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THERMAL METHODS GROUP  
ANALYTICAL DIVISION  
ROYAL SOCIETY OF CHEMISTRY

# Tmg NEWS

*Editor: Peter J Haines, 38 Oakland Ave, Farnham, SURREY, GU9 9DX, ENGLAND*

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**NUMBER 16 SUMMER 1996**



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## EDITORIAL

It seems that this issue of *TMG NEWS* has arrived very quickly. This is probably because we have all been busy attending TAC '96 and other meetings and preparing for 11th ICTAC in Philadelphia. Of course, there was some other work to do as well!

Our cover for this issue is a portrait of a brilliant scientist who was born 200 years ago. The son of the "organiser of victory" during the French Revolution, he introduced many new ideas in thermodynamics. He died, in 1832 in a hospital for the mentally insane. His name is Nicholas Leonard Sadi Carnot, famed for the Carnot Cycle. The picture was kindly provided by the Academie des Sciences, Paris.

If you know of people new to thermal analysis, please draw their attention to the Workshop on DSC, TG and HSM and the leaflet in the centre of this issue! We hope that the Winter 1996 issue will contain reports from ICTAC and from the AGM meeting plus lots more, provided you send it by the deadline of 1st December, 1996! Have a splendid Summer!

**Peter J Haines**

## **CHAIRMAN'S NOTES**

“Yes, Editor, I have written some notes for TMG NEWS!”

It does not seem to matter today whether you are working in industry or academia. Time seems to be at a premium like never before. That is something that makes the voluntary efforts of all the Committees in the RSC so commendable. The TMG Committee is no exception and, after a year as Chairman, I would like to take this opportunity to pay tribute and thank my Committee for such hard work in making 1995-6 one of the most successful years in terms of meetings.

The AGM last November attracted close to 100 delegates. With a focus on calorimetry, many new faces, particularly from industry, heard a varied programme organised by Tony Beezer. It featured excellent plenary contributions from Erwin Marti (Ciba-Geigy, Switzerland) and Aziz Bakri (Grenoble, France).

More recently, the Spring Meeting saw the first UK Thermal Analysis and Calorimetry Symposium. Held in Leeds and organised by Ted Charsley and Steve Warnn~ton. This was a milestone for the TMG. For many years, I have been advocatinu the idea of a single thermal meeting where people in all aspects of TA could attend and be sure to find papers of interest to their field of work. This two day event attracted over 100 and covered diverse topics from polymers to pharmaceuticals. calorimeters to microscopes and more besides. Plenary contributions came from overseas with Jean Rouquerol (Lyon, France) and Ingemar Wadso (Lund, Sweden) while the UK pro~ided a further 30 oral and poster contributions. You can read more about the meeting in this issue of IMG News. Socially the meeting was a great success, a factor again made possible by the generous sponsorship from the manufacturing community.

Preparations are already under way for the TAC '97 meeting, organised in Oxford by Keyna O'Reilly from OCAMAC. It will take a step forward, in that it will be a parallel session, two day gathering. This will be made possible through combining with TAAN'I II (Thermal Analysis of Advanced Materials II). Four plenary lectures will be supplemented with speciality sessions of polymers, composites, pharmaceuticals and instrumental techniques

With a new poster competition to encourage the participation of young scientists, I hope you will put TAC 97 in your diary and join me in Oxford in April.

*Continued*

## CHAIRMANS' NOTES (continued)

Have you seen that the TMG is on the Web? The RSC runs a homepage and now the TMG, thanks to Stuart du Kamp, is there too with details of meetings. At least no one can accuse the Group of not keeping up with the times... and, yes, the rumours are true. Cyril Keattch does now own an answerphone!

1996 sees the end of an era. After many years serving on the Committee as Chairman and then as Treasurer, Jenny Hider is standing down at the AGM. With many activities higher up in the RSC, coupled with a busy work and family life, Jenny has decided to leave. On behalf of the IMG, I would like to express my thanks to Jenny and wish her all success in the future.

In conclusion, may I ask you all a question? What do **you** think of the IMG? Does it meet your requirements? Do you like the meetings. or should we be doing things differently? The Committee is elected to serve you and your requirements in thermal methods. If you want to see change, please write to me or come along to our meetings and express your opinions. I look forward to hearing from you.

**Jezz Leckenby**

(Jezz may be contacted via TopoMetrix Corporation, 18, Hill St, Saffron Walden, Essex, CB10 1JD,  
Tel: 01799 524913; Fax: 01799 526619; E-mail: topometrix.uk @ dial.pipex.com)

## LIGHT RELIEF?

While the Nomenclature Committee of ICTAC toil away refining and defining, the Institute of Physics decided to propose a New System of Units. Full details are available from the Editor, but your attention is drawn to two units of importance to Thermal Analysts:

**Mass:** measured in tablespoons of mercury (Note: 1 HgTb~ 0.16 kg)

**Temperature:** measured in Kevins (1 Kev~ 47.3 K)

1 Kevin is defined as the temperature drop required to make an average inner city male teenager put on a proper coat when going down to the pub.

**Any other bright ideas?**

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**TAC 96:**  
**UK NATIONAL THERMAL ANALYSIS AND**  
**CALORIMETRY SYMPOSIUM**

TAC 96 was held at Bodington Hall, a few miles to the north of Leeds, on April 17th and 18th, and represented a new departure in British thermal analysis meetings. The aim was to provide an open forum for the presentation and discussion of current research work in all areas of thermal analysis and calorimetry. The meeting was jointly organised by the Thermal Methods Group and the Thermal Analysis Consultancy Service (TACS), Leeds Metropolitan University.

Apart from the notable exception of the recent anniversary meeting, our meetings have been addressed at specific themes, with the result that thermal analysts from different subject areas have been rather isolated from each other. Furthermore, there has been no obvious venue for those new to the field to air their work. These problems were more than adequately overcome by this meeting, which attracted around 100 delegates, with a good balance between industry and academic establishments, including several research students.

We were fortunate to have two excellent plenary lectures, by Dr. Jean Rouquerol (Centre de Thermodynamique et Microcalorimétrie, Marseille, France) on Controlled Transformation Rate Thermal Analysis, and Prof Ingemar Wadso (Chemical Centre, University of Lund, Sweden) on Isothermal Microcalorimetry. The rest of the programme comprised 22 oral contributions and 22 posters. The recently developed technique of Modulated Temperature DSC was well represented, with six oral and one poster presentations, showing the interest that this new method has stimulated. In addition to our plenary lecturers from overseas, there were also delegates from Denmark, Holland, India and the USA.

Selected papers from the symposium are due to be published in a special edition of *Thermochimica Acta*, which will be edited by Prof Ted Charsley (TACS), and Dr. Peter Laye (School of Chemistry, Leeds University).

*continued*

## **TAC '96 continued**

During the informal dinner, the winner of the TMG Award was announced as Gary Foster of Birkbeck College, University of London, for his work on the use of *in situ* dielectric analysis for monitoring the moisture content of materials.

The meeting was well supported by the manufacturers of thermal analysis and calorimetry equipment, who displayed their latest instruments; those present were The Laboratory of the Government Chemist, Linkam Scientific, Mettler-Toledo, Netzsch Instruments, Novocontrol International, PETA Solutions, Rheometric Scientific, Setaram, V.A.Howe & Co., TA Instruments and Thermometric Ltd. The delegates seemed particularly grateful (some more than others!) for the generous sponsoring by the manufacturers of a wine reception during the exhibition.

The success of the meeting should mean that the concept of an annual symposium is now firmly established. The next, TAC 97, is to be organised by the IMG and the Oxford Centre for Advanced Materials and Composites (OCAMAC), and will, we hope, build upon a highly encouraging start.

**S.B.Warrington**

## **TMG AWARD FOR 11TH ICTAC**

The 1996 IMG Award for the best essay on a topic in thermal analysis or calorimetry by a British scientist under the age of 35 has been awarded to **Gary M Foster** for his essay on **“Simultaneous non-invasive microwave dielectric spectroscopy and dynamic mechanical analysis for studying drying processes in complex heterogeneous materials”**

The essay will **be presented** at 11th **ICTAC in Philadelphia.. Gary is a research assistant in the** Chemistry Department at Birkbeck College, London A profile of Gary and an abstract of his essay will appear **in a later issue of IMG NEWS.**

**Many congratulations, Gary!**

# TAC 96

## INDEX OF PAPERS CONTRIBUTED.

**NOTE: Abstracts are only** given for the Plenary Lectures. Fuller papers will be published in a **special edition** of Thermochimica Acta later this year.

### **Plenary Lecture 1:** Dr J.Rouquerol, (CTM, Marseille, France)

**An Overview of Controlled Transformation Rate Thermal Analysis Today:**  
Multiple Definitions, Forms, Applications and Expectations. page 12

### **Plenary Lecture 2:** Prof.I.Wadsö, (Chemical Center, University of Lund, Sweden)

Isothermal Microcalorimetry. Overview and Discussion page 13

### **Other Oral Presentations** (Speaker underlined)

B.Cassel, M.Divito, K.Fielder & S.Goodkowsky.

Investigation of Low Energy, Low Temperature Polymer Transitions using the New Pyris 1 DSC

M.W.Lewitt Proton Magnetic Resonance Thermal Analysis: A Users Overview.

A.Hammiche, H.M.Pollock, J.N.Leckenby, M.Song, M.Reading & D.J.Hourston.

Scanning Thermal Microscopy of Polymer Blends: Phase Separation, Localised Calorimetric Analysis.

M.Odlyha, G.Foster & T.Y.A.Chan

In Situ Non-Invasive Dielectric Spectroscopy for Monitoring Moisture Content of Materials

L.Benoist Heat of Evaporation & Sublimation with the TG-DSC III

G.M.B.Parkes, P.A.Barnes, E.L.Charsley,

Recent Advances in Rate Controlled Methods of Materials Characterisation.

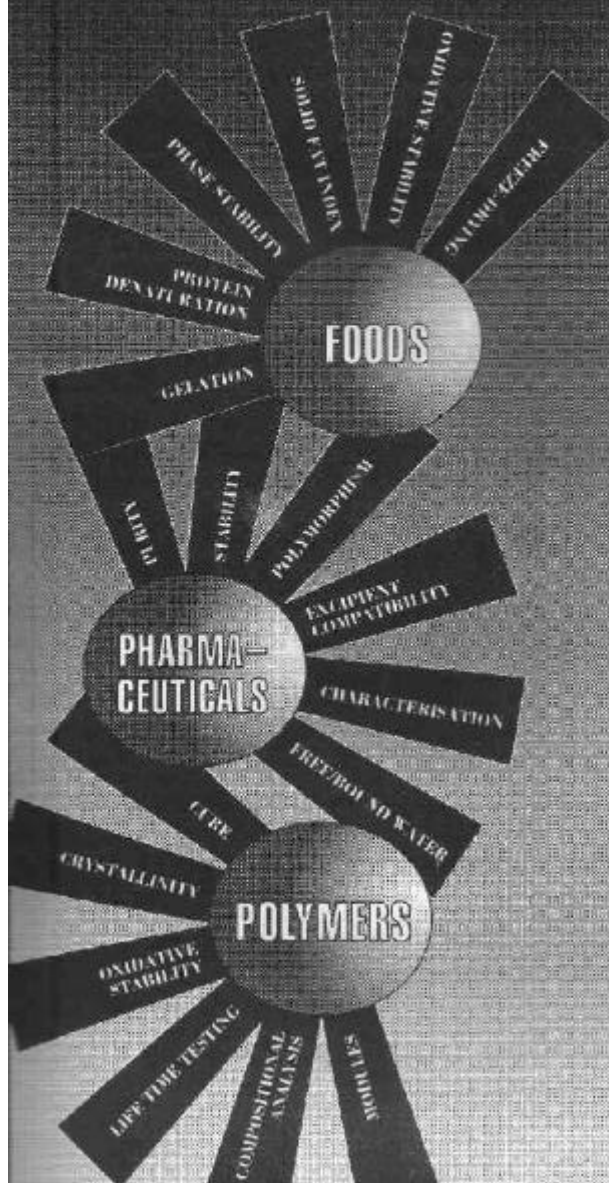
C.M.Allen, K.A.Q.O'Reilly, P.V.Evans & B.A.Cantor

Differential Scanning Calorimetry of Al-Fe-Si Alloys

A.P.Day, A.T.Dinsdale, D.M.Hayes & M. J.Richardson

Enthalpy Changes in Alloy Systems

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E.L.Charsley Thermomicroscopy of Bismuth Oxide .A Video Shown in Memory of Arthur Brammer, Senior Experimental Officer, TACS, Leeds Metropolitan University

M.Reading Modulated Temperature Differential Scanning Calorimetry: A Brief Review.

R.A.Bottom, J.K.Arthur, G.Widman  
Flexible Temperature Programming Alternating DSC

T.J.Lever & M.Chahinian  
Improved Characterisation and Understanding of the Glass Transition by Modulated DSC.

M.Song, DJ.Hourston, H.M.Pollock & A.Harnmiche Applications of Modulated-Temperature DSC to Heterogeneous Polymer Systems.

D.Q.M. Craig  
The Study of Pharmaceutical Systems using Modulated DSC.

K.A.Q.O'Reilly & B.Cantor           Cyclic DSC of Metals

V.Zholobenko, A.Garforth & J.Dwyer  
TGA-DTA Study on Calcination of Zeolitic Catalysts

F.Biddlestone & J.N.Hay:           Ageing Effects in Crystalline Polymers

D.J. H.Edwards  
The Application of Thermal Analysis to the Evaluation of Polymeric Packing Materials

A.A.Garforth, S.G.Fiddy, A.Ghanbari-Siakhali, Y-H.Lin, P.N.Sharratt & J.Dv~yer  
Catalytic Degradation of High Density Polyethylene: An Evaluation of Mesoporous and Microporous Catalysts using Thermal Analysis.

D.M.Price Thermal Analysis of Fibres and Films in Solvents and Vapours.

B K Kandola S Horrocks and A R Horrocks  
Evidence of Interaction in Flame-Retardant Fibre-Intumescent Combinations by Thermal Analytical Techniques

G.Foster & M.Odlyha  
Dynamic Mechanical Analysis for the Evaluation of the Deacidification Treatment of Cellulosic Materials

## POSTER PRESENTATIONS

.A.Cavicchioli, M.Odlyha, G.Foster, J.M.Slater & M.Appleton Novel Piezoelectric Chemical Sensors for Monitoring Indoor Environments.

D.M.Price & S.P.Church

FTIR Evolved Gas Analysis of the Decomposition products of cellulose diacetate and Clarifoil.

G.Bhaskar, J.L.Ford & D. A.Hollingsbee

Comparison of Water Uptakes of Hydrocolloids determined by DSC.

C.O.Giwa & S.Morris

Thermal Behaviour of Selected Cathode Materials in Molten Nitrate Electrolytes.

D.M.R.Georget, M.Guardo, A.Ng, A.C.Smith & K.W.Waldron

Mechanical and Thermal Analysis of Raisin Components.

S.M.Deshpande, B.K.Patil

Thermal Analysis of some Lanthanide Chelates of 2-(2'-Hydroxy-3'methoxy phenyl) 6-methylbenzothiazolyl hydrazone.

.M. Song, D.J.Hourston, H.M.Pollock & A.Hammiche.

Macromolecular Diffusion and Interface Development between two Compatible Polymers Studied by Modulated-Temperature DSC.

C.N.Rhodes & D.R.Brown

Surface Acidity Measurements on Solid Acid Catalysts using Simultaneous TG-DSC and Ammonia Adsorption.

.C.B.McCrystal, J.L.Ford & A.R.Rajabi-Siahboomi

A Study of the Interaction of Water and Cellulose Ethers using DSC.

J.A.Turner & K.M.Thomas

The Application of Simultaneous Thermal Analysis to the Determination of the Fate of Injected Coal in a Pilot Scale Blast Furnace Simulation Rig.

**POSTER PRESENTATIONS..., *continued***

D.D. Shepard, K.R. Smith, P.V.Gabbott, P.E.Clarke

A New Ultrasonic Measurement System for the Cure Monitoring of Thermosetting Resins and Composites.

G.M.Bell, J.Ferrara, J.Joannou, P.Newbatt

Effects of Stress on the Thermal Transitions of Foods using Differential Thermal Rheometry.

A.G.Coutts

Investigation of the Unusual Crystal Properties of R.PR *111905* by **DSC**.

Y-H.Lin, P.N.Sharratt, A.Garforth & J.Dwyer

Investigation of the Deactivation of Ultrastable Y Zeolite by Coke Formation during the Catalytic Degradation of High Density Polyethylene.

S.Holmes, A.A.Garforth, B.M.Maunders & J.Dwyer

Pyrolysis GC-MS Study of Zeolitic Coke.

M.J.Jenkins & J.N.Hay

Enthalpic Relaxation as a Model for the Glass Transition.

S.Tamburic & D Q.M.Craig

Thermorheological Analysis of Mucin Gels and Polymer/Mucin Mixtures.

D Q M Craig, A Kee, S.Tamburic & D.Barnes

A Thermal Investigation into the Rheological Synergy between Xanthan and Locust Bean Gum.

M.Gogebakan, P.Warren, I.T.H.Chang & B.Cantor

Crystallisation Studies of Amorphous AlYNi Alloys.

R.P.Tye

AC Calorimetry Methods Applied to Thermoanalysis Applications.

A.Charlton & J.J.Macnab

Isoperibol Calorimetry at Low and High Temperatures.

R.F.Callahan, M.Gallop, P. V.Gabbott & P.E.Clarke

Characterisation of Molecular Mobility Using the Thermally Stimulated Current Technique.

AN OVERVIEW OF CRTA TO-DAY:  
MULTIPLE DEFINITIONS, FORMS, APPLICATIONS AND EXPECTATIONS

J.Rouquerol

Centre de Thermodynamique et de Microcalorimétrie dii CNRS,  
26 rue dii I4lème R.I.A., 13003 Marseille, France

The concept of Controlled transformation Rate Thermal Analysis (CRTA) was progressively built and enriched as the needs, the technical possibilities and the field of application were increasing [1]. The basic idea was always to **control the heating of the sample after its own reaction to heating**. This feed-back can be detected with the help of any appropriate physical property of the reactant (the ‘sample’) or product: mass, volume, amount of heat or gas exchanged etc... CRTA was first applied to thermal decompositions, under ~vacuum” and then extended to other types of transformation and atmosphere (inert or reactive gas). Also, it was first used with the simplifying condition of a constant rate of transformation, and then extended to the case of any pre-determined transformation programme. Finally, after being used with a close, direct control of the transformation rate after the selected physical property, it was then extended to the case of a “loose” control also involving the temperature [2,3].

Used for more than a quarter of a century, and always to describe a full method (not a simple technique), the acronym “CRTA” can cover these extensions. In case of need, a term like “Sample Controlled Thermal Analysis” (SCTA) could do also, but we must be careful before adding new terms and acronyms if not really necessary... *continued over*



Dr Rouquerol lecturing at TAC ‘96

## **Rouquerol Plenary Lecture., *continued***

All above forms of CRTA have different merits which will be considered and illustrated in view of the main expected applications : **thermal analysis proper** (requiring resolution, sensitivity and meaningful temperatures), kinetics (requiring accurate and meaningful measurement of the rates, temperatures and energies of activation), preparation of new materials (requiring a high homogeneity and control of the temperature and partial pressures throughout the sample).

An unexpected conclusion is that we cannot say that these successive steps must be considered as real “improvements superseding the previous ones”. Instead, each of them brings to light a new aspect of CRTA and, in some respect, opens a new window in the world of Thermal Analysis.

1. J.Rouquerol, Thermochim. Acta, **144**, (1989) 209-224.
2. M.Reading in “Thermal Analysis .Techniques and Applications”, E.L.Charsley and S.B.Warrington (Eds)., R S C, (1992) 126-155.
3. P.A.Bames, GM B Parkes, D.R.Brown and E.L.Charsley,

### **ISOTHERMAL MICROCALORIMETRY. OVERVIEW AND DISCUSSION**

**Ingemar Wadso**  
**Thermochemistry, Chemical Center,**  
**University of Lund, P0 Box 124, S-22 100 Lund, Sweden**

Isothermal (non-scanning) microcalorimeters are employed both in thermo-dynamic work and as analytical tools, in the latter respect usually as “process monitors”. They are normally of the thermopile heat conduction type and are typically used in the temperature range 20-80°C

Important thermodynamic applications include ligand binding studies by use of titration techniques, for example in pure and applied biochemistry. Investigations of sorption of solutes and of vapours on solids have proved to be of significant fundamental and technical importance like measurements of dissolution and transfer enthalpies of slightly soluble compounds.

*...continued over*

## **Wadso Plenary Lecture ...continued**

Many isothermal microcalorimeters are currently used for the characterization of the long term stability of technical materials and products such as explosives and pharmaceutical compounds and of (after-) curing processes. The thermal power sensitivity of isothermal microcalorimeters is orders of magnitude higher than for conventional DSC instruments operated in an isothermal mode. This implies that degradation processes can be studied at much lower temperatures by use of isothermal microcalorimeters than with DSC. Typically, degradation rates in the order of one percent per year can be determined.

Much method development work and fundamental investigations have been reported for living cellular systems: microorganisms, animal and human cells and tissues, small animals and plant tissues. Potential practical applications are envisioned for areas like pharmacology and ecology.

Non-specific methods like calorimetry are attractive from the point of view of their broad application range and they are particularly well suited for the discovery and quantitative assessment of unexpected or unknown phenomena. However, it is often felt as a serious shortcoming that calorimetric techniques are unable to identify the processes. It is therefore desirable to combine (micro)calorimetry with specific sensors (electrodes) in the reaction vessel, which are used in parallel with the calorimetric measurements. Alternatively, the calorimeter can be coupled to external specific analytical instruments. It is judged that significant developments of such combined techniques will take place.



**Professor Wadso lectures at TAC '96**

## **HAPPY MEMORIES OF TAC'96**



**From left to right: Jezz Leckenby, Chairman, TMG; Prof. Ted Charsley, TACS; Prof. Ingemar Wadso and Dr Jean Rouquerol, the Plenary Lecturers and Steve Warrington of TACS and Vice Chairman, TMG**



**Dr Gurli Mogenson from Denmark (left) with Fiona Power of Linkam Scientific Instruments at the TAC 96 Exhibition.**

**THE ROYAL SOCIETY OF CHEMISTRY**

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**THERMOMICROSCOPY**

on

Wednesday 11 September, 1996

at

Kingston University, Surrey

from

9 am. until 6 p.m.

*Kingston University*

# THERMAL ANALYSIS WORKSHOP

Thermal analysis is a group of techniques where the changes in materials as they are heated are studied. It is widely used in analytical laboratories, in quality control and research into polymers, pharmaceuticals, minerals and special organics.

**This workshop will be a “hands on” day, providing information and training in the theory, instrumentation and applications of three major techniques:**

*Differential Scanning calorimetry (DSC)*

*Thermogravimetry (TG or TGA) and*

*Thermomicroscopy (HSM)*

## **Who should attend?**

The workshop will be tailored principally to the needs of **people new to thermal methods**, especially technical staff, analysts, chemists and material scientists and **graduate researchers** who wish to know what may be done by thermal methods. For **existing users**, it would be suitable as a refresher course and for updating.

## **How many will be on the course?**

Numbers will be **strictly limited** in order that every participant can take an active part! The speakers and demonstrators are expert practitioners in thermal methods from both manufacturers and users. The lectures will give an overview of the instrumentation, operation and applications of each method, and will be supplemented by extensive illustrated course notes and by informal discussions. The practical sessions will illustrate typical experiments on materials such as polymers, pharmaceuticals and give an “appetiser” to the range of thermal methods.

The workshop will be supported by Instrument manufacturers including:

*Linkam Scientific Instruments*

*TA Instruments Ltd*

*Mettler-Toledo Ltd*

*Perkin-Elmer Ltd*

*Netzsch Mastermix Ltd*

*Rheometric Scientaia Ltd*

and a wide range of literature and publications will be available.

## **PROVISIONAL PROGRAMME**

- 8.30-9.15 Registration and Coffee
- 9.15-9.45 Introduction to Thermal Methods  
Dr M. Odlyha, Birkbeck College, London
- 9.45-10.30 Thermogravimetry  
Dr S.Warrington, TACS, Leeds
- 10.30 - 10.45 Coffee Break
- 10.45- 11.30 Differential Scanning Calorimetry  
Dr S.Woodisse, Brunel University
- 11.30- 12.00 Thermomicroscopy  
P Haines, Kingston University
- 12.0- 1.15 Lunch
- 1.15 - 6.00 Practical Sessions with hands-on experiments in all the above methods.
- 

Dr C J Keatch, Hon. Sec, TMG  
P.O.Box 9,  
LYME REGIS  
Dorset.  
DT7 3BT

## **THERMAL ANALYSIS WORKSHOP**

Kingston University, Penrhyn Road, KINGSTON, KT 1 2EE  
Wednesday, 11th September, 1996, 9 am. - 6 pm.

### **COURSE FEE:**

**£100.00**

To include all course material, lunch, tea and coffee and participation in all experimental sessions **PLUS** 1 year's membership of the TMG

For recognised students the TMG offers a reduced fee of £70.00

Fees do not include accommodation which is available at hotels close to the University from about £42 per night.

### **Special Needs**

The course organisers will do their best to provide for any participant with special dietary, transport or other needs. Please tell us if you wish to attend and require special facilities.

## **REGISTRATION FORM**

Please return to **Dr C.J.Keattch. by 8th August 1996.**

Please register me for the **Thermal Methods Workshop** at Kingston on **14th September 1996. I** enclose registration fee of £  
(Please note that proof of student status **is required** to qualify for reduced fee)  
Please send details of accommodation (delete as appropriate)

**Title**

**Initials**

**Surname**

**Organisation**

**Address**

**Phone no.**

**Fax**

## **“MICRO’96”**

2nd to 4th July, 1996 at the Novotel, Hammersmith, London

Details from the Royal Microscopical Society,

37/38 St Clements Rd, Oxford, OX4 1AJ, Tel 01865 248768

## **“12th EUROPEAN SYMPOSIUM ON POLYMER SPECTROSCOPY (ESOPS 12)”**

8th-10th July, 1996 LYON, FRANCE.

Besides the IR, Raman, NMR and MS there will be sections on Electrical and Mechanical spectroscopy.

For more details contact: G Lachenal, Université Lyon 1, Laboratoire des Matériaux Plastiques et Biomatériaux 43 Bd du 11 Novembre, 69622, VILLEURBANNE CEDEX FRANCE, Tel: 33-72-43-12-1 1 Fax: 33-72-43-12-49

## **“RESEARCH AND DEVELOPMENT TOPICS IN ANALYTICAL CHEMISTRY”**

22nd and 23rd July, 1996, Nottingham Trent University

Details from The Secretary, Analytical Division, R&D Topics Conference, RSC, Burlington House, LONDON W1V 0BN. Tel: 0171 437 8656

### **VERY ADVANCE NOTICE!**

## **“ESTAC 7”**

The 7th European Symposium for Thermal Analysis and Calorimetry

31st August to 5th September, **1998**

Details from Prof G Liptay, Institute for General and Analytical Chemistry,

Technical University, H 1521 Budapest, Hungary

## **KINGSTON UNIVERSITY APPLIED CHEMISTRY OPEN DAY**

There will be a programme of talks and discussions at KINGSTON UNIVERSITY'S Penrhyn Road Centre, Kingston upon Thames, on **Wednesday** 18 September 1996. from 9.00 am to 4.00 pm. This event is free of charge and will provide interested organisations with a chance to meet some of the academic, consultancy and technical staff of Kingston University's School of Applied Chemistry. You will find out about the School's excellent track record of working with industry through its research and consultancy programmes, and how the extensive laboratory facilities can be made available to help your business, through Kingston Analytical Services (KAS) and the Kingston Polymer and Composites Consultancy (**KPCC**).

Anyone who attends this event can claim a 50% discount (of up to £200) on the next service they require from either KAS or KPCC following the event. A buffet lunch will be provided on the day. Places are limited and are allocated on a first-come-first-served basis. If you would like to book a place, please contact Simon Feegrade at Kingston University Enterprises Limited: Tel: 0181-547 7273; Fax: 0181-974 8035, e-mail s.feegrade@Kingston.ac.uk KUEL. Millennium House, 21 Eden Street, Kingston upon Thames, Surrey KT 11 BL.

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# **AFCAT-GEFTA SYMPOSIUM FREIBURG '96**

**Symposium on Thermal Analysis and Calorimetry**

**24th to 26th September, 1996**

**Albert-Ludwigs-Universität, Freiburg, GERMANY.**

The scientific program is intended to have a broad, general character to interest scientists from Universities and Industries working with Thermal Analysis and Calorimetry

Details from: GEFTA Office, Prof Dr V. Kramer,

Kristallographisches Institut der Universität Freiburg,

Hebelstr. 25, D-79 104 Freiburg, GERMANY

Tel: 49 761/203 6436: Fax: 49 761/203 6434

e-mail: kraemer@rufuni~freiburg. de

## **“ABC '96”**

**INTERNATIONAL MEETING ON**

**THE APPLICATIONS OF BIOCALORIMETRY**

(DIFFERENTIAL SCANNING CALORIMETER)

(ISOTHERMAL TITRATION CALORIMETER)

30 th September to 1st October, 1996

St Anne's College, Oxford, England.

Sponsored by Heath Scientific Co. Ltd. and MicroCal Inc.

For details contact: Phill OKane, at Heath Scientific, 01908 646700

THE ANNUAL GENERAL AND SCIENTIFIC MEETING OF THE TMG:

**“THERMOMECHANICAL PROPERTIES OF  
POLYMERIC MATERIALS”**

A Joint Meeting of the TMG with The Structural Materials Group of the SCI  
and the Macro Group of the SCI and RSC.

13th November, 1996

SCI Lecture Theatre,

Details from : Dr C J Keatch, (see back cover)

**“OXIDATIVE BEHAVIOUR OF MATERIALS  
BY THERMOANALYTICAL TECHNIQUES”**

21st -22nd November, 1996

New Orleans, Louisiana, USA.

**A Meeting organised by ASTM Committee E-37 on Thermal Measurements**

All aspects of oxidative behaviour by thermoanalytical techniques will be examined,  
with emphasis on the use of calorimetry, DSC, PDSC, DTA, TG, DTG and DETA.

**Further Information from the Symposium Chairman:**

Dr Alan T Riga, The Lubrizol Corporation, 29400 Lakeland Boulevard, Wickliffe,  
OHIO, OH 44092, Tel: 216/943-1200 Ext 2301, Fax: 216/943-9022

# TAC 97

## THE SECOND UK NATIONAL THERMAL ANALYSIS AND CALORIMETRY SYMPOSIUM

together with

## THERMAL ANALYSIS OF ADVANCED MATERIALS II

14 and 15 APRIL 1997

15

## OXFORD CENTRE FOR ADVANCED MATERIALS AND COMPOSITES

jointly organised by

## TMG and OCAMAC

TAC 97 will provide a general forum for the presentation and discussion of current research and development work in all areas of thermal analysis and calorimetry.

Thermal Analysis of Advanced Materials II will discuss• the application of thermal analysis methods to the characterisation of structural and electrical ceramics, polymers, metals and composites, in order to develop advanced materials processing techniques and improved material structures and properties.

Contributed papers may be either in oral or poster form and the proceedings will be published in a special issue of *Thermochimica Acta*.

Cost: Registration, including two nights accommodation, is expected to be £180-£200, registration including conference dinner only, is expected to be £110-£130, with reduced rates for students and members of the TMG or OCAMAC.

The invited speakers are:

Professor Dr H J Fecht . Technische Universität Berlin  
“Containerless Modulation Calorimetry”

Dr A L Greer . University of Cambridge  
“Thermal Analysis of Metastable Materials”

Professor Dr J Van Humbeeck . Katholic Universiteit Leuven  
“The Study of Reversible and Irreversible Transformations in Metals and Alloys by DSC”

Professor J L Ford . Liverpool John Moores University

For more information, to submit a paper or register, please contact:

Ms Diane Taylor, OCAMAC, Department of Materials,  
University of Oxford, Parks Road, Oxford OXI 3PH, UK  
Tel: +44 1865 273652 Fax: +44 1865 283333

## **PERSONAL NEWS**

**The John Barton Retirement Symposium** will be held at the University of Surrey on Wednesday, 11th September, 1996 and will comprise a number of talks from distinguished speakers who have been associated with John during his career. Further details from Dr Ian Hamerton, University of Surrey, Department of Chemistry, Tel: 01483 259587, Fax: 01483 259514

**Dr David Morgan**, our Immediate Past Chairman, is anxious to collect any and every piece of Archive Material relating to thermal methods and the Thermal Methods Group. If you are turning out your old files or cupboards and find such material, ring David on 01602 363138

**Dr Fred Wilburn**, our ICTAC Affiliate Councillor has had a most severe spell of illness, including a brief, (but dramatic!) time in hospital. Happily, Fred is well on the way to recovery and working as hard as ever. We wish him a complete and speedy recovery. Take care, Fred!

### **WE'RE ON LINE!**

**The TMG now has a section on the**

**Royal Society of Chemistry Pages:**

**<http://chemistry.rsc.org/rsc/>**

**with the addition [tmg.htm](#) for information on news,  
meetings and people!!**

e-mail Stuart du Kamp on  
[stuart\\_r\\_du\\_kamp@sbphrd.com](mailto:stuart_r_du_kamp@sbphrd.com)  
for more information

## NEW PUBLICATIONS

**“Thermal Analysis of Materials” Robert F Speyer,  
Marcel Dekker, New York, 1994, 285 p., \$110**

This monograph deals with the fundamentals of DTA/DSC, TG dilatometry, thermal conductivity and viscosity measurement. A feature of this text is that it covers the broader topics of heat transfer, furnace control and temperature measurement and that it gives many examples with special emphasis on glasses and materials science. An introduction to thermodynamics, temperature measurement and material properties and furnace control systems occupy Chapters 1 and 2 and Chapter 3 contains the theory and instrumentation of DTA and of both types of DSC illustrated by applications and examples from glass, glass-ceramic, magnetic and other materials. The practical approach to experimental technique is well described and the factors affecting measurement, plus the problems of the baseline are drawn to the readers' attention.

Methods of data manipulation by computer programmes are treated in Chapters 4 and 6, Programmes written in Quickbasic are given in full, although the different kinetic equations are given little space. The relationship between the original signal and the result after processing is made clear. Although Chapter 5 is titled Thermogravimetric Analysis (and abbreviated as TO!), and deals with the instrumentation of TG, it also includes TG/DTA and mass spectrometric EGA. The chapter concludes with an excellent and very full case study of glass batch fusion which shows the advantage of using DTA, DTG and X-ray analysis together to follow changes in a multi-component reactive system.

There are some surprising omissions. Dilatometry and thermal expansion is explained in Chapter 7 and instrumentation and operation dealt with fully, together with the theory and applications of laser interferometric dilatometers, but no mention is made of Dynamic Mechanical Analysis. Thermal conductivity and diffusivity measurements form the basis of Chapter 9 including radial heat flow, calorimetric and laser flash methods but no mention is made of the use of modified DTA assemblies for these measurements.

It is most interesting to have as Chapter 8 an account of heat transfer and of pyrometry, including total radiation and infrared pyrometry. Chapter 10 is an account of the theory and determination of the viscosity of liquids and of glasses including the problems of measuring high viscosities at temperatures up to 1400 °C

*continued*

In his Introduction, the author says that “there is an inherent danger in trusting black boxes,, and it is the intention of this monograph to elucidate their inner workings and provide some insight into their operation” This text goes a long way to achieving these aims and gives a good selection of chapter references and footnotes and two Appendices with instrument vendors and supplementary reading. It can be thoroughly recommended as a most useful introduction to thermal methods for material scientists although the price makes it an expensive textbook.

**Peter J Haines.**

**Differential Scanning Calorimetry. An Introduction for Practitioners”, G Hohne, W Hemminger and H J Flammersheim. Springer-Verlag, Berlin, 1996, 222 pp, DM 178.**

A very comprehensive text, in English, dealing with the instrumentation theory, calibration, interpretation and applications of DSC with excellent diagrams. critical appraisal and over 200 references.

**A full review of this book will appear in the Winter 1996 TMG NEWS.**

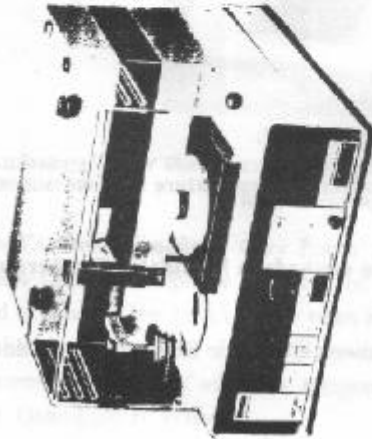
**“Dielectric Analysis of Pharmaceutical Systems”, Duncan Q M Craig. Taylor & Francis, London, 1995, 232 pp, £65.00**

This book outlines the theory and practice of dielectric spectroscopy and analysis, and gives the methods available for making measurements and examples of how the technique has been used to study systems of pharmaceutical and medical reference. Materials studied include solutions, emulsions, semi-solids, polymers, proteins and biological tissues. It will be of value to pharmaceutical scientists, physicists and chemical engineers.

A fuller review article is available from **Jed Marson at Novocontrol International, 01905 640947**

## DSC AUTOSAMPLER

- Load up your samples and let it do the work!



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Walsall, West Midlands  
WS9 8UG

Tel. 01922 59006

Fax. 01922 53320

**NETZSCH**  
Thermal Analysis

- The Exact Solution!

**“Building Thermal Analysis”, Andreas Athienitis A Mathsoft Electronic Book:  
Contact Adept Scientific plc, 6 Business Centre West, Avenue One, Letchworth  
SG6 2HB Tel: 01462 480055 Fax: 01462 480213**

Covering heat transfer in buildings, a tool for study or an on-line design assistant for engineers and architects. A compendium of real-life examples.

**“Degradable Polymers, Recycling and Plastic Waste Management”**

**A-C Albertsson, S J Huang (Eds), Marcel Dekker, New York, 1995, 344 pp, \$150**

The proceedings of the International Workshop in Stockholm offers detailed discussions and analysis of renewable resources, degradation and test methods, processing and products .polymer blends and much more!

**“Polymer Devolatilization” R J Albalak (Ed)**

**Marcel Dekker, New York, 736 pp, \$225**

**Two Reprints on Modulated Temperature DSC:**

**“Principles for the interpretation of Modulated Temperature DSC measurements:  
Pt. 1 The Glass Transition:” and**

**“A comparison of different evaluation methods in Modulated Temperature DSC”  
both written by Dr. U Schawe.**

Available from Paul Clarke, PETA Solutions, do Perkin Elmer, Beaconsfield. Tel 01494 679272

## **NEWS FROM THE MANUFACTURERS**

**GEARING SCIENTIFIC, 1 Ashwell St, Ashwell Herts SG7 5QF**

**Tel: 01462 742007**

**Fax: 01462 742565**

A new state of the art range of **LaserComp Thermal Conductivity** test apparatus which covers the range  $-20^{\circ}\text{C}$  to  $100^{\circ}\text{C}$ , without needing liquid nitrogen, is available from Gearing Scientific. New plate temperatures can be achieved within minutes and results show excellent accuracy and repeatability.

**Autosampler for the LaserComp Thermal Conductivity System available from Gearing Scientific.**

**DMTA Developments:** Gary Foster and John Gearing have set up a DM Users Club for telephone support, and help with on-site calibration, training and service. They have over 20 years of experience to offer to industrial and University laboratories. Real-life problems such as testing brake pads under extreme conditions of vibration, temperature and environment are ideal for the Gabo Eplexor system.

**Contact John Gearing on 01462 742007**

**Hidden Analytical Ltd.**

**420 Europa Boulevard, Gemini Business Park, Warrington, WAS SUN**  
**Tel: 01925 445225 Fax: 01925 416 518**

Hidden have moved to larger premises and have a new Moisture Sorption Analyser-IGASorp This is a dedicated moisture analyser for the characterisation of materials with respect to real-world temperature and humidity environments. Typical applications include pharmaceuticals, polymers, packaging materials, textiles and foodb' products

Besides this Hidden also supply Gas Analysers, Surface Analysis apparatus, gravimet;I~ analysers and much more.

For details **contact Paul Stonadge on 01925 445225 Fax: 01925 416518 or e-mail: stonadge~hidden.demon.co.uk**

**PETA Solutions**

**do Perkin-Elmer Ltd, Post Office Lane, Beaconsfield, Bucks, HP9 1QA Tel: 01494 679272**

The latest addition to the Perkin Elmer DSC range, the Pyris I DSC has been presented at two introductions during late June: There will also be two, 2-da~ Training Courses on the Fundamentals of DSC on August 7th-8th, 1996 and Octobei 23rd-24th, 1996.

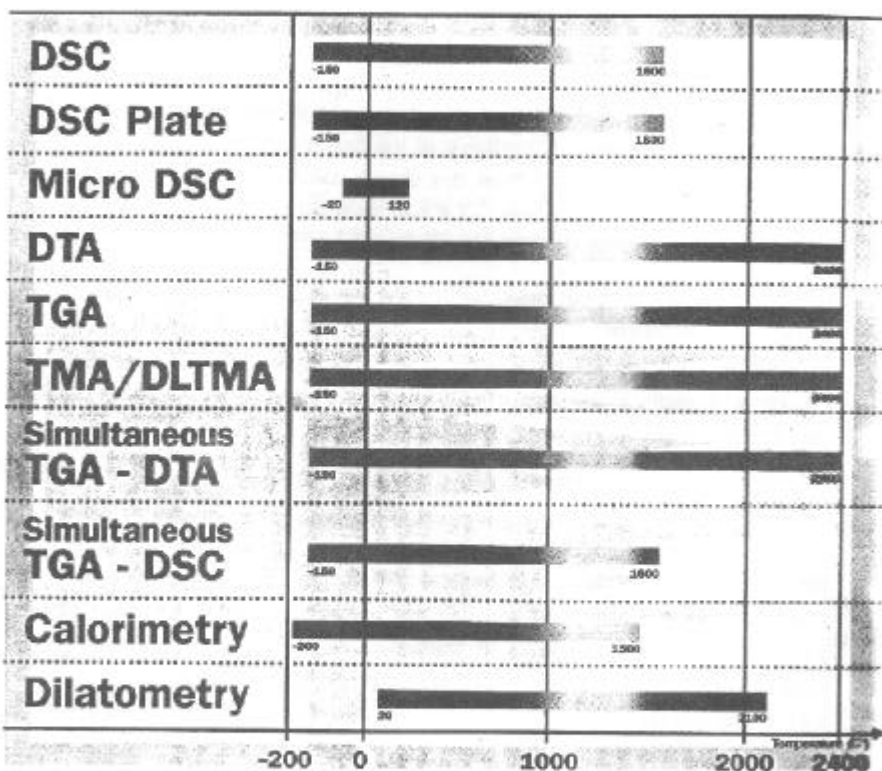
For details of the Pyris system, or the courses

**Contact: Paul Clarke on 01494 679272**

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**T A Instruments Ltd**  
**Europe House, Bilton Centre, Cleeve Rd, Leatherhead,**  
**Surrey, KT 22 7UQ**  
**Tel: 01372 360393 Fax: 01372 360135**

### **Sale of TA Instruments to Waters.**

Waters Corporation has announced that it has entered into an agreement to purchase TA Instruments. Completion of the deal is expected by the end of June 1996. TA are “excited about this change, which will be good for the employees and for the customers. TA will maintain its own identity in the market.

By joining Waters, TA customers will be able to obtain a more complete line of synergistic materials characterization techniques from a common supplier. In addition, TA has always believed in strong customer support (training courses and applications assistance by ‘phone). Waters shares this belief and you may therefore rely on a continuation of support.

New **DMA 2980 Dynamic Mechanical Analyser**. TA announces a new DMA for evaluating modulus and damping properties of materials including polymers, ceramics and metals. It features multiple modes of deformation, frequency multiplexing (0.01 to 200 Hz), a temperature range from -150 to 600 °C and can take a wide variety of sample forms and sizes. Controlled by the OS/2TM Thermal Solutions software, it may also be connected to a Rheology Analyst Controller and run in combination with a rheometer to provide full range (liquid to solid) characterisation of material deformation! flow properties.

For further information, contact

**John Kelly at T A Instruments Tel 01372 360363.**

# **THERMAL METHODS GROUP**

## **MISSION STATEMENT**

The Thermal Methods Group (TMG) promotes awareness of all thermoanalytical, calorimetric and related techniques by a range of activities, including a regular programme of scientific meetings, training workshops and publications.

## **STRATEGIC OBJECTIVES**

The TMG will~

- act as a focus for thermal analysis in the UK and contribute to the furtherance of thermoanalytical and calorimetric techniques worldwide through the support of European and International organisations in the field
- organize a regular programme of meetings on thermal analysis, calorimetry and related techniques. Joint meetings with other Groups of the Learned Societies and with other European thermal analysis Groups will be encouraged.
- publish *TMG NEWS* to keep its members in touch with the proceedings of recent meetings and details of future meetings and developments in instrumentation as well as the fortunes of their fellow thermal analysts
- encourage younger scientists working in thermal analysis to present their work to a wider audience by making the TMG Award to provide expenses for attendance at major European (ESTAC) and International (ICTAC) meetings
- promote the education of thermal analysts by holding regular seminars and workshops and by encouraging the exchange of information between the instrument manufacturers and users of thermal equipment.

## **ORGANISATION**

The Thermal Methods group is a subject group of the Analytical Division of the Royal Society of Chemistry, but its membership is open to all workers regardless of discipline. It is affiliated to the International Confederation for Thermal Analysis and Calorimetry (ICTAC) and collaborates closely with the European thermal analysis Groups.

The TMG Committee consists of a Chairman, Vice-Chairman, and Treasurer and six other members made up of representatives from academia, research institutions, industry and the instrument manufacturers

Scientific meetings are arranged twice yearly. Generally, a two-day meeting is held in Spring where presentations cover all aspects of thermal analysis and calorimetry. The Autumn meeting is usually on a specific theme and incorporates the Annual General Meeting of the TMG. In addition, a training workshop or a meeting covering educational aspects of thermal analysis is held once a year. bursaries are available for younger scientists to attend this meeting.

# THE THERMAL METHODS GROUP

## Royal Society of Chemistry, Analytical Division

Chairman	Mr J Leckenby,	Topometrix Corporation, Saffron Walden
Vice-Chairman:	Dr S B Warnngton,	TACS, Leeds Metropolitan University,
Hon. Secretary:	Dr C J Keattch,	Industrial & Laboratory Services, Lyme Regis
Hon. Treasurer;	Mrs J A Hider,	Chessington
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	Dr R Bottom,	Mettler-Toledo, Leicester
	Prof J L Ford,	Liverpool John Moores University
	Dr J N Hay,	University of Birmingham
	Dr K O'Reilly	OCAMAC, Oxford University
	Dr A J Ryan,	Materials Science Centre, UMIST, Manchester
Immediate Past Chairman:	Dr D J Morgan,	British Geological Survey, Nottingham
Co-opted:	Mr P J Haines	(Editor, TMG News), Farnham.
	Mr S R du Kamp,	(Publicity) SmithKline Beecham, Worthing
	Dr F W Wilburn	(ICTAC Affiliate Councillor), Southport.

**For** further details about any aspect of the Group's activities, please contact

**the Hon. Secretary: Dr C J Keattch, PhD, C Chem, FRSC**

Industrial & Laboratory Services,

P0 Box 9, Lyme Regis, Dorset, DT7 3BT

Tel: 01297 442221 Fax: 01297 442836