

NUMBER .3, 1983

This newsletter contains details of the third TMG Award. It will be presented at the European Symposium on Thermal Analysis and Calorimetry (ESTAC 3) and is for £350. A new feature of the newsletter is the inclusion of information on commercial thermal analysis equipment. This has been supplied by the manufacturers or their agents and includes the names of the representatives from whom further information can be obtained. There is an account of meetings held since the last newsletter and an indication of plans for the future. Arrangements for the ESTAC meeting in Switzerland next year are well in hand and members should have received the first circular. In this newsletter we include information on 'Thermal Analysis Abstracts' which are prepared under the auspices of the International Confederation for Thermal Analysis (ICTA). We are acting on a suggestion made at the Fulmer Grange meeting on the application of computers and microprocessors by including details of exchange computer programs. On a more personal note, we report the move of D. Dollimore to the U.S.A. and the retirement of R.C. Mackenzie from the Macaulay Institute for Soil Research in Aberdeen.

This newsletter is sent free to members of the TMG. If you would like to be kept informed of forthcoming events but are not a member, join the Group by filling in the application form

My two years as chairman of the TMG have been a pleasant experience and I wish to thank members of the group for the opportunity of serving in this capacity. Certainly, I have gained considerably from the meetings and I have had the pleasure of meeting many thermal analysts who have become good friends. I wish my successor well in the confidence that members of the committee will give their whole-hearted support. I am convinced that the success of the group will continue.

Many of you will be reading this newsletter at about the time that we will be marking the retirement of R.C. Mackenzie from his long and distinguished career at the Macaulay Institute. Robert was one of the founders of the TMG and ICTA. Much will be spoken and written in the months ahead about his contribution to thermal analysis — all I wish to say at this time is “Thank you Robert, and may you and Hilda enjoy a long and happy retirement”.

G.M.Clark

COMMITTEE NEWS

The major concern of the committee has been with plans for future meetings. The low attendance at the one-day meeting on the Application of Thermal Analysis to the Conservation and Use of Energy was a disappointment but it was recognised that this is likely to be the rule for specialised meetings. The TMG must continue to consider the needs of such groups as well as those of the wider membership when drawing up a programme of events. During the next year there will be a meeting on Catalysis arranged jointly with the Surface Reactivity and Catalysis Group of the Faraday Division and a more general meeting at Leeds University on Applications of Thermal Analysis. The committee has also been concerned jointly with the Clay Mineral Group of the Mineralogical Society in planning a meeting as a tribute to R.C. Mackenzie.

The committee also considered the future of the TMG Award. Again there was a feeling of disappointment at the small number of applicants. A number of proposals were made to change the nature of the award. It was eventually decided to retain the rules for a third award which has now been announced. It is hoped that the increased value will help to popularise the award which is open to both members and non—members of the group.

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Vice-Chairman: Dr. P.G. Laye, Department of Physical Chemistry,
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Dr. F.P. Glasser, Department of Chemistry,
University of Aberdeen.
Dr. L.F. Jones, Johnson Matthey Chemicals Ltd.
Mr. R.J. Howes, B.P. Research Centre.
Mr. T.B. Smallwood, Blue Circle Technical Research Division.

Immediate Past Chairman: Mr. E.L. Charsley, Stanton Redcroft Ltd.

Co-opted Dr. A.A. Hodgson, Cape Asbestos Fibres Ltd.

Dr. R.C. Mackenzie, Macaulay Institute of Soil Research.

NEWS OF MEMBERS

D. Dollimore has been appointed Professor and Chairman of the Chemistry Department at the University of Toledo, Ohio, U.S.A.

A special one-day symposium was held at Salford University to mark the occasion of his departure from Salford after 26 years to take up his new appointment. During his time at Salford his research interests have included gas adsorption, catalysis, flocculation, and, of course, thermal analysis. Here his contribution has been marked by more than 200 scientific research publications and two books. He has been chairman of the TMG and played an active part in the instigation of the first European Symposium of Thermal Analysis at Salford in September 1976. This was an instant success and laid the foundation for ESTA 2 in Aberdeen where he acted as Chairman of the Scientific Committee. He received the Mettler Award for Thermal Analysis in 1980.

The special one-day symposium for David was organised at short notice by his friends and colleagues at Salford, particularly G.R. Heal and D. Griffiths. A scientific programme was arranged with speakers from amongst ex-students and colleagues. A measure of his popularity and standing was reflected in the attendance of over 40 people, many of whom had re-arranged busy schedules. The sessions were chaired by Professor S. Bark and D. Griffiths. Six papers were presented covering a wide range of topics including zeolites, catalysis, microscopy, computer simulation, thermal analysis and gas adsorption. The final paper was given by David and was an entertaining account of the research which he intends to carry out at Toledo on the flocculation of clays and silts by polymers.

Following the meeting there was a dinner with the emphasis very much on informality. On behalf of the TMG G.M. Clark presented David with a pair of goblets in recognition of his achievements. The day was a most enjoyable occasion becoming a nostalgic reunion for old friends.

L.F. Jones

R.C. Mackenzie is to retire from the Macaulay Institute for Soil Research, Aberdeen.

A joint meeting between the TMG and the Clay Minerals Group of the Mineralogical Society is to be held on the 7-8th November 1983 at the Bonnington Hotel, Southampton Row, London WC1. The subject of the meeting

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ROYAL SOCIETY OF CHEMISTRY

Analytical Division

Thermal Methods Group

Chairman:

G.M. Clark, B.Sc., Ph.D.,
C.Chem., F.R.S.C.

Honorary Secretary:

C.J. Keattch, Ph.D.,
C.Chem., F.R.S.C.

The Thermal Methods Group of the Analytical Division of the Chemical Society announces the availability of the Third TMG Award. The award will be to the value of £350 and will be presented to the winner at the Third European Symposium on Thermal Analysis and Calorimetry ESTAC 3 to be held in Interlaken, Switzerland, 9—15th September 1984. All persons under 35 years of age and working in the field of thermal analysis are eligible. Selection will be by submission of an essay of not less than 2000 words on a topic involving thermal analysis.

For further details contact: Dr. C.J. Keattch, Honorary Secretary, Thermal Methods Group, Analytical Division, Chemical Society, P.O. Box 9, Lyme Regis, Dorset. Tel: 02974—2472. The closing date for submission of essays is 2nd April 1984.

is Clays, Minerals and Thermal Analysis. Monday the 7th November will be devoted to papers on thermal analysis and will be followed in the evening by a conference dinner. Professor G. Lombardi (University of Rome) will give an invited lecture on the study of volcanic formations by thermal analysis.

Members of the group should have received notice of this meeting. Further details can be obtained from the Hon. Secretary, Dr. C.J. Keatch.

ANNUAL GENERAL MEETING

This will be held at 5.00 p.m. on the 7th November immediately after the scientific session of the meeting on Clays, Minerals and Thermal Analysis at the Bonnington Hotel. The agenda will be sent to all members of the group.

THERMAL ANALYSIS ABSTRACTS

Thermal Analysis Abstracts is an abstracting service published since 1972, originally by Heyden and now by Wiley Heyden, under the auspices of ICTA. It abstracts on a world-wide basis any paper containing the results of experiments involving thermoanalytical methods including calorimetry, even if the primary concern is not with thermal analysis.

Annually about 2500 abstracts are published in six issues. Abstracts normally extend to about 75 words and include the authors and their addresses and a full reference to the original publication. A unique feature is the comprehensive keyword index which codes the specific thermoanalytical content of each paper into (i) generic methodology, *e.g.* TG, DTA, (ii) technique, *e.g.* dilution of sample, low temperature, (iii) broad classification of materials, *e.g.* polymers, metals, drugs, (iv) elements and compound classes, *e.g.* esters, oxides.

There are five teams of abstractors who scan original journals and submit abstracts of relevant papers to their Regional Editor who forwards them to the Assistant Editors and Editor-in-Chief (Dr. J.H. Sharp, University of Sheffield) who arranges for typing, printing and index preparation. The average time delay between the publication of a paper and its abstract is about one year. However, the Thermal Analysis Abstracts is not primarily published as a rapid information service but as a comprehensive

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compilation of thermoanalytical work in abstract format which is particularly valuable as an easy-access reference source.

The present cost is £125 per annum (less 25% discount for members of ICTA). Prospective subscribers should contact the Subscriptions Department, John Wiley and Sons, Baffin Lane, Chichester, Sussex.

EXCHANGE OF COMPUTER PROGRAMS

Microcomputers are being used increasingly in thermal methods, and many laboratories are building their own interface systems and writing their own programs. It was suggested at the TMG meeting at Fulmer Grange that it would be very helpful to pool the knowledge gained amongst members of the group. The newsletter will therefore publish brief details of programs and systems, together with the name and address of the person to be contacted for further details and program listings. If you wish to contribute, please send details to Dr. G.R. Heal, Department of Chemistry and Applied Chemistry, University of Salford, Salford MS 4WT.

List of Computer Systems and Programs

An Automated Data Collection System for DSC, Enabling Calculation of Specific Thermal Capacity and Enthalpy

This describes an ADC to link a Perkin-Elmer DSC-2 to a Research Machine 380Z microcomputer. The acquisition of sample, reference and calibrant data lead to the calculation of thermal properties of samples as a function of temperature.

Contact: Dr. P.L. Russell, Principal Scientific Officer, Flour Milling and Baking Association, Chorleywood, Hertfordshire WD3 5SH.

Program listings available: see also *Thermochimica Acta*, 62 (1983) 187-196.

Interfacing an AIM 65 Microcomputer to a Thermal Balance

A description of a system using two ADCs to interface to a TG750 which uses a BASIC main program and a machine code sub-routine to control the ADCs. Data are printed on the ROCKWELL AIM 65's own printer or stored on cassette tapes. The system has been used for kinetic analysis.

Contact: Dr. G.R. Heal, Department of Chemistry and Applied Chemistry, University of Salford, Salford 145 4WT.

Program listings available: see also Proc. 2nd Europ. Symp. on Thermal Analysis, (1981) p.72.

INSTITUTE OF PETROLEUM

The formation of the panel (ST-G-9) to study the application of thermal analysis to testing petroleum products was reported in the last newsletter. The panel has continued to be actively concerned with the experimental measurement of oxidative stability, volatility and wax appearance temperatures. Further details about the group can be obtained from the new chairman, Mr. S.R. Wallis, Castrol Research Laboratories, Whitchurch Hill, Pangbourne, Reading, Berkshire TG8 7WR. Tel. 07357-4321.

COMMERCIAL EQUIPMENT

This section contains details of equipment from six companies. This is the first of our reports and we hope to up-date the information as necessary in future newsletters.

Du Pont

The 1090 Thermal Analyser remains the centre of the Du Pont range of equipment. It is the most totally committed thermal analysis system available today. It has the capability of controlling one of a range of different modules which include the innovative dual sample DSC 912 and the powerful 982 Dynamic Mechanical Analyser. While recording data on disc in real time, the operator can perform simultaneous analysis routines on data from a separate experiment. Also the 1090 may be accessed at the same time *via* a computer interface and the operator may be setting up the instrument for a further experiment: that is a total of four operations carried out simultaneously.

Data analysis and the ease of manipulating data have been the key advances in thermal analysis instrumentation during the past five years. In the past, a purity or kinetic analysis would have been regarded as a chore. Now an operator-proof automatic analysis can generate such data in seconds rather than hours and such analyses are becoming routine. Du Pont

are able to supply a library of software with dedicated end utility program available on disc. For further details please contact: Jezz Leckenby, Du Pont (U.K.) Limited, Clinical & Instrument Systems Division, Wedgwood Way, Stevenage, Hertfordshire SG1 412\$. Tel. 0438-734691. Telex. 925591.

Mettler

Mettler have increased their range of thermal analysis equipment with the introduction of the new FP800 Thermosystem. The current family of instruments, the TA3000 and FP800 series, have been designed to allow the user to put together a thermal analysis system exactly as their requirement or budget dictates. This enables the user to buy only what he really needs — which in these days of budgeting restrictions must be of benefit.

With the FP80, the techniques cover the measurement of basic parameters such as melting, boiling and cloud temperatures as well as dropping and softening points. In addition, two modules for thermal microscopy are available; the most sophisticated, the FP84 features visual observation, whilst simultaneously recording the energy changes *via* a DSC output. This combination is proving to be a very powerful aid to the interpretation of thermal analysis results as the scientist is able to see what is going on as well as evaluate the DSC curve. To complete the FP8D range there is a DSC module available. This is designed specifically to be robust and is ideal for quality control application: standing up to the most rigorous treatment. The FP80 system can be linked to peripheral instruments such as a recorder, a printer or a computer since the necessary data interfaces are built in to the control unit as standard items.

The commonly preferred alternative to the FP80 is the more sophisticated TA3000 system, which is capable of DSC, TG and TMA measurements. The TA3000 includes a thermal analysis computer which contains a complete data handling software package. The system provides the user with fully automated analysis and immediate print-out of both the experimental conditions and results both in graphical and numerical formats. The numerical presentation makes it particularly easy for results to be compared with little risk of mistakes occurring due to the interpretation of graphs. Obviously this increases efficiency as the potential for errors is greatly reduced.

In addition to the instrumentation mentioned above, the Mettler Applications Laboratory periodically produces application notes describing current developments in the thermal analysis field. For further information on the TA3000, FP800 or application notes contact: Miss Janet Bray, HSE Scientific Instruments, Manor Royal, Crawley, Sussex RH1D 2Q12. Tel. 0293-31100.

Netzsch

Netzsch U.K. Limited began marketing in the U.K. the well-tried Netzsch range of thermal analysis equipment for this parent company at the beginning of 1983. A complete range of equipment is offered including STA systems (-16D⁰C to +2400⁰C), separate TG and DTA systems, a modern heat flux DSC system with an absolute electrical calibration system for extremely accurate quantitative measurements and a range of dilatometers, both classical and differential systems, working in horizontal or vertical configuration. In addition, a new radiation heated DTA system gives stable base lines at very high heating rates combined with high sensitivity. Evolved gas analysis systems are available including density bridges, thermal conductivity bridges and proven quadrupole mass spectrometry coupling systems. A proven data acquisition system applicable to all products is now available and can be coupled with Hewlett Packard HP-80 series personal computers. All software, both data acquisition storage and application software is written in BASIC for the HP-80 series systems.

Associated instrumentation includes thermal testing for refractoriness under load and creep in compression testing, modulus of rupture at elevated temperatures, pyrometric cone equivalent and crosswire and parallel wire thermal conductivity systems. For further information contact: Dr. David

L. Cox, Sales Manager, Thermal Analysis, Netzsch U.K. Limited, Loomer Road, Chesterton, Newcastle-under-Lyme, Staffordshire ST5 7PZ. Tel. 0782-564717. Telex. 367204.

Perkin-Elmer

This company produces a range of thermal analysis equipment of which the Thermal Analysis Data Station and the DSC-4 are recent additions. The Data Station (TADS) is a powerful microcomputer that connects to all Perkin-Elmer thermal analysis instruments. TADS provides instrument control, data acquisition and storage. TADS is programmable in BASIC and includes an extensive library of analytical routines for fast, accurate results.

Perkin-Elmer offer a complete library of TA applications and general purpose software for TAOS. This includes software to perform purity, kinetics, partial areas and specific heat analysis on the Perkin-Elmer DSC-2 and DSC-4 differential scanning calorimeters.

The Model DSC-4 is a compact, modular differential scanning calorimeter with Perkin-Elmer's unique direct power compensation design. It is ideal for routine applications demanding speed: a microprocessor controller helps the analyst to select parameters quickly, runs the analysis unattended and displays the results. Simple programming, methods storage and recall and fully automatic operation make thermal analysis easy, even for inexperienced operators. The low mass furnace of the OSC—4 helps assure rapid controlled and linear heating and cooling rates. The operating temperature range is from ambient to 600 ~ in standard mode and down to ~17DO with the sub ambient accessory. Heating rates to 200⁰C/min with equilibrium time under 30 sec, enable more samples to be run in less time, and with better resolution. The DSC-b is programmed from the System 4, which can also control Perkin-Elmer's TGA, TMA, DMA and high temperature DTA units. It is compatible with the thermal analysis data station for total computer aided thermal analysis. For further information please contact: Trevor Lever, Perkin—Elmer Limited, Post Office Lane, Beaconsfield, Buckinghamshire HP9 1QA. Tel. 014946-5151.

Setaram

The Bio Calorimeter is the latest high sensitivity scanning calorimeter manufactured by Setaram of France and available exclusively in the United Kingdom from Clandon Scientific Limited. The calorimeter is of the well known Calvet heat flux meter type but gains its very high sensitivity of 0.2 vVJ (0.1 vW is achievable with the use of an additional amplifier) by use of circulated liquid as the thermostatic medium. The calorimeter is differential and can be scanned up or down between ~200 and +100 at rates of up to 1⁰C/min. Access to the 1 cm³ cells of the calorimeter is possible to allow the introduction of mixing of samples or reagents.

Applications include protein transformation changes, denaturation, specific heat measurements and the growth and metabolism of bacteria. Recently, one of these units was installed in the National Institute for Medical Research, where it will be used to study the molecular organizations --

and interactions of biologically important molecules such as microproteins and glycos-amino glycans. The only directly equivalent calorimeter in applications is Russian built and Clandon feel Setaram's latest introduction will be welcomed by many scientists. For further information on this or any other of Setarams range of thermal analysis equipment please contact:

Alan Pendrey, Clandon Scientific Limited, Lysons Avenue, Ash Vale, Near Aldershot, Hampshire GU12 5QR. Tel. 0252-514711.

Stanton Redcroft

Stanton Redcroft Limited has joined with Carbolite Furnaces Limited to form part of a newly formed technology company, Thermal Scientific Plc. Thermal Scientific will act as a holding company and Stanton Redcroft and Carbolite Furnaces will continued to operate independently, each with its own Board of Directors, but will benefit considerably from co-operation in research and development and other areas.

Hugh Sykes, the Chairman of Thermal Scientific becomes Chairman of the Stanton Redcroft Board with John Redfern (Managing Director), Ted Charsley (Research Director) and Stephen Upton (Production Director). Richard Potts, formerly Sales Manager of VG Gas Analysis Limited, has joined the Company as Sales and Marketing Director. Other technical appointments made earlier in the year include those of Jim Swan, formerly of Servomax Limited, as Head of Research and Development, and Steve Warrington, formerly of the Leeds Polytechnic Thermal Analysis Group, as Senior Scientist in the Consultancy Service.

On the instrument front, Stantons have introduced the DAPS2 data processing system based on a Commodore PET microcomputer with added high resolution graphics, the low cost package features a high resolution 4-channel data acquisition unit, dual drive floppy disc system, high speed digital tape unit and 6 colour graphics plotter. The system which can be interfaced to the complete range of Stanton equipment comes complete with comprehensive thermal analysis software package. In addition integrated simultaneous TG-DTA-DTG mass spectrometric system is also available. The system is based on the Stanton Redcroft STA 780 simultaneous TG-DTA unit linked to a Spectrum Scientific Dataquad quadrupole mass spectrometer. For further information on the above instruments please contact: Mike Stevens, Sales Office Manager, Stanton Redcroft Limited, Copper Mill Lane, London SW17 0BN. Tel. 01-946-7731.

RECENT MEETINGS

Applications of Computers and Microprocessors in Thermal Analysis -11-12th November 1982 at Fulmer Grange, Stoke Poges, Berkshire

There were 56 participants at this meeting, which included a visit to the nearby laboratories of Perkin-Elmer Limited to see a range of thermoanalytical equipment. The introductory lecture was by Professor D. Betteridge, who spoke on his general guidelines for CITFOC (Chemists in the Field of Data Computation).

The other speakers proved to have used a wide variety of microcomputers such as Pet, Apple, Nova2, Hewlett-Packard and Aim 65. The thermal equipment used came from an equally wide range of manufacturers and included TG, DTA, DSC, EGA, calorimetry, GC and GC-MS. Much of the interfacing was homemade to some extent, involving analogue to digital converters and often scanners for several channels to log several instruments at once or to run simultaneous techniques on one sample. One speaker described the commercial Trivector data logging system. Common points stressed by many speakers were as follows: the various instruments produced analogue signals at a variety of levels, so each needed its individual amplifier to match the ADC range; temperature and weight could be recorded at about 1 sec intervals but DTA required a higher rate, up to 10 per sec, to preserve fine detail; the ADC needs to have a precision of at least 12 bits to record temperature to 0.1 °C.

Data storage and presentation were described. Many speakers made use of dot-matrix printers to produce a graphical display almost as good as a chart recorder. Most systems used polynomial equations to convert thermocouple emf to temperature and included some numerical processing to perform base line corrections, smoothing, differentiation, interpolation and averaging. Applications included identification, quantitative analysis, purity control kinetic measurements and glass transition determination.

Summaries of the lectures will be published in *Anal. Proc.*

G.R. Heal

Application of Thermal Analysis to the Conservation and Use of Energy

6th January 1983 at the Scientific Societies Lecture Theatre, Savile Row, London, WI

About 21 people attended this meeting which was arranged with the Thermal Analysis Panel (ST-G-9) of the Institute of Petroleum, It included an account of the work of the panel on the determination of oxidative stability of lubricating oils. Numerous instrumental and operating factors affected the recorded oxidation temperatures, and results from different laboratories often showed significant discrepancies. Other subjects discussed at the meeting were proximate analysis of coals and cokes and the efficiency of different cokes in the production of zinc and lead. A quite different topic was the use of DSC to investigate the freezing behaviour of hydrocarbon fuels. The evaluation of diesel fuel volatility using TG was discussed. The technique was found to provide a viable alternative to conventional techniques and was particularly valuable in assessing unusual alternative fuels both distillate and non—distillate. Although the audience was small the lectures attracted considerable interest which gave rise to some lively discussion.

FUTURE MEETINGS

Clays, Minerals and Thermal Analysis - 7-8th November 1983 at the Bonnington Hotel, Southampton Row, London WCI

Applications of Thermal Analysis - 5—6th April 1984 at Leeds University, Leeds LS2 9JT

Details of this meeting will be available shortly and will be circulated to members of the group.

ESTAC 3 — 9-15th September 1984 at the Congress Centre Casino, Interlaken, Switzerland

The symposium will include both thermal analysis and calorimetry. Sessions are planned on:

- Inorganic Chemistry and Metallurgy
- Earth Sciences
- Organic Chemistry and Polymers

Biological Sciences including Medicine and Pharmacy
Industrial Applications
Theory and Instrumentation

The first circular is available. Further information from: the General Chairman,

Dr. E. Marti,
Im Langen Loh 181,
CH-4054 Basel, Switzerland.

Catalysis - 21st November 1984

This is a joint meeting with the Surface Reactivity and Catalysis Group of the Faraday Division. The venue for this meeting has not been finalised.