THERMAL METHODS GROUP
ANALYTICAL DIVISION
ROYAL SOCIETY OF CHEMISTRY

TMG NEWS

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This issue of the Newsletter marks a change in the publication time from mid-summer to early new year. In this way we can report on the annual general meeting and introduce new members of the committee. As usual we shall be giving a preview of forthcoming events. For this year the important occasion is the meeting to celebrate the coming-of-age of the TMG. The publication of two articles is planned to coincide with the meeting: one on the TMG and the other on thermal analysis. Both the time and venue for the meeting have been changed from those first proposed. It will now be held in November at the Bonnington Hotel, London. Members may remember we held a very successful gathering at this hotel in 1983 for Dr. R.C. Mackenzie. Once again congratulations are due to Robert, this time for gaining the ICTA – Du Pont Award 1985. It is with very great pleasure that we can report that Dr. C.J. Keattch has been awarded the Distinguished Service Award of the Analytical Division. The timing is particularly appropriate in our anniversary year since Cyril has served as Secretary of the TMG since its inception.

This Newsletter is sent free to members of the TMG. If you have any items for inclusion please contact the Editor.

COMMITTEE NEWS

The first priority of the committee has been the anniversary meeting. After much discussion the venue and format of the meeting have been agreed with the intention of attracting the maximum attendance. The detailed planning is in the hands of a working party headed by Or. Nowell. Early last year arrangements were finalised for the 8th Thermal Analysis School at Salford University. The committee was delighted with the response which made this event a great success. The committee is also pleased with the success of the final meeting of the year on biological applications planned by Mr Hardy. Members may recall that the TMG Award was to be made in connection with last year's ICTA meeting. In the event the award was not made but it remains available for a future occasion. There was some discussion of the future direction for the TMG in the context of the considerations by the Analytical Division of its group structure. When planning events the committee is acutely aware of financial constraints, the ever—increasing number of scientific meetings and the limited time available to members. Fortunately our financial situation is very satisfactory thanks to the efforts of our Treasurer. Our meetings are generally well attended but the committee has noted that members of the Group form only a small fraction of the total attendance. The committee was delighted to respond to the invitation of the Nordic Society for Thermal Analysis for a joint meeting in 1986. Members of the Group should have received the first circular. Drs. G.M. Clark and P.G. Laye are associated with the organising committees.

ANNUAL GENERAL MEETING

This was held on the 14th November 1985 at Fulmer Grange, Stoke Poges, Berkshire. The annual report and financial statement had been circulated to members of the group. This year there were a number of changes in the officers and committee members. Dr. D.V. Nowell was elected Chairman and Dr. P.G. Laye became Immediate Past Chairman. Two new members of the committee are Dr. J.T. Pearson and Dr. R.S. Whitehouse. Mrs J.A. Hider (née Rumsey) continues as a committee member for a second year.

PRESENT OFFICERS AND COMMITTEE MEMBERS 1985—86

Chairman:	Dr. D.V. Nowell, Division of Chemical Sciences, The Hatfield Polytechnic, Roe Green, Hatfield, Herts AL1O 9AB Tel. No. 07072 79560
Vice Chairman:	Mr. P.J. Haines, Department of Analytical and Biological Chemistry, Kingston Polytechnic, Penrhyn Road, Kingston upon Thames, Surrey KT1 2EE Tel. No. 01 549 1366
Hon. Secretary:	Dr. C.J. Keattch, Industrial and Laboratory Services, P.O. Box 9, Lyme Regis, Dorset DT1 3BT Tel No. 02974 2472
Hon. Treasurer:	Dr. R.H. Still, Department of Polymer Science and Technology, University of Manchester Institute of cience and Technology, P.O. Box 88, Manchester M6D 1QD Tel. No. 061 236 3311
Members:	Mr. M.J. Hardy, Beecham Pharmaceuticals Mrs J.A. Hider, Stanton Redcroft Dr. T.J. Lever, Perkin Elmer Dr. J.T. Pearson, Huddersfield Polytechnic Dr. R.S. Whitehouse, Evode Limited
Immediate Past Chairman:	Dr. P.G. Laye, Leeds University
Co-opted:	Dr. A.A. Hodgson Dr. R.C. Mackenzie

Annual Report of the Honorary Secretary, 1985

On 21st Movember 1984 the Group held a joint meeting in London, with the SRCG (Faraday Division) entitled “Applications of Thermal Methods in Catalysis”. Over 70 people attended and enjoyed a meeting of lively discussions and papers of a high standard.

“The Use of Thermal Methods in Assessing Hazards and Safety of Chemical Reactions” was the title of a one-day meeting held, also in London, on 25th January 1985. Again, a good audience resulted and the standard of the presented papers was high.

An interesting observation on the above meetings was that, in both cases, only a small percentage of participants were Group members. This fact has necessitated a change of emphasis in advertising future meetings.

The 8th Thermal Analysis School was held, at Salford University 15-19th April 1985. There were 23 participants; almost the maximum for which a school of this nature can cater.

Membership of the Group continues to remain fairly static at 326.

It is a pleasure to record the grateful thanks for the expert secretarial facilities provided by the staff at Burlington House.

C.J. Keattch

New Members

Mrs Jennifer Hider (née Rumsey) joined the TMG Committee in November 1984. Jenny is Product Manager for thermal analysis instrumentation at Stanton Redcroft. She joined the company in 1978 as a scientist in the Consultancy Laboratory carrying out testing of customer samples, instrument demonstrations and application studies. The service also offers contract testing facilities and Jenny has been involved for a number of years in studies on pyrotechnic systems, the results of which have been reported at several TMG meetings. Jenny was promoted to Deputy Head of the Consultancy Service in April 1985, and has recently moved to her present position.

Jenny graduated from the University of Manchester Institute of Science and Technology with a first class honours degree in Chemistry in 1976 and prior to joining Stantons she worked at Unilever Research in Port Sunlight, Merseyside.

Dr. John Pearson is Principal lecture in Physical Chemistry at Huddersfield. He obtained his Ph.D. degree for research into gas kinetics at the University of Wales. He spent two years in the U.S.A.: first as a research fellow at Duke University, North Carolina and then at the Camille Drefus Research Laboratories, Triangle Research Institute. His present research interests are in two areas: thermal analysis of metal carboxylates and ammonium sulphur salts; high pressure liquid chromatography of cephalosporin antibiotics.

Dr. Whitehouse joined Evode Limited on leaving school. He continued his studies at Wolverhampton Polytechnic where he gained his Graduateship of the Royal Institute of Chemistry and subsequently his Ph.D. degree. His involvement in research has been with the wide range of Evode products. He was responsible for research and development of all solvent based and aqueous adhesives and recently became Projects Manager of the Forward Technology Group. Dr. Whitehouse was responsible for the introduction of thermal analysis to the Evode Group and holds the position of Group Consultant for thermal analysis techniques.

NEWS OF MEMBERS

Mention has already been made of the awards to Drs. Keattch and Mackenzie. In addition, we can report that Dr. G.M. Clark, a former Chairman of the Group, has been appointed Deputy Director of Nene College Northampton. He took up his post on 1st January 1986.

THERMAL ANALYSIS SCHOOL

The School was held for the third time at the University of Salford between the 15th and 19th of April, 1985. It was again well supported and attracted twenty three participants almost the maximum the School could accommodate. The aim was to give hands-on experience of the instrumentation.

The programme consisted of introductory and in-depth lectures on the main areas of the thermal methods, these being followed by a series of presentations on the application of the complementary methods to pharmaceuticals, polymers, inorganics and minerals. The practical programme was devoted largely to a series of case studies in order to give as wide an experience and coverage as possible to the participants.

Once again the School was very well supported by the instrument companies and special thanks are due to the following and their representatives for their help, cooperation and provision of equipment.

Du Pont (UK) Limited
MSE Scientific Instruments
Perkin-Elmer Limited
Polymer Laboratories Limited
Stanton Redcroft

One of the highlights of the week was a visit to the Greater Manchester Museum of Science and Technology on the Tuesday evening. Thermal methods were well in evidence as we journeyed through two centuries of industrial development. We were also priviledged to see some of Joule's original apparatus, not on display to the general public. The course dinner was taken outside the university at a delightfully fashioned old village on the outskirts of Bolton, in the Last Drop Inn. A convivial evening was enjoyed by some forty participants and many of us will not forget the

“honky tonk piano renderings”.

Salford provided us with a beautiful and warm spring week and the University catered for us very well and provided excellent facilities and support.

D.V. Nowell

ESTA 3

It is now some time since the Symposium but this is the first Newsletter since then and we include a short report.

The third European Symposium on Thermal Analysis and Calorimetry (ESTAC 3) was held from the 9th to 15th September 1984 at the Congress Centre in Interlaken, Switzerland. The U.K. contingent numbered approximately 30 and presented 18 papers one of which comprised the TMG Award lecture delivered by Dr. A. Rahman of the University of Aberdeen. His title was “Application of Thermal Analysis in Surface Chemical Investigations of Zirconia Gels” and it was pleasing to see a large audience for his excellent presentation.

A feature of the Symposium was the emphasis on poster presentations. This form of delivery is particularly favoured in Europe and offers the opportunity for detailed yet informal discussions - perhaps the TMG could take greater note of this seemingly more efficient and enjoyable (to both presenter and audience) mode of presentation?

As befits a large Symposium, the programme of social events and trips was exhaustive and exhausting. These included a spectacular Conference Dinner including local entertainments, a memorable trip by funicular railway up the “Schynige Platte” from which the panorama of the Bernese Dberland mountains is breathtaking, and a last night boat trip on Lake Thun only marginally spoiled by a thunderstorm.

This was ESTA/ESTAC’s first venue outside the U.K. and Dr. Marti (Conference Chairman), Professor Oswald (Scientific Programme Chairman), and the whole of the organising committee are to be congratulated. Personally I regret that Conference Proceedings were not

published and that it was not possible for all participants to be resident in one place but these do not detract from a very successful and enjoyable event. Undoubtedly the ESTAC symposia are now well and truly launched and will remain regular highlights of the Thermal Analysis calendar for a considerable time. The original pioneers in the TMG should be well pleased with the outcome.

ESTAC 4 goes to Jena in the German Democratic Rupublic

G.M. Clark

ICTA

The 8th ICTA Conference, 18-23rd August 1985 in Bratislava, Czechoslovakia was attended by over 400 delegates from 32 countries. The scientific sessions were held in the Congress Hall and the delegates were accommodated mainly in nearby hotels. There were several plenary and award lectures but otherwise the programme relied heavily on poster sessions. This had the advantage of easing language difficulties and accommodating the large number of contributions. Dr. Lazarev and Dr. Kudinov, the President and Secretary respectively of the Russian Thermal Analysis Society presented medals to 16 scientists including Dr. Mackenzie for their contributions to thermal analysis. The medals were in honour of the 125th anniversary of the birth of Dr. Kurnov the pioneer of thermal analysis in Russia. A feature of this conference was a series of workshops on a variety of topics. The proceedings of the conference were distributed to each delegate and will be published in Thermochimica Acta.

The Governing Council of ICTA approved a number of changes in the structure and operation of ICTA including a change in the name of the meetings from Conference to Congress. The following Officers were elected

President	Professor	H.J.Seifert
Vice President	Professor	S . St. J. Warne
Secretary	Professor	S. Yariv
Membership Secretary	Dr.	H.G. MacAdie
Treasurer	Dr.	J.S. Crighton

The 9th ICTA will be held in Jerusalem, Israel in 1988.

COMMERCIAL EQUI PMENT

The following information has been supplied by the companies concerned or their agents.

Columbia Scientific Industries

Columbia Scientific Industries are the manufacturers of the accelerating rate calorimeter (ARC). This is a computer—controlled adiabatic calorimeter using a sample of several grams contained in a sealed system working over a temperature range ambient to 500^0C. Purpose-designed to simulate the conditions of material processes, storage or transportation the ARC has become recognized as the technique for the investigation of exothermic reactions and how they may lead to thermal runaway. The isothermal stepping heat-wait-search' operating procedure enables reactions to be detected at self—heating rates from $O.01^0C\ min^{-1}$. The experiment then proceeds under highly adiabatic conditions with data stored in the control processing unit. The computer will process the data to give complete time-temperature-pressure figures including the self-heat plot and time to maximum rate corrected for worst case hazard data'.

Columbia Scientific Industries International Office is located in Milton Keynes. From this Office a full customer sample service and instrument demonstration are available. Technical information sheets and articles describing applications in almost all fields where exothermic reactions are of concern are available. For further details please contact:
Dr. Martyn Ottaway, CSI Corpn. Intl., 101 Garamande Drive, Milton Keynes MU8 8DD, Tel. No. 0908-569595.

Du Pont

The new Du Pont 9000 thermal analyser and 9900 computer/thermal analyser systems are based on a series of components that can be tailored to meet the specific needs of the analyst, whether these needs be as basic as temperature progranning and recording or as complex as multimodule data acquisition and computation.

The 9000 thermal analysis system consists of a keyboard/display, twopen digital plotter, module interface and appropriate analysis module. Options include an expanded random access memory (RAM) and a six-pen

digital plotter. The instrument is designed to provide complete temperature/time programming with volatile data storage capability. The unit can be easily upgraded to the 9900 computer/thermal analyser since all components of the 9000 system are used with the 9900. When coupled to any Du Pont Thermal analysis module, the 9000 controls the equipment, thermal progranning, data collection and plotting.

The 9900 computer/thermal analysis system consists of a professional computer with a dedicated Du Pont operating software system, CRT display screen, floppy disk data storage, module interface, two-pen digital plotter, and desired analysis module with appropriate software. Expanded versatility is available through the addition of a 10 or 20-megabyte Winchester disc drive, dot-matrix printer, and a six-pen digital plotter. A choice of a monochromatic oreight-colour CRT is offered.

The Du Pont TA operating software gives the computer/thermal analyser multitask capability, providing increased sample throughput and allowing the thermal analyst to perform many operations simultaneously. The 9900 system can be expanded to accommodate multiple thermal analysis modules. In this configuration, the central computer can simultaneously control and acquire data from up to four different modules. The 9900 computer/thermal analyser features an extensive library of Du Pont thermal analysis software which can be supplemented with operator-generated analysis software.

For further details please contact: Jezz Leckenby, Du Pont (UK) Ltd., Wedgwood Way, Stevenage, Herts., SG1 4QN. Tel. No. 0438-734015, Telex. 825591.

Netzsch

Netzsch (UK) Ltd., market the Netzsch range of thermal analysis instrumentation in the UK. The current product line includes a full range of thermal analysis and thermal testing equipment which includes:

1. Thermobalances working in the temperature range -170^{0}C to 2400^{0}C dependent on furnace.
2. Differential thermal analysers working in a similar range.
3. Simultaneous TG-DTA units.
4. A new heat flux differential scanning calorimeter between -14O^{0}C and 500 ^{0}C.

5. A new high temperature 0SC 25^{0}C ... 1400^{0}C.
6. A full range of classical and differential dilatometers and thermomechanical analysers.
7. Thermal conductivity testing of refractories up to I600^{0}C.

All Netzsch thermal analysis instrumentation can be linked via a standard data acquisition interface with built in buffer memory to personal microcomputers for both automatic control of the instrument and automatic data acquisition and data processing/evaluation. Netzsch offer full software packages written for Hewlett—Packard HP8O series micros.

Recent developments include a vapour pressure balance for the determination of small vapour pressures in the range 1 to 1O^{5}Pa (10-2 to ~ mbar) with temperatures up to 350^{0}C, a water vapour furnace system for TG and STA measurements in water vapour saturated atmospheres in the temperature range ambient to 900^{0}C, a new capillary STA-MS coupling system.

For further information on any of our products contact: Mr B Boardman, Netzsch (UK) Ltd., Loomer Road, Chesterton, Newcastle, Staffs. ST5 7PZ. Tel. No. 0782 563300, Telx. 367204.

Perkin Elmer

Since the last newsletter, Perkin—Elmer have introduced new instruments to both the TADS and 7 Series range of thermal analysis equipment.

The TADS Series

Perkin—Elmer bring automation to thermal analysis with the introduction of the DSC-4 Robotic System (DSC-4R5). The Robotic System comprises a removable 48 position carousel and pneumatically controlled robotic arm. The thermal analyst can now run up to 48 samples (with optional re-run) totally unattended. The system was specifically designed for the repetitive nature of the quality control laboratory. The software permits new samples to be added, or the order of samples to be changed, without interrupting the analytical cycle. Full diagnostics are performed during the loading and removal of each sample from the DSC sample holder. The system has been tested for 30,000 hours to ensure long-term reliability.

With the Robotic System it is now possible to do all the “one-off’ samples through the day and leave the routine work to run overnight. In

the morning all the samples will have been run, the data stored on disk, and all curves (together with any calculations) will have been plotted out.

The 7 Series

Perkin-Elmer have recently introduced the TMA 7 to complement the existing state-of-the-art DSC 7 and TGA 7 analysers. The TWA 7 is consistent with the design approach that runs throughout the entire range:- "no-compromise". Dimensional changes as small as 0.4 ijm/cm are routinely measured through the use of a highly sensitive linear variable differential transformer (LV0T), which provides exceptional baseline performance over the entire temperature range. The low mass furnace permits heating - and cooling - rates as fast as 100 C/mm to be obtained.

The multi-tasking capability of the 7500 computer has been expanded to give the analyst three completely separate tasks. The ability to run up to three analysers simultaneously - performing different experiments - has obvious advantages to both the research and quality control laboratories.

For further information, please contact Dr. T. Lever, Perkin Elmer Ltd., Post Office Lane, Beaconsfield, Bucks. HP9 IQA. Tel. No. 04946 5151.

Setaram

Setaram of France offer, through Clandon Scientific Limited, an extensive range of Thermal Analysis equipment. Whether one is looking for rapid temperature scanning, high sensitivity, wide temperature range and small sample volume or a combination of these parameters, then Setaram have an instrument to suit your needs. These include the unique C8O Calvet Calorimeter, two Differential Scanning Calorimeters, the High Sensitivity Micro-DSC Scanning Calorimeter, the Simultaneous TG-DSC Calorimeter, as well as several sensitive high and low temperature Calvet Microcalorimeters and Thermo and Microthermoanalysers.

The C80 can be used between ambient and 300^0C and the wide range of cells available for this instrument enable mixing, vacuum, high pressure and flow experiments to be carried out. The Micro-USC Batch and Flow Calorimeter has a power resolution down to 0.2 microwatt which makes it suitable for biological applications requiring high sensitivity.

The DSC 2000 K is specifically designed for isothermal and scanning work at high temperatures and is particularly useful for ceramics and refractory materials.

A recent addition to the Setaram range is the TG/DSC 111 Analyser enabling TG/DSC/DTG to be carried out simultaneously on the same sample over a temperature range of -123% to 827^{0}C achieving sensitivities of 1 microgram (TG) and down to 10 microwatts (DSC).

Application Files which comprise a series of typical experiments are available for Elastomers, Cements/Plasters, Thermal Hazards, Fossilised Fuel and Gas Adsorption/Catalysis. Setaram are always willing to run tests on customers' samples in their extensive laboratories in Lyon and report their findings in the form of a detailed report. For further information please contact: Alan Pendrey, Clandon Scientific Limited, Lysons Avenue, Ash Vale Nr. Aldershot, Hampshire GU12 SOR, Tel. No. 0252-514711.

Stanton Redcroft

Stanton Redcroft announce their new CETA Computer Enhanced Thermal Analysis system designed specifically for thermal analysts. A powerful experimental control and data processing system, it is capable of being integrated with a wide range of thermal analysis modules — DSC, DTA, TO, TMA and simultaneous TG-DSC and TG—DTA. The system features high resolution colour graphics with an eight colour screen display (700 x 500) and with two, three and four channel versions, it is capable of acquiring data from a range of modules. The user is provided with a comprehensive suite of custom designed thermal analysis software which includes automatic analysis and the ability to create user-defined methods. Specialist programmes such as purity, kinetics and compositional analysis will be available shortly. The system offers multi—modular capability and enables the user to analyse data on the high resolution screen while collecting other data from new experiments. A range of hardware options makes it possible to tailor a system to each user's particular requirements. Storage options include a Winchester Hard Disc Drive and there is a choice of multicolour A3/A4 size plotters.

Also available is a new consultancy service leaflet giving details of the wide range of thermal analysis techniques available on a contract

research basis.

For further details please contact: Mike Stevens, Stanton Redcroft Ltd., Copper Mill Lane, London SW17 OBN. Tel. No. 01-946—7731.

FUTURE MEETINGS

Joint Nordic—English Symposium on Thermal Analysis and Calorimetry

20-23rd August 1986, Bergen, Norway. The Symposium will cover all aspects of thermal analysis and calorimetry including theory and applications in any field. The programme will consist of plenary lectures, short oral presentations and poster sessions. A round table discussion or work shop on the topic 'The Use of Thermal Analysis in Relation to Oil Exploration and Production' will be included in the programme.

The first circular has been sent to members of the Group. Dr. P.A. Barnes of Leeds Polytechnic has been invited to give a plenary lecture. Further details concerning the symposium may be obtained from Norvald G.jelovik, Norsk Hydro a.s., P.O. Box 4313, N—5013 Nygardstangen, Norway.

Anniversary Meeting - 13-14th November 1986, Bonnington Hotel, London. The first circular for this meeting will be available shortly.

The following meeting may be of interest to members of the Group.

11th Experimental Thermodynamics Conference - 7-9th April 1986, University of Reading.

The conference was founded to promote interest in experimental thermodynamics, to improve techniques and to stimulate applications in science and technology. Membership is open to all those with an active interest in experimental thermodynamics. Members will be accommodated in Wessex Hall, which is situated on the University campus. Some accommodation will be available for delegates' wives but no formal ladies' programme will be arranged.

Registration for the conference will commence at 11.00 on Monday 7th April 1986, in the Chemistry Department and the opening session for presentation of papers will be at 14.00. The programme will include plenary sessions, contributed papers and short contributions, with the Guggenheim Lecture as an additional feature.

Short contributions should not exceed five minutes; no previous notice or abstracts are required. The registration fee for the conference is £15, but this fee is waived for all students.

Please contact Dr. R.A. Schulz, Department of Chemical and Process Engineering, University of Surrey, Guildford, Surrey GU2 5XH, by 28th February 1986.

RECENT MEETINGS

Pharmaceutical and Biological Applications of Thermal Analysis
14-15th November 1985, Fulmer Grange, Stoke Poges, Berkshire.

Once again the Group chose Fulmer Grange for its 1985 Autumn meeting, The programme of lectures was:

1. Plenary Lecture - Applications of DSC in Pharmaceutical Development by Professor Alain Li Wan Po, Queens University Belfast.

2. Applications of Thermomechanical Analysis by Dr. R.C. Rowe.

3. Applications of Thermosonimetry to Organic Crystals by Dr. G.M. Clarke.

4. Purity Oeterminations of Triethanolamine Alkyl Sulphates by Dr. A.A. Badwan, Dr. K.C. James and Dr. W.J. Pugh.

5. Evaluation of Solid State Disorder of Drugs and Excipients by Dr. P. York and Dr. D.J.W. Grant.

6. Stability Prediction by DSC by Dr. 0. Harget.

7. A Practical Guide to Purity Measurements by Dr. T. Lever.

8. Studies in Polymorphism and Hydration by Dr. A. Smith.

9. DSC of Human Stratum Corneum by Professor B.W. Barry and Mr M. Goodman.

10. Thermal Analysis of Freeze Dried Preparations by Dr. J. Collett, Dr. A.J. Phillips and Dr. R.J. Yarwood.

11. Pharmaceutical Evaluations by Simultaneous DSC - Thermomicrocopy by Dr. C.D. Jones.

12. Flow Microcalorimetry by Dr. A. Beezer.

13. Application of Thermal Methods to the Food Industry by Dr. A. Wright.

The 14th November was the only day in the month to experience fog at Heathrow Airport, which was unfortunate, since the Plenary Lecturer, Professor Alain Li Wan Po was due to arrive there a little before mid-day. In the event he arrived at Luton Airport at a little after one o'clock, which left him just under the hour to accomplish a 50 mile coach/taxi journey. Needless to say he was late but with his customary efficiency the Group Chairman rearranged the programme (just as if he were back at Leeds Universityl) The Plenary lecturer finally arrived and presented an excellent lecture which was the hall mark of all the other contributions. It is hoped that most if not all the papers, will appear as extended abstracts in Analytical Proceedings, probably towards the end of 1986.

The 50 or so delegates not only enjoyed the lecture sessions but also the customary good food and hospitality which typifies Fulmer Grange.

C.J. Keattch