydrated layered or sheet silicates. The basic building blocks of silicate mineral structures are  $\text{SiO}_4^{4-}$  tetrahedra which can "polymerise" in a variety of ways to form chains, rings, bands, sheets and three-dimensional structures. Each type of structure has a characteristic Si:O ratio being 4:10 for sheet silicates, i.e.  $\text{Si}_4\text{O}_{10}^{4-}$ . Since Si and Al atoms are of a

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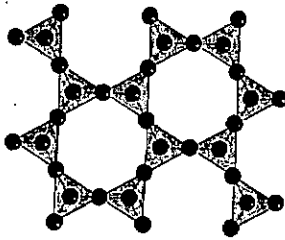
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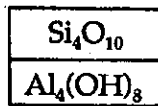
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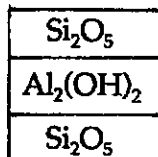
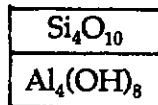
similar size Al may replace Si in  $\text{SiO}_4^{4-}$  tetrahedra and the charge is balanced by introduction of a cation e.g.  $\text{Na}^+$ ,  $\text{Ca}^{2+}$  etc into the structure.



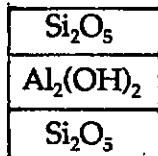
Clay mineral structures consist of silicate tetrahedra linked through corners to form an hexagonal pattern extending infinitely in two dimensions.



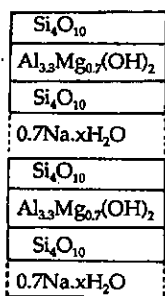
The layer of  $\text{SiO}_4$  tetrahedra is bonded to a layer of magnesium or aluminium ions to form a two-layer structure. Such layers may stack one upon the other to form the clay mineral kaolinite (China clay),  $\text{Al}_2\text{Si}_2\text{O}_7(\text{OH})_2$ .



A three-layer structure forms when the layer of metal and hydroxide ions is sandwiched between two tetrahedral silicate layers. The minerals pyrophyllite,  $\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2$  and talc,  $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$  contain stacks of three-layer structures.



If substitutions occur in either the silicate or the metal hydroxide layers of talc and pyrophyllite then the smectite structure containing four layers is formed. Substitution of aluminium by magnesium in pyrophyllite requires charge compensation in the interlayer region:

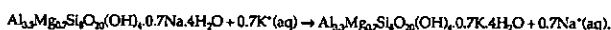


Each of these clay structures has very weak van der Waals forces bonding the structural units. The mechanical properties of clay minerals are due to these weak interlayer forces. All of the clays are very soft and have lubricating properties e.g. talc feels greasy and is the major component of soapstone. The softness and whiteness of kaolinite allows its use as blackboard chalk.

Another important property of clay minerals is their capacity to function as ion exchangers. For example an edge -OH group may behave thus:



It is found that kaolinite and pyrophyllite have very low ion exchange capacities because they have few accessible broken -OH bonds at their crystal edges. Smectite minerals such as montmorillonite have high cation exchange capacities because their interlayer cations can be completely replaced by other ions:



Anion exchange with ions like  $\text{HPO}_4^{2-}$  which fit onto the edges of the silicate layers occurs to a limited

extent. Since soils contain about 50% mineral matter and humus and much of the mineral matter is clay, then clays act as plant nutrient reservoirs in soils by storing many of the essential elements,

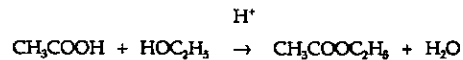
e.g.  $\text{NH}_4^+$ ,  $\text{HPO}_4^{2-}$ ,  $\text{H}_2\text{PO}_4^-$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{K}^+$ ,  $\text{Zn}^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Co}^{2+}$  in their exchange sites.

These ions can then pass into the soil solution and be absorbed by plant roots.

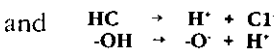
In montmorillonite the interlayer cations are normally hydrated and the interlayer region tends to attract water into itself by electrostatic, hydrogen bonding and osmotic pressure forces. The result is that montmorillonite swells when wetted and becomes very slippery and even liquid. So montmorillonite-water slurries are used in drilling rigs to cool the drill, lubricate the cutting edges and lift the debris to the surface. The Abbotsford landslide of 1979 which demolished nearly 60 houses in a Dunedin suburb was caused by slippage along a thin layer of mudstone containing montmorillonite which had become saturated with water. Montmorillonite is a common weathering product of volcanic ash and has been partly responsible for many of the landslides occurring during heavy rain storms in the Taupo-Rotorua-Bay of Plenty region.

Montmorillonite not only absorbs large quantities of water into its structural interlayers but also can accommodate a wide variety of organic compounds because of electrostatic and van der Waals forces of attraction. This means that montmorillonite is an excellent absorbent and this characteristic is utilised in the decolourising and purification of vegetable oils such as cooking oil and Margarine with Fuller's Earth. Fuller's Earth is a silty material with good absorbent properties due to the presence of montmorillonite.

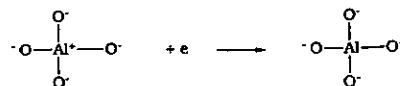
Many organic chemical reactions require acid catalysts e.g. esterification:



An acid can be either a proton donor:



or an electron acceptor:

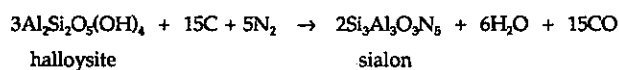


Therefore, in clay mineral structures accessible-OH groups and Al atoms substituting for Si are potential acidic catalytic sites. Several different types of organic reactions have been successfully conducted often with close to 100% yields in the presence of clay mineral catalysts. Just as ZSM-5 has electron pair acceptor sites and a channel size such that only organic hydrocarbons up to  $\text{C}_{12}$  may form from methanol so the montmorillonite structure may be modified to give a shape-selective catalyst with larger cavities than are found in any zeolite structures including ZSM-5. This is achieved by introducing large polymeric oxycations such as:

$\text{Al}_{13}\text{O}_4(\text{OH})_{24}(\text{H}_2\text{O})_{12}^{7+}$  (made from aluminium chloride and sodium hydroxide solutions) or  $\text{Zr}_{12}(\text{OH})_4(\text{H}_2\text{O})_{10}^{2+}$  into the interlayer space. These large ions replace the interlayer hydrated cations and prop the layers apart to form "pillared" clays. The

cavities are sufficiently large for the very long-chain hydrocarbon molecules present in heavy crude petroleum to enter and be "cracked" into smaller, more useful hydrocarbons such as those found in diesel, petrol and kerosene. Pillared clays have not yet been used commercially in oil refineries because they exhibit a high tendency for coke generation and their hydrothermal stability is inferior to that of the zeolites used in normal cracking operations. Zeolite catalysts do not accommodate the large molecules present in heavy crudes and hence pillared clays have enormous catalytic potential for the petrochemical industry. The problems of coking tendency and hydrothermal instability in pillared smectites are receiving much attention. If modifications to improve their performance are not possible then there are many processes requiring less refractory catalysts where pillared clays could be utilised.

Some of the largest white clay deposits of economic significance in New Zealand contain the disordered, hydrated kaolin mineral, halloysite which has physical properties unsuitable for ceramic use. Recent research in DSIR Chemistry laboratories has demonstrated that halloysite can be carbothermally reduced at about 1400°C in the presence of nitrogen gas to give a sialon phase:



Sialons, like silicon nitride  $\text{Si}_3\text{N}_4$ , have low density, high toughness and wear resistance but, in addition, are easier to sinter and are more chemically durable. Such materials are used in specialised products ranging from engine components and cutting tools to medical implants.

Clay minerals are, indeed a versatile resource and continue to receive much research attention internationally.

#### Further Reading

*Ceramics - Materials of the Past, Present and Future*, K.J.D. McKenzie and D.S. Perera, *Chem. N.Z.*, No. 33.

*Chemistry - An Ecological Approach*, R.G. Gymer, Harper and Row.  
*Advanced Inorganic Chemistry*, F.A. Cotton and G. Wilkinson, Interscience.

*Chemistry of the Elements*, N.N. Greenwood and A. Earnshaw, Pergamon.  
*New Ceramics from Indigenous New Zealand Minerals*, K.J.D. MacKenzie, I.W.M. Brown and G.V. White, *Chemistry in New Zealand*, 55, 78-84, 1991.

## THE HAEMOGLOBIN OF THE BRINE SHRIMP, ARTEMIA

ANNA M JELLIE.

Supervisor: Dr Clive N A Trotman

The intention of this project is to study the expression and structure of invertebrate haemoglobin. The haemoglobin protein of the crustacean species ARTEMIA is made up of individual units called protein domains. The protein is quite different from that of the mammalian globin as far as packing of the protein domains is concerned, however each individual domain is very similar.

An understanding of how the ARTEMIA haemoglobin fulfils its function despite significant structural differences from the mammalian counterpart will widen our



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Pearson Biologicals 137 Kilmore Street Christchurch Telephone: (03) 365-2556 Fax: (03) 365-0920 Contact: David Robertson

A Division of SGS New Zealand Limited 43 Church Street Onehunga Auckland Telephone: (09) 636-8186 Fax: (09) 634-1344 Contact: Ian McGill

understanding of the structure and function of the whole haemoglobin family.

Initially we are interested in the structure of the gene which codes for the haemoglobin protein. We are interested in how the coding regions, i.e. the regions coding for the protein, are arranged on the gene with respect to the non-coding regions and how this compares with mammalian globin genes. We already have evidence that the ARTEMIA haemoglobin gene is arranged quite differently.

The second phase of this work relates to actual protein structure. It is of considerable interest to know how the structure of ARTEMIA haemoglobin differs from other globins since this will extend the definition of the descriptive template of all globins. It is planned to isolate one of the repeating structural units of the protein for crystallisation and analysis.

In the long term, knowledge of the natural packing and linkage of these units may suggest ways in which mammalian globins could be aggregated in a blood substitute.

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## **IRON REGULATION OF PYOVERDIN PRODUCTION IN THE HUMAN PATHOGEN PSEUDOMOMAS AERUGINOSA**

T.R.MERRIMAN AND H.E. CUNLIFFE

Supervisor: Dr Iain L. Lamont

*Pseudomonas aeruginosa* is a Gram negative bacterium that infects people already debilitated by other diseases. It is particularly prevalent in hospitals and can be lethal to cystic fibrosis sufferers. During infections *P. aeruginosa* secretes a number of compounds, called virulence factors, which promote infection. One of these is pyoverdin, a chelating agent, that scavenges the iron necessary for the growth of the bacteria from host iron-binding proteins. Pyoverdin is secreted only during conditions of low available iron in the environment, as is the case during an infection. Our research projects involve the cloning and study of the master genes which respond to the signal of low free iron in the environment and instruct *P. aeruginosa* to synthesise pyoverdin, thus assisting this bacteria to infect.

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## **DETERMINATION OF THE NUCLEOTIDE SEQUENCE OF DNA BY SCANNING TUNNELLING MICROSCOPY**

STEPHEN SOWERBY AND ANDREA BRIDGMAN.

Supervisor: Professor George B. Petersen

Nucleic acid sequencing technologies currently available are reliant on the electrophoretic separation of DNA molecules differing in length by one residue. Gel based systems are typically capable of resolving molecules of 200 to 500 nucleotides in length, so that large DNA molecules must be broken into many smaller ones (subcloned) before sequencing. It has been estimated that, using such techniques, the sequencing of a length of DNA the size of the human genome would take some 30,000 scientist years and cost US\$3

billion. For genomic sequencing to be feasible, then, rates of data collection must be increased by several orders of magnitude, and a new method is desirable. Efforts to use electron microscopy to sequence DNA by direct inspection were initiated in the 1960s. It was then proposed that individual nucleotides could be identified by the attachment of an electron dense marker atom, and some promising results were obtained. Progress was hampered both by restricted microscope technology, and a lack of in depth understanding of nucleic acid chemistry. Since both these restrictions may now be surmounted, it seems timely to reinvestigate the use of microscopy as a sequencing tool.

Progress to date has concentrated on the development of methods for the labelling of DNA with electron dense marker atoms, by both direct chemical modification and indirect enzymic means. Investigations continue into methods for the mounting and visualisation of DNA, and the improvement of labelling techniques.

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## **CLONING AND CHARACTERISATION OF A cDNA CLONE ENCODING ASPARTATE AMINOTRANSFERASE FROM LUPINUS ANGUSTIFOLIUS**

CHRIS S. WINEFIELD

Supervisor: Dr Kevin J.F. Farnden

Aspartate aminotransferase (AAT) plays a key role in nitrogen metabolism in plants. It has been found to be involved in the shuttling of reducing equivalents from the cytoplasm to chloroplasts, mitochondria, glyoxysomes and peroxisomes, via the malate aspartate shuttle (Wightman F, Forest JC, 1978. *Phytochemistry* 17, 1455-1471). AAT has also been proposed to be involved in the transamination of oxaloacetate formed by PEP carboxylase in the mesophyll cells of some C4 plants (Hatch MD, Mau SL, 1973. *Archives of Biochemistry and Biophysics* 156, 195-206).

A *L.angustifolius* root tip cDNA library was constructed in the lambda Zap II expression vector (Stratagene) and screened using monoclonal antibodies (MAb) raised against AAT-P<sub>1</sub> from *L.angustifolius*. One 1452 base pair clone was isolated. The encoded cDNA sequence has high homology to both plant and animal AAT sequences. The clone, converted to the phagemid form, was expressed in *E. coli*. The expressed protein was enzymatically active and could be immunocomplexed with AAT-P<sub>1</sub> MAbs.

Future work will look at the expression of AAT isoforms in various tissues of *L. angustifolius*. Levels of expression will be quantitated using RNase protection assays.

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## **L-ASPARAGINASE IN LUPIN**

BRETTREDDINGTON

Supervisor: Dr K.J.F. Farnden

Temperate legumes, such as lupin, assimilate the ammonia produced from either symbiotic nitrogen fixation, the reduction of soil nitrates or the hydrolysis of seed storage compounds, mainly into asparagine. Asparagine is used by these plants as the major

transport compound to deliver nitrogen to developing tissues. Imported asparagine is hydrolysed in these tissues by asparaginase producing aspartate and ammonia for the synthesis of amino acids and other nitrogenous compounds.

Plant asparaginases have been shown to have two distinct forms based on cation requirements. A potassium-dependant form has been demonstrated in the young leaves, root tips and flowers of *Lupinus arboreus* and *Lupinus angustifolius*, and a potassium-independent asparaginase has been purified from the developing seeds of *L. arboreus* and *L. angustifolius*. A potassium-independent asparaginase cDNA has previously been cloned from a cDNA library prepared from the developing seeds of *L. arboreus*.

The aim of this project is to clone a potassium-dependent asparaginase cDNA, and to achieve this a system has been developed for the direct genetic selection of functional cDNA clones. A strain of *Saccharomyces cerevisiae* deficient in asparaginase was generated and this strain was complemented by the expression of the lupin potassium-independent asparaginase cDNA. As this *S. cerevisiae* strain can only grow if an active asparaginase is being expressed, this would be a powerful system for selecting functional asparaginase cDNAs from cDNA libraries, without relying on either DNA probes or antibody probes. cDNA libraries will be constructed using mRNA from plant tissues expressing potassium-dependent asparaginase and then screened using this system in order to isolate and characterise cDNA clones for this enzyme.

## PHD STUDY: SYNTHESIS OF THE SIDEROPHORE PYOVERDINE BY *PSEUDOMONAS AERUGINOSA*

J.L.RAE

Supervisors: Drs Iain Lamont and John Cutfield

*Pseudomonas aeruginosa* infects the lungs of cystic fibrosis sufferers, often in a fatal manner. In these conditions of low iron the bacteria secrete pyoverdine, a yellow-green fluorescent siderophore. Siderophores are comparatively small iron-binding compounds found in many bacteria, and fungi. The pyoverdine takes iron from the patients own proteins and is reabsorbed into the bacteria, where the iron is released.

Pyoverdine<sub>pa</sub> is 1500 Daltons in size and consists of a partly cyclic peptide linked to fluorescent chromophore

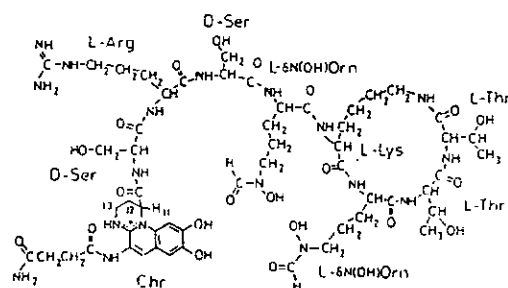


Fig 1. The structure of Pyoverdine<sub>pa</sub>

Presently, little is known about the biosynthetic pathways for pyoverdine synthesis. This project aims to investigate these pathways by analysing the pyoverdine

intermediates produced by mutant *Pseudomonas* bacteria, and determining the missing enzymes that convert the intermediates into normal pyoverdine.

Understanding some of the steps involved in the formation of pyoverdine may lead to future treatments of *Pseudomonas* infections; for example, the creation of specific enzyme inhibitors to block some of the steps in the pyoverdine biosynthetic pathway, preventing the bacteria from proliferating.

## CHARACTERIZATION OF AMINO ACID TRANSPORT IN CULTURED MAMMALIAN CELLS

BOONCHUANLOW

Supervisor: Associate Professor M.R. Grigor

Nutrients such as glucose and amino acids are transported into cells by carrier-mediated systems. Kinetic studies show that there are at least five different systems of amino acid transportation in mammalian cells depending upon the type or group of amino acids the protein (transporter) moved across the membrane, and whether the system is driven by energy or is facilitative. Hypertension may result from the restriction of blood flow by enlargement (hypertrophy) or increase in the number (hyperplasia) of vascular smooth muscle cells (VSMC) in response to certain vasoactive agents. Either effect will certainly involve the uptake of amino acids for the synthesis of constituent proteins and/or as a source of energy. The project will look at both basal stage and potential stimulation of the transport of selected amino acids into VSMC isolated from aortae of normotensive rats and the New Zealand Genetically Hypertensive rats.

## THE ASPARTYL PROTEINASE OF *CANDIDA ALBICANS*

R.J. WRIGHT, P.S. McNABB, A.D.J. SCADDEN

Supervisors: J. Cutfield, S. Cutfield, G.W. Emerson, and P.A. Sullivan

*Candida albicans* is frequently encountered as a commensal of the digestive and vaginal tracts of mammals, but it is also a common opportunistic pathogen. An aspartyl proteinase secreted by *C. albicans* is considered to be a virulence factor of the organism. There is a strong correlation between secreted proteinase activity and the relative virulence of different strains of *C. albicans*.

The gene encoding the enzyme of *C. albicans* strain ATCC 10261 was cloned using a probe generated by the polymerase chain reaction. Sequencing established that the open reading frame of 1194bp encodes a protein comprised of a prepro sequence of 56 amino acids and a secreted protein of 341 residues. The protein contains the two highly conserved aspartate residues at positions 88 and 274.

Recently the gene for a secreted aspartyl proteinase was cloned from *C. albicans* ATCC 10231. This gene has 73% overall identity with the gene from strain ATCC 10261. Some changes are significant and could alter the properties of the enzyme. Proteinase prepa-

rations from different strains of *C. albicans* do differ in substrate specificity, susceptibility to denaturation at alkaline pH values, inhibition and pI values.

As the sequence homology (73%) between the two supposedly equivalent genes was low, Southern hybridisations were performed using genomic DNA from *C. albicans* strains 10261 and 10231 to investigate the relationship between the two aspartyl proteinase genes. This showed that both genes are present in strains ATCC 10231 and ATCC 10261. The levels of expression of both genes is now being investigated. Other current work includes: crystallization of the strain 10261 proteinase, overexpression of the cloned gene in *Saccharomyces cerevisiae* to produce pure protein for crystallization and analysis of the upstream regulatory elements using DNA binding and footprinting assays.

## THE MOLECULAR BASIS OF LONG-TERM POTENTIATION: A MODEL FOR MEMORY

JOANNA WILLIAMS, TRISTAN ROBERTSON, CAROL RICHARDSON, SARA MASON, WICKLIFFE ABRAHAM Supervisor: Warren Tate

A widely accepted model for the long-term storage of information in the brain is referred to as long-term potentiation (LTP). The model suggests that an increase in synaptic efficacy coincides with the ability to store information. The aim of our work is to examine the molecular events associated with this synaptic enhancement.

Using Northern analyses we have shown an increase in the expression of immediate early genes (eg. *zif268*), with the early stages of LTP in awake rats. We are also investigating other candidate genes (eg protein kinases) which may be involved in the cascade of events that maintains LTP.

Other studies, *in vitro* have established the phosphorylation status of a protein synthesis factor, EF-2 changing with LTP. Interestingly, EF-2 phosphorylation is associated with an increase in immediate early gene expression in cells going from a quiescent to a proliferative state in mitosis.

## PROVERDINE GENES OF PSEUDOMONAS AERUGINOSA

IRENEROMBEL

PhD Supervisor: Dr I. Lamont

*Pseudomonas aeruginosa* is a bacterial species that can cause infection in immuno-compromized humans. This requires the synthesis of extracellular products which contribute to the infection. One of these products is a low molecular weight compound called pyoverdine (Figure 1). This is an iron-chelating agent with a binding constant for iron of  $10^{32}$  which enables *P. aeruginosa* to acquire iron from the patient. The acquisition of iron allows bacterial growth to occur, and hence the infection itself, to ensue. Production of pyoverdine was observed to be negatively controlled by iron at a physiological level, suggesting that the genes required for pyoverdine biosynthesis might be subject to iron-regulation.

The aim of my project was to isolate and characterise pyoverdine-biosynthesis genes from *P. aeruginosa* and to then determine the mechanism by which the expression of these genes is regulated.

A 16kb region of DNA that contained genes required for the pyoverdine was cloned. Regulation of these genes was examined by isolating RNA from *P. aeruginosa* grown in high and low-iron concentrations.

Northern hybridization analysis showed that the cloned DNA coded for five messenger RNA (mRNA) species that were negatively regulated by iron. To determine the mechanism which might be involved in transcriptional regulation, four promoters were identified and isolated from the cloned DNA. The activity of these promoters was shown to be negatively regulated by iron. The DNA of two of these promoters was sequenced and the mRNA start sites were determined.

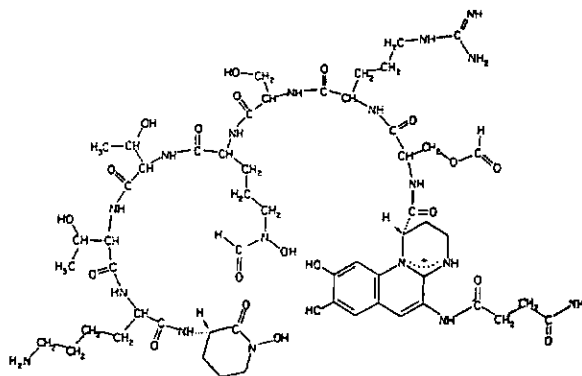


Figure 1: Structure of pyoverdine

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# BRANCH NEWS

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## MANAWATU

Congratulations to Dr Kevin Marshall, who has just taken office as the new Chief Executive of the NZ Dairy Research Institute. Kevin has been an Institute Member since 1966.

The Branch has been particularly active over the past two months in the area of chemistry awareness and education. Chemistry week in the Manawatu was marked by a public chemistry quiz in the local newspaper (developed and marked by local committee members, special thanks to David Officer). The week also included a titration competition for secondary school students held at Massey University Chemistry Department. 63 Students took part. As a follow-up, a chemistry quiz has been held in local schools in September.

Sunday 20th September was careers expo in Palmerston North, where the Manawatu branch put on a stand with an impressive set of displays, including molecular models of allomorphs of carbon and the protein azurin, computer and video displays, and a demonstration of the every popular Belousov-Zhabotinsky reaction.

Branch meetings have included a joint meeting with the NZ Institute of Food Science and Technology in July to hear Dr Owen Fennema, a visiting expert and author of a major textbook in the area of food chemistry. The joint meeting was such a success that we are having another joint meeting in September, this time to address the weighty topic of flavours and packaging of wine.

A short biography of Dr Kevin Marshall, new Chief Executive of the New Zealand Dairy Research Institute follows.

## BIOGRAPHY

### KEVIN MARSHALL

Dr K R Marshall graduated from the Chemical Engineering Department of the University of Canterbury in 1963. He was employed as a Research Officer in the Engineering Department on the New Zealand Dairy Research Institute, and granted an Institute Fellowship to study biotechnology at Birmingham University, England.

Dr Marshall returned to the Whey Products Section of the Institute. He was involved in research on fermentation of whey and the earlier research on the then new technology of ultrafiltration.

During this time Dr Marshall completed his PhD at Massey, researching the production of lactic acid from whey.

Dr Marshall served as Head of the Whey Products Section of the Institute and, later, of the newly-formed Effluent Technology Section. After a period as Assistant Director of the Institute, Dr Marshall moved to the New Zealand Dairy Board in 1983, initially as Executive Manager, Technical and, for the last two years as the Board's Corporate R & D Manager.

In his role as Corporate R & D Manager Dr Marshall has had an active role in the establishment and operations of the industry's R & D Policy Board. As part of his R & D coordination role, Dr Marshall has had a major involvement with the industry's R & D centres overseas and in dairy companies, as well as with the Institute.

Dr Marshall and his wife, Julie, have five adult daughters. Dr Marshall took office as Chief Executive of New Zealand Dairy Research Institute at the beginning of August 1992.

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## NEW SCIENCE QUALIFICATIONS LAUNCHED

### ADVANCED CERTIFICATE IN APPLIED SCIENCE

### DIPLOMA IN APPLIED SCIENCE

October 1 saw the launch of two new qualifications in the science field at Auckland Institute of Technology. At a public meeting bringing together science teachers and employers the Auckland Institute of Technology announced that it would commence offering two new programmes in the science area.

These programmes are the Advanced Certificate in Applied Science and the Diploma in Applied Science. These programmes will gradually replace the New Zealand Certificate in Science at Auckland Institute of Technology.

### DESCRIPTION

The new programmes form a staircase of qualifications which are intended will lead to a degree. This staircase is shown in figure one. The advanced certificate represents two years full time study after sixth form or one years full time study after a successful seventh form. The diploma programme follows after the advanced certificate and should take another year of full time study to complete. Approval and accreditation is awaited for the degree programme which will represent another year of full time study.

### FLEXIBILITY - THE KEYNOTE

These new qualifications have been designed to give the maximum flexibility to students and employers in all aspects of the programme - mode of study, content, entry points and exit points.

### PART TIME STUDY

All of the above programmes will be available for part time study. The Department of Applied Science has a firm commitment to the concept of part time study alongside relevant employment as the best format for vocational education. The creation of these full time programmes has become necessary as students have had increasing

difficulty in finding employment while studying. Under the NZCS programme the student could not receive their qualification without having completed three years relevant work experience which meant for some students took five years to finish. Students will be able to receive the new qualifications without being employed. However, credit will be given for work experience to encourage them to find employment in industry. It is anticipated that students will commence their studies full time and transfer to part time after gaining either the Advanced certificate or Diploma.

## MODULAR FORMAT

The course material has been presented in a modular format. This will allow student who wish to pursue unusual avenues of study to follow their interest. It will also allow employers who want their employees to study specific areas to tailor the program to their needs. All the material in the existing New Zealand Certificate is in the new programme but now students and employers will be able to combine it in ways that suit their industry. For students without a clear direction, pathways through the modules are recommended as leading to employment in specific areas.

## ENTRY POINTS

With increasing numbers of students staying at school for a seventh form year there has been an increase in the variation in achievement levels of students entering tertiary study. Combining this with the three years study right provisions it becomes imperative that students get full recognition for their achievement at school when entering a new programme. The Advanced Certificate can be entered at the first or second year level in all or some of the subjects depending on the students performance in bursary examinations. A pass of 50% or better in a subject allows a start at the second year level. Thus a student with bursary passes in sciences should be able to complete the degree programme in their three years of study right provision.

## EXIT POINTS

Many students find that they embark on a qualifications only to find that it is beyond their capabilities while many potential students view a three year programme as too daunting for them. The new programme allows students to leave the programme at a variety of levels. The student who finds the going too tough can leave with an Advanced Certificate or Diploma. Thus they will get recognition of their achievements and leave with a qualification that they can show to prospective employers. The students who are unsure of their ability can start on the Advanced Certificate programme and if they find they can perform well they can advance to a Diploma and then to a Degree

without having to "backtrack".

It is also anticipated that some employers will find the lower level qualifications well suited to employment positions requiring less highly trained personnel.

## RECOGNITION OF OTHER QUALIFICATIONS

The new programmes have provision for recognition of other qualifications. A large number of cross crediting provisions have been devised. The most important is that for holders of the New Zealand Certificate in Science. This will cross credit into the new programmes half way through the Diploma year. Thus an NZCS holder will have one year of part time study to gain a Diploma and three years of part time study to gain a Degree. Other qualifications on top of the NZCS will usually count as well.

## COMMUNICATIONS and MATHEMATICS

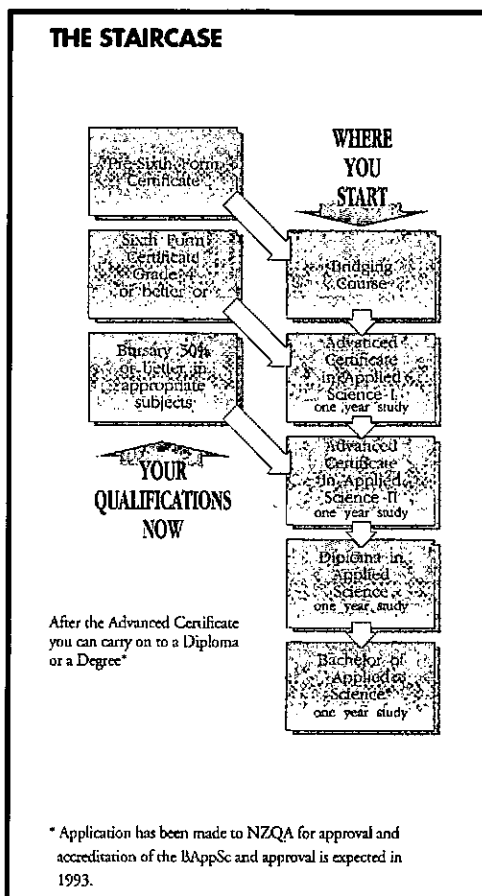
In line with recent surveys employers the new programmes include an increased amount of communications training. This is aimed at communication in a manufacturing and scientific environment and should lead to improved performance in the field of report writing and presentations. Thus certain modules of communications are compulsory at each level of the programme.

There is also and increased emphasis on mathematics and computer literacy in the new programmes as these are areas employers have highlighted as

being important in new employees. Thus mathematics and statistics modules are compulsory at different levels of the programme.

## THE FUTURE

The provision of new, more flexible programmes will allow students and employers to develop the skill base needed to support New Zealand Industry in the 21st Century.



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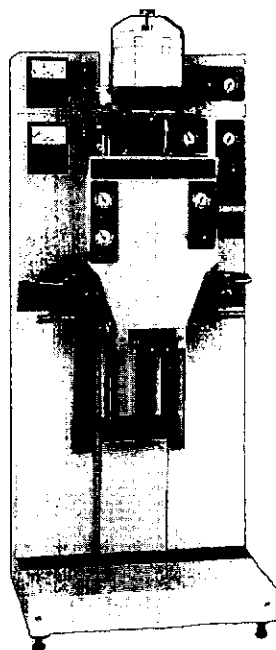
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DSC 111.. A unique transducer, Remarkable performances. The DSC measurement shows at what temperature a sample is transformed or reacts, what quantity of heat is linked to this transformation, if the reaction is exothermic or endothermic.. The DSC 111 provides, a unique transducer, a high measuring sensitivity, a very wide choice of experimental variety of measurements, exciting coupling facilities.

For control and analysis. The multimodule 92, Thermal Analysis system is an expanding assembly built around the controller CS 92, piloted by the computer PC 92. Different modules can be connected: DSC 92(-140°C tp 550°C). DTA 92(20°C to 1750°C). TGA 92 (20°C to 1750°C) with DTA option. TGA 92.. Up to 1750°C with DTA option. The TGA 92 is made up of

a robust and very sensitive microbalance, a furnace and atmosphere and vacuum control modules for working with inert or reactive gases or a vacuum. Coupling with a gas analyzer (GC,MS) is easy to set up. A DTA rod (identical to that in the DTA 92) can be hung from the balance so as to combine mass variation and thermal effect on the same sample. Applications of the TGA 92 and TG-DTA 92. Dehydration, decomposition of minerals, ores, inorganic products. Oxidation and combustion of organic products and fuels. Distinguishing polymers by degradation. Investigating ceramics.

**For further information contact: ICI Instruments PO Box 68-330, Newton, Auckland 1. Telephone 0-9-373 5765 Facsimilie 0-9-360 0683 Mobile 025-920 833**

## Cahn Thermal Systems The Standard for precision Gravimetric Analysis

### From John Morris Scientific

Superior sealing for an oxygen-free environment and improved reproductibility of studies with polymeric materials.

Corrosive resistant chamber for protective coating studies on turbine blades and other metallic components.

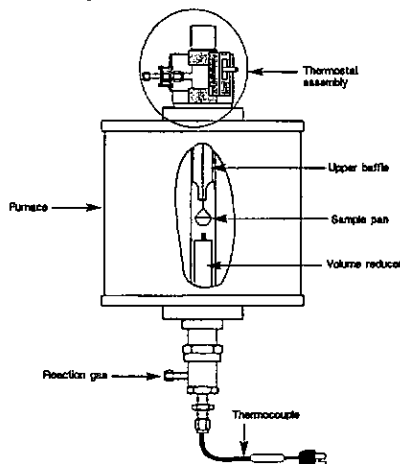
A range of 0.1 micrograms to 100 grams covers everything from resin powders to whole components (composite sections, etc).

Large chamber for meaningful sample size (steel plates etc.) Chamber is large enough for ancillary equipment that would be needed in diamond film CVD studies. Check out other TG models: 131 is ideal for interfacing with mass spec; 121 is ultra sensitive; 151 handles pressure to 1500 psi; handles high vacuum, corrosive gases and temperature to 1700°C. Greater software capability provides flexibility to customise experiments and manipulate data. Easy step up, run analysis and output modes. 60 ramp and isotherm segments possible over a 48 hour period.

### TG-FTIR Accessory

The TG-FTIR Accessory heats the evolved products from a thermogravimetric experiment to a controlled temperature before they exit to the FTIR transfer line. This temperature is maintained by two electric heaters with a thermostat that has a range from 40°C to 260°C. The heaters are designed for either 110 or 220 volt operation. A specially designed mounting bracket provides a rigid but adjustable support for the assembly. A graphic ferrule for connection to the transfer line provides a high temperature seal with flexibility. A quartz volume reducer and quartz baffle minimizes the volume in the reactor tube available for the evolved products from the TG experiment. These components assure continuous removal of the evolved products from the reaction zone. Tars from coals or oligomers from polymers are not deposited on the walls of the reactor tubes or anywhere in the apparatus. Specifically, no deposits occur on the extension wire from the balance. The design of the baffle provides a constant sweep of the extension wire with purge gas from the balance chamber. A small diameter

version is available for the TG-121 and a larger version for the TG-131. Each version is compatible with either the TG-121 or TG-131. For maximum effectiveness, gas separation practices should be followed.



View of the TG-FTIR Accessory Installed in Furnace of TG.

**John Morris Scientific Ltd.**  
Auckland PO Box 6348 Wellesley Street Auckland

Phone (09) 444-5836 Fax (09) 444-0974  
Toll Free 0800-651700  
Wellington (04) 528-7600  
Fax (04) 286-704  
Christchurch (03) 653- 825  
Fax (03) 666-975

## Thermal Analysis /FT-IR

Bio-Rad Laboratories integrated thermal analysis and FT-IR instrumentation so that both thermal and spectroscopic data are acquired, displayed and archived by the same data system.

### Thermogravimetric/FT-IR

In this configuration, purge gas sweeps the combustion and/or decomposition gases evolving from the sample through a heated gas cell. Infra-red radiation from the FT-IR spectrometer passes through the heated gas cell and is focused onto a high-sensitivity detector. The addition of the FT-IR facilitates the identity of the evolved gases and aids investigations into mechanism of combustion and/or thermal decomposition.

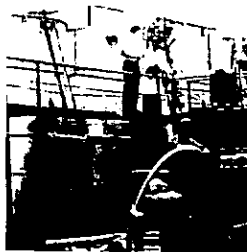
As examples of the power of this technique, TGA/FT-IR has been used successfully to identify the pyrolysis products of a number of polymers including PTFE, polybutadiene, silicone rubber, polyvinyl acetate and epoxy resins.

### Differential Scanning Calorimetry/FT-IR

In this configuration, the DSC cell is placed on the stage of an FT-IR

# HAZARDOUS AND LIQUID WASTE MANAGEMENT

*Are you satisfied with your company's waste disposal practices?*



**M**anaging hazardous waste is an increasingly difficult and complex challenge and is a continuous process of responding with technology to the challenges posed by technology.

In addition to our New Zealand services to manage solid waste, Waste Management NZ Ltd has since 1985 operated a hazardous/liquid waste treatment facility in Manukau City.

Waste Management NZ Ltd is committed to the complex, on-going process of challenge and response necessary to ensure the safe and effective management of hazardous wastes.

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- Waste removal and transport
- Emergency spillage response
- Consultancy
- Waste treatment and disposal
- Sample retrieval and analysis
- Recycling



**WM Waste Management N.Z. Ltd.**



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- Box 356, Rotorua, Ph: 073 490 222. Fax: 073 461 659

Name \_\_\_\_\_  
Address \_\_\_\_\_  
Ph: \_\_\_\_\_

microscope operated in reflectance mode. Infrared radiation from the FT-IR is focused by the microscope onto the sample and the reflected radiation is focused onto a high-sensitivity detector. Thus, the FT-IR is used to monitor structural and/or chemical changes in the sample as it passes through various thermal transition states.

Combined DSC/FT-IR has been used to monitor structural changes in a number of polymers including PET, polypropylene, polyethylene, poly (ethylene vinyl alcohol) and a number of epoxy resins.

**Simultaneous Thermal Analysis/FT-IR** Recently, Bio-Rad Laboratories announced the interfacing of an FT-IR to thermal analysis instrumentation capable of simultaneous TGA/DSC. This will allow correlation of evolved gas identification and accompanying structural changes on the same sample. As thermal analysis becomes more and more sophisticated, the power and sensitivity of FT-IR will be used more and more to probe the chemical and structural changes associated with combustion and thermal degradation.

**For further information contact Bio-Rad Laboratories New Zealand headquarters in Auckland.**

## THERMAL ANALYSERS

### PL-DSC

Integral cooling chamber with programmed cooling - ideal for studying reversible changes such as melting-crystallisation, and for quench cooling to increase sample throughput.

\* Rugged, corrosion-resistant cell - for prolonged life time and money savings. The user replaceable cell can be handled without a service call.

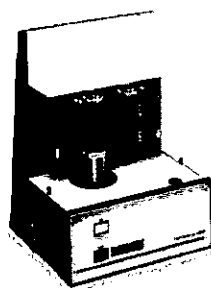


- \* Positive pan placement - means increased reproducibility from sample to sample.
- \* Optional data analysis programs - for kinetics, purity and heat capacity studies.

Temperature range: -160°C to 77°C  
Heating rate: Up to 100°C/min  
Cooling rate: From 500°C to -150°C in [greater than sign] 20 minutes (liq-

uid nitrogen quench) Calorimetric range: +/-100mW Noise: Typically 2uW rms Accuracy: +/-1% of enthalpy changes.

### PL-STA



Simultaneous DSC/TGA Analysers.

- \* Run TGA-DSC characterisations of materials in half the time.
- \* Identical experimental conditions for both measurements. Eliminates problems with sample inhomogeneity and removes correlation problems with separate DSC/TGA data.
- \* Simultaneity enables you to identify the exact weight at the onset of endo/exotherms - for samples that degrade, cure or contain volatiles.
- \* Accurate TGA temperature calibration using DSC temperature calibration. Designed for Evolved Gas Analysis for STA-MS or STA- FTIR work.

Temperature ranges: -125°C to 625°C (DSC/TGA) Ambient to 1000°C (DSC/TGA) Ambient to 1500°C (DSC/TGA) Ambient to 1640°C (DTA/TGA) Vacuum: Down to 10<sup>-6</sup> torr. Mass ranges: 0- 50mg, 0-100mg, 0-250mg. Tare: 50mg, 100mg, 150mg

Sci Tech is pleased to announce the installation of a Thermal Sciences Simultaneous STA 1500 and DSC into the departments of Chemistry and Geology at Auckland University. This is the first system of this type to be installed in New Zealand. Contact Professor Black or Dr Allan Easteel, Auckland University.

### PL-TGA

- \* A small swept volume furnace for precise atmospheric control and immediate response for gas switching. The high concentration of effluent gases is ideal for transfer to an FTIR for Evolved Gas Analysis.
- \* Water-cooled for greater sample throughput.

\* A wide assortment of pan styles - from traditional platinum to disposable aluminium, with unique



high volume pans for low density material.

Temperature ranges: Ambient to 1000°C Ambient to 1500°C

**For further information contact: Sci Tech, Science and Technology (NZ) Ltd.**

**Auckland 270-3332,  
Wellington 566-6096,  
Christchurch 383-1146,  
Dunedin 477-7860.**

## Modular Thermal Analysis Systems

Shimadzu Corporation has released a new range of Thermal Analysis Instrumentation through its New Zealand distributor Douglas Scientific.

Comprising individual modules, the TA-50 series includes Differential Scanning Calorimetry (DSC-50), Thermogravimetry (TG-50), Differential Thermal Analysis (DTA-50) and Thermomechanical (TMA-50) Units.

Each stand alone unit can be controlled by its own microprocessor via a keyboard and liquid crystal display. All feature fully programmable heating rates, the ability to operate in various gaseous atmospheres (and vacuum) and standard analog and RS-232C outputs. All operational functions are incorporated into a single compact instrument including detector, temperature and gas control and computer interface.

The compact design of these instruments mean that the accuracy and functionality of these systems are not limited even though the space in your lab is. The slim 173mm units house all functions required to perform the most sophisticated thermal analysis applications. Each module or group of modules can be interfaced with and IBM or compatible computer. Utilising the Shimadzu TA-50 controller and new windows based software, a powerful multitasking environment can be developed to control and process data from any one or a combination of instruments. The analysers may be operated simultaneously, collecting data in the background, while data analysis, monitoring the initiated analysers, or even word processing may be performed in the foreground. The variety of modules and software packages mean that the hardware and software configurations available are extremely wide ranging and surprisingly affordable.

Ease of operation is assured: Thermal analysis is executed and analysis parameters are entered by using the membrane keypad, which is incorporated in the front panel of each instrument. The cell cover of the DSC-50 and the furnace of the TGA-50(H), DTA-50 and TMA-50(H) are moved to operating positions with a touch of the START key. At the end of a programmed run the cooling cycle is automatically started.

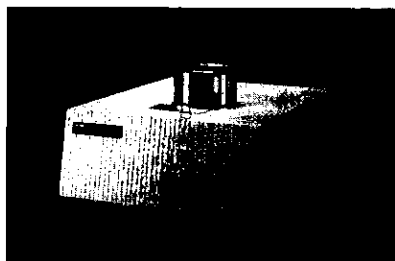
The most important factor in obtaining reliable data in thermal analysis is detector performance. Superior system control and data processing software cannot provide accurate reliable data if the system has an ordinary detector. The Shimadzu range of instruments use finely manufactured, performance proven detectors to ensure the accuracy and reliability of the data you gain from your tests.

Covered by the comprehensive Douglas Scientific warranty and factory trained service support the TG-50, DTA-50, DSC-50 and TMA-50 provide a fresh, flexible, reliable and affordable alternative in "modular" TA systems.

**For more information on the Shimadzu range of Modular Thermal analysis instruments please contact :- Douglas Scientific, PO Box 45 027, Auckland Ph: 837 5447, Fax: 837 5446, Outside Auckland freephone 0800 735 725.**

### DSC 7 Differential Scanning Calorimeter

Using a unique "power compensated" design, the DSC 7 combines unsurpassed calorimetric accuracy, precise temperature control, and the fastest heating and cooling rates in the business.



### TGA 7 Thermogravimetric Analyzer

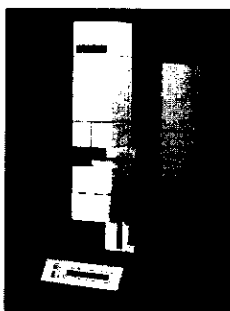
Modern ultramicrobalance design and a small low mass furnace provide superior weight change meas-



urements as a function of temperature. Automatic furnace positioning adds to its ease of use, reproducibility and safety.

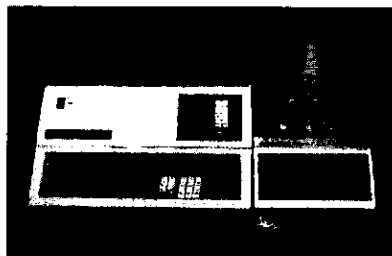
### TMA 7 Thermomechanical Analyzer

A new temperature-controlled detector allows the sensitive measurement of dimensional changes as small as a few microns. Electronic sample force loading and multiple probe types add to the versatility of the TMA 7.



### DTA 1700 High Temperature Differential Thermal Analyzer

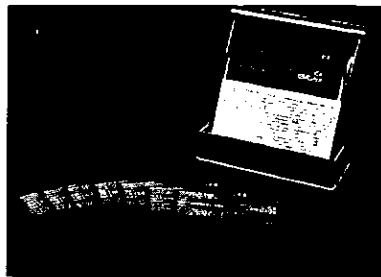
This classical DTA allows the characterization of materials to temperatures as high as 1500°C. With DTA and "heat flux" DSC modes of operation, complete high temperature characterization is possible.



### Computers and Thermal Analysis Software Programs

Data handling options range from Personal Computers to Workstations. Operating systems from DOS to IDRIS, functionally similar to the UNIXr Operating System. And dozens of standard and

advanced thermal analysis application software programs.



#### MAJOR ACCESSORIES

**DSC 7 Robotic System** - for the automated, unattended analysis of up to 48 DSC samples.

**DPA 7 Double Beam Photocalorimetric Accessory** - for the study of UV-promoted reactions by DSC.

**TG/FT-IR Accessory** - for the analysis of the gases during TGA experiment by FT-IR.

**AD-4 and AD-6 Ultramicrobalances** - for accurate, precise weighing in the milligram or microgram ranges.

**For further information contact Perkin Elmer Pty Ltd New Zealand Branch PO Box 22159 Otahuhu Auckland Ph (09) 276 2230 Fax (09) 276 5602**

*From page 78*

programme sub-committee by the middle of next year; this will give symposia organisers adequate time to set their programme of invited speakers and seek additional sponsorship. Proposals received after April 15 next year will be dealt with as speedily as possible although decisions regarding suitability etc. will be influenced by those proposals already accepted. No new proposals can be considered after January 15, 1994.

The organising committee has established a fund to sponsor "Young Scholars" to the Congress from developing countries around the Pacific Basin. The NZ delegate has been appointed convener of the selection panel.

Although four years away, Pacificchem '95 is something for you to plan for - ask anyone who was at Pacificchem '89. You will be reminded of this and updated with information through these pages as it comes to hand.

*Brian Halton, Pacificchem '95 Representative, Department of Chemistry, Victoria University, PO Box 600, Wellington.*

# BIO-RAD IS FIRST IN FT-IR

Molecular identities emerge quickly and easily with state-of-the-art FT-IR spectrometers and accessories.



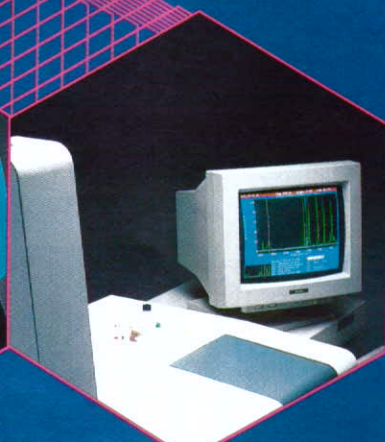
*Bio-Rad makes the FTS 60A, with the 896 interferometer, the only FT-IR with precision step scan operation, TRS and 2D IR.*



*Bio-Rad makes the Tracer, the first GC/IR with real-time picogram sensitivity.*



*Bio-Rad commercialized the FT-IR microscope, and now offers the UMA 500 research system with fingertip controls.*



*The new Bio-Rad FT-Raman spectrometer is the first to offer precision step-scan capability.*

If FT-IR spectroscopy is critical to the outcome of your research, you must evaluate Bio-Rad first as a source of versatile, high performance FT-IR spectrometers and accessories. For more information on the above instruments, call 0-9 443 3099 or Toll Free: 0800 805 500, Fax: 0-9 443 3097 or write: Bio-Rad Laboratories Pty Ltd, PO Box 100 051, Auckland 10.



*The FTS 60A with the acclaimed 896 interferometer*

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Thermal Analysis

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**WINNING HAND**

**◆ TMA-50**

Dynamic Thermomechanical Analyser

- ◆ High fidelity detection of dimensional changes in sample.
- ◆ Wide dynamic measuring range  $\pm 2,500\mu\text{m}$ .
- ◆ Differential expansion measurement is available.
- ◆ Wide load range — up to 500gf and resolution of 0.010gf.
- ◆ Stand-alone or PC controlled operation.
- ◆ Diversity of loading programs — constant load, constant rate, constant elongation and cycle load. Stress-strain and stress relaxation measurements of materials can be readily obtained.

**♠ TGA-50**

Thermogravimetric Analyser

- ♠ High sensitivity and vibration resistance ensured with low mass suspension mechanism.
- ♠ Excellent baseline stability is ensured by this suspension system.
- ♠ Measurement in a variety of gas atmospheres.
- ♠ Availability of large volume crucibles enables measurement of samples with minute weight changes.
- ♠ Stand-alone or PC control operation.
- ♠ Wide temperature range — ambient — 1000°C TGA-50 ambient — 1500°C TGA-50H

**♥ DSC-50**

Differential Scanning Calorimeter

- ♥ Excellent signal to noise performance for high sensitivity analysis.
- ♥ Wide dynamic range.
- ♥ Hi fidelity temperature controller ensures extremely reproducible temperature programs.
- ♥ Wide temperature range, ambient — 725°C with optional accessory for subambient operation.
- ♥ Your choice of stand-alone operation or multi-tasking PC control.
- ♥ Low running costs.

**♣ DTA-50**

Differential Thermal Analyser

- ♣ Quick response and High sensitivity. Achieved by a small heat capacity detector — also enabling measurement of relatively large signals.
- ♣ Accurate Temperature Control ensures smooth and rapid transition from heating/cooling to isothermal hold.
- ♣ Low volume furnace for rapid response and convenient and speedy gas purging.
- ♣ Easily accessible detector and sample setting area.
- ♣ Stand-alone or PC controlled operation is available.

Windows — PC control for 1-4 Thermal Analysers: Multi-tasking system allows simultaneous control of up to four analysers. Foreground/background operation allows reprocessing of existing data at the same time as the instrument(s) are acquiring new data. Windows platform offers an easy and intuitive user interface. Wide range of accessories for all applications including autocooling device and atmosphere control. Additional application software library to meet additional data processing needs. Local support and applications assistance — factory trained engineer.

For further information and advice contact today.

**SHIMADZU** and **DOUGLAS Scientific**

Name: \_\_\_\_\_  
 Address: \_\_\_\_\_  
 Phone: \_\_\_\_\_ Fax: \_\_\_\_\_

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