

NUMBER 4, 1984

The TMG will soon be celebrating its 21st anniversary. The intention is to hold a special meeting at Easter in 1986. York has been suggested as the venue with a programme which will include both scientific and social activities. The officers of the TMG would welcome suggestions from members regarding the meeting and any other suitable events to celebrate the anniversary. It may seem a long way ahead but arrangements will have to be made during the next year. More immediate is ESTAC 3 to be held this September in Switzerland; members should have received the second circular by now. *A propos* this meeting, the TMG committee has decided that the 3rd TMG Award be made to Dr. A.A. Rahman. It is hoped that the 4th Award will be associated with the 8th ICTA in 1985. Since the previous newsletter there has been a rather special meeting held in honour of Dr. R.C. Mackenzie to mark the occasion of his retirement. The current issue gives an account of this meeting and also the one held at Leeds University this Easter. It also contains updated information on commercial equipment.

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

COMMITTEE NEWS

Once again the priority of the committee has been the scientific programme. The committee continues to be concerned at the trend towards low attendance at meetings, since this will place serious financial constraints on the group and its future activities. There was something of an up-turn at the Leeds meeting where the costs were restricted to a minimum. Even so the attendance was lower than that on similar occasions in the past. It looked as if the competition for the TMG Award was going to be a disappointment but almost at the last minute it became very competitive with a number of excellent contributions. In part, this may have been the result of the publicity poster 'Ski with the TMG' which was circulated as widely as possible. The committee has tentatively proposed that the 4th Award should be associated with the 8th ICTA.

Detailed planning of future meetings is in the hands of the Vice-Chairman and the Secretary. For next year two meetings have been arranged, one on hazard assessment and the other on a biological topic. A firm booking has been made at Fulmer Grange, Berkshire for the second meeting but details have still to be finalized. A small sub-committee has been formed to plan the 8th Thermal Analysis School which is to be held in 1985. This group has decided the general format of the School and is now working on the final details. A provisional booking has been made at the University of Salford. One item which has been the subject of discussion is the group's anniversary to be celebrated in 1986. Besides the scientific and social meeting other ideas which have been suggested include articles on thermal analysis and the history of the group itself. Much has still to be decided and it is hoped that the anniversary will provide a new impetus for the group.

ANNUAL GENERAL MEETING

This will be held on 21st November 1984 at the Scientific Societies' lecture theatre, Savile Row, London. Further details including the time and agenda will be sent to members of the group.

PRESENT OFFICERS AND COMMITTEE MEMBERS 1983-84

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- Members: Professor F.P. Glasser, University of Aberdeen
Mr. P. J. Haines, Kingston Polytechnic
Mr. M.J. Hardy, Beecham Pharmaceuticals
Dr. G.R. Heal, University of Salford
Mr. R.J. Howes, B.P. Research Centre
Dr. T. Lever, Perkin Elmer
- Immediate Past Chairman: Dr. G.M. Clark, North East Surrey College of
Technology
- Co-opted: Dr. A.A. Hodgson, Cape Asbestos Fibres Ltd.
Dr. R.C. Mackenzie, Macaulay Institute of Soil
Research

TMG AWARD

The committee has pleasure in announcing that the winner of the 3rd Award is Dr. A.A. Rahman for his essay entitled 'Application of Thermal Analysis in Surface Chemical Investigations of Zirconium Gels'. The award is for £350 and an illuminated certificate which will be presented at ESTAC 3.

Dr. Rahman comes from Bangladesh where he obtained his first degree at Dacca University. After a period as a lecturer at Dacca he came to this country to work with Professor K.S.W. Sing at Brunel University where he obtained his Ph.D. in 1977. After periods in France and Dxford he joined Professor F.P. Glasser in the Department of Chemistry, University of Aberdeen in 1981 as a research fellow working on cementitious materials.

CLAYS, MINERALS AND THERMAL ANALYSIS

A meeting in honour of Dr. R.C. Mackenzie

This was a two day meeting on the 3rd and 4th November 1983 arranged jointly between the TMG and the Clay Minerals Group of the Mineralogical Society to mark the occasion of Dr. R.C. Mackenzie's retirement from the Macaulay Institute of Soil Research in Aberdeen. It was held in the Bonnington Hotel London and attracted thermal analysts and mineralogists alike both from the U.K. and abroad. The first day was organized by the TMG and started with a tribute to Robert given by Professor T.S. West, Director of the Macaulay Institute of Soil Research. It was followed by nine papers on thermal analysis concerned mainly with mineralogy. Professor G. Lombardi from the University of Rome gave an invited lecture entitled 'Thermoanalytical Methods in the Study of Volcanic Formations Latium (Italy) and of their Alteration Products'. The second day contained 1D lectures which were preceeded by the Distinguished Member Presentation. Some 70 people attended each day. On the first evening there was a conference dinner at which the Chairman of the 1MG presented Robert with a set of crystal glasses on behalf of the group. In addition Professor Lombardi presented an inscribed tiled plaque from the friends in Italy who had worked with Robert at the Macaulay Institute.

There is to be a special edition of the journal 'Clay Minerals' which will contain 12 of the papers presented at the meeting. The cost will be £8 to members of either group. It is due to be published in December 1984 and will contain 180-200 pages.

THERMAL ANALYSIS SCHOOL

The School is held every three years and it is planned to hold the next (8th School), 15-19th April 1985 at the University of Salford. The School will again be residential and the number of places will be limited. The aim is to provide a solid background in both the theory and practice of thermal analysis. The manufacturers of thermal analysis equipment will be closely involved, providing an opportunity for the participants to gain 'hands-on' experience with newly developed equipment. Practical experience based upon relevant case studies is an established feature of the School and as far as possible is related to the participants' own fields of interest. The programme will be supported by evening activities, including visits and a course dinner. The first circular will appear in the autumn 1984. The cost will be about £300.

D.V. Nowell

ESTAC 3

The third European Symposium on Thermal Analysis and Calorimetry will be held at the Congress Centre Casino, Interlaken, Switzerland, 9-15th September 1984. The second circular has been published. The scientific programme will include papers on chemical thermodynamics, thermal analysis, calorimetry, solid state physics and material science. The sections are:

- Instrumentation
- Inorganic chemistry, metallurgy
- Earth science, ceramics and glasses
- Organic chemistry
- Polymers
- Biological Science, including medicine
- Pharmacy
- Industrial applications

The programme includes award lectures, plenary lectures, round table discussions and sessions of lectures and posters. The contributions will be published in two volumes of 'Thermochimica Acta'. Further information from the General Chairman, Dr. E. Marti, Im Langen Loh 181, CH—4054 Basel, Switzerland.

ICTA

The 8th International Conference on Thermal Analysis will be held in Bratislava, Czechoslovakia, 19-23rd August 1985. Members should have received the first announcement and preliminary registration form. The provisional programme includes the topics:

Instrumentation for thermal analysis and calorimetry

Advances in theory of thermal analysis including kinetics
and calorimetry

Thermal analysis in inorganic chemistry, ceramics and earth science

Thermal analysis in organic chemistry, biology, pharmacy and
medicine

Thermal analysis in polymer sciences, including investigations on
glasses

Thermophysical research, applied science, environmental and
industrial applications

Workshops are planned on the topics:

Thermal characterization of non-stoichiometric oxides and other
compounds and their application to energy storage and conversion

Current problems of reliability of kinetic data evaluated by thermal
analysis

The use of computers and data storage in everyday thermoanalytical
practice

Theory and practice of less common thermoanalytical techniques

It is the intention that most papers will be presented as posters. Further information will be given in the second announcement which will be sent out in August 1984.

Details are available from the Organizing Committee of the 8th ICTA, do Slovak Technical University, 812 43 Bratislava, Czechoslovakia.

EXCHANGE OF COMPUTER PROGRAMS

A scheme for the exchange of computer programs was announced in the previous newsletter. Brief details of programs will be published with the name and address of the person to be contacted for further details. 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.

INSTITUTE OF PETROLEUM

The following background information on the Petroleum Institute and the Thermal Analysis Panel ST-G-9 formed the abstract to a paper presented at the meeting at the University of Leeds.

The Institute of Petroleum was formed in 1913 and is open to all interested in oil and has 6000 members. The Standardization Committee (ST-G) is one of several reporting to the Council. The Sub-committee ST-G-A is concerned with Analysis and has Panels including ST-G-9 established in 1982 to consider thermal analysis. The Panel's membership is 15 and includes representatives of oil companies, engine manufacturers, instrument manufacturers, users of petroleum products (a government establishment, and a public transport authority) and a university.

The Panel ST-G-9 aims to produce standard methods acceptable to the oil and related industries. It is currently examining the oxidation stability of lubes, volatility of lubes and fuels, wax appearance temperature of fuels and oxidative stability of grease. A problem area is the attainment of reproducibility between different laboratories, possibly with different equipment in the oxidative stability and volatility tests. In the latter case, the procedure for temperature calibration and the design of the thermo—gravimetric equipment are important factors.

S.R. Wallis

Further details about the group can be obtained from the Chairman, Dr. S.R. Wallis, Castrol Research Laboratories, Whitchurch Hill, Pangbourne, Reading RG8 7QR, Tel. No. 07357—4321.

COMMERCIAL EQUIPMENT

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 500⁰C. 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 0.01⁰C mm¹. 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 European Office has recently moved to Milton Keynes. From this Office a full customer sample service is available and instrument demonstrations. Technical information sheets and articles describing applications in almost all fields where exothermic reactions are of concern are available. For further details please contact Or. Martyn Ottaway, CSI Corpn. Intl., 101 Garamande Orive, Milton Keynes MU8 800, Tel. No. 0908-569595.

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 OSC 912 and the powerful 982 Dynamic Mechanical Analyser. While recording data on disk 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, i.e. a total of four

operations can be carried out simultaneously.

Two new products have been added to the range in 1984. First, there is the dual sample PRESSURE DSC cell. Compatible with the 912 cell base, it offers throughput advantages to industries such as petrochemicals and catalysts. Secondly, there is the new 1600 degree High Temperature OTA cell which is fully compatible with existing systems offering improved baseline and sensitivity performance.

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. Cu Pont are able to supply a library of software with dedicated and utility programs available on disk. For further details please contact: Dr. David C. Harget, Cu Pont (UK) Ltd., Finished and Fabricated Products Department, Speciality Products Division, Wedgwood Way, Stevenage, Herts SG1 4QN. Tel. No. 0438-734692, Telex 825591.

Mettler

Mettler Instruments AG have expanded the versatility of Mettler TA3000 Thermal Analysis system. They have taken the successful Mettler TC10 computer noted already for its ease of use with extensive data handling capabilities, and linked it to the IBM personal computer. This has resulted in a Thermal Analysis system which can be tailored to suit any laboratory's requirements and budget. The basic system has been extended two—fold. Firstly in its data handling programmes and secondly in its ability to have a choice of detectors to cope with corrosive samples or for high sensitivity requirements. Further the addition of an IBM dual-disc drive system gives the TA3000 system extended data storage capabilities with bi-directional communication. Thus the operator can create almost unlimited experimental times, reprocess data at will, or process data at a convenient time to fit in with the laboratory schedule. With facilities already included such as direct connection of a balance, automatic sample handling and the ability to link methods including data handling calculations, TA3000 can be programmed to work with a minimum of experience and very little supervision. The system has the capability to be extended further to include the complete IBM personal computer with

disc drive, coloured monitor and keyboard, supplying the laboratory with a system of immense versatility. There have been other developments in the Mettler Thermal Analysis TA3000 range namely in the area of accessories. An example of this is the bending and flexure kits available for the Thermo-Mechanical Analysis module.

The range of Thermal Analysis instruments is completed by the Mettler FPBOO system. This range covers the simple measurement of melting points, boiling points, and cloud points. Other modules support the TA3000 system by providing a hot stage for a microscope and simultaneous DSC measurement which is invaluable in the interpretation of DSC experiments.

Finally the system has a low cost DSC module and can be readily linked to personal computers. Data is currently available to link the FPBOO system to the Epson HX20 computer. Further details and demonstrations can be obtained by contacting Carl D. Jones, Mettler Product Manager, MSE Scientific Instruments, Manor Royal, Crawley, West Sussex, RH10 2QQ. Tel. No. 0293—31100.

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°C to 2400°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 -140W and 500% .
5. A full range of classical and differential dilatometers and thermomechanical analysers.
6. Thermal conductivity testing of refractories up to 1600°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 HP80 series micros.

Recent developments include a vapour pressure balance for the determination of small vapour pressures in the range 1 to 10⁻² Pa (10⁻² to 10⁻¹ mbar) with temperatures up to 350^oC, a water vapour furnace system for TG and STA measurements in water vapour saturated atmospheres in the temperature range ambient to 900^oC, a new capillary STA—MS coupling system. For further information on any of our products contact: Dr. D.L. Cox, Netzsch (UK) Ltd., Loomer Road, Chesterton, Newcastle, Staffs. ST5 7PZ, Tel. No. 0782—564717, Telex 367204.

Perkin Elmer

To complement their existing range of TAOS Series thermal analysers, based on the Model 3600 Data Station, Perkin—Elmer have recently introduced the 7 Series Thermal Analysis System. This unique system allows completely independent, simultaneous operation of multiple thermal analysers or data analyses tasks. Highly sensitive and reliable, fast and easy to use, the 7 Series is ideal for laboratories with a large sample throughput, where speed and precision are essential.

The following applications illustrate the capabilities of the 7 Series:

A cure reaction of an epoxy can be carried out using one instrument at the same time as a second instrument is performing a sub—ambient glass transition upon an elastomer.

A high temperature thermogravimetric analysis up to 1500 deg C can be in progress while previously stored DSC or TGA data are being analysed and viewed in colour on the screen of the Perkin-Elmer 7500 Professional Computer.

A heat/cool/reheat DSC experiment to ascertain the thermal history of a polymer can be carried out at the same time as stored data on other experiments are being compared and a third set of data is being printed out.

The 7 Series is controlled by the Perkin—Elmer 7500 Professional Computer, a fast, multi—tasking, 16—bit microcomputer with soft keys, high resolution colour graphics, two floppy disc drives and a mass storage

Winchester hard disc. The 7500 permits multi—tasking instrument operation and data analysis. Multiple thermal analysis modules can be operated under independent instrument control; at the same time, they can take data and store them on the Winchester hard disc of the PE7500 for later analysis. Data can be taken from a thermal analysis module, results calculated and reports printed out from previously collected data - all at the same time.

The new, advanced thermal analysis modules for the 7 Series include: the DSC7 a highly sensitive, power—compensating differential scanning calorimeter offering rapid heating and cooling rates up to 500 deg c/mm; and the TGA 7 thermogravimetric analyser, with automatic furnace cooling, high sensitivity and an optional high temperature furnace for operation up to 1500 deg C.

The 7 Series is supported by TAS 7, an extensive library of thermal analysis software. This library contains software routines for the operation and analysis of data obtained from the 7 Series thermal analysis modules and includes multi—tasking programs.

The 7 Series is fast and easy to operate. The entire system is controlled from the keyboard of the PE7500 and its eight bezel-mounted soft keys. There are no long delays after keying in instructions - the response of the system is virtually instantaneous. Screen prompts guide the operator throughout the most complex of analyses, from instrument setup to data analysis, and instrument status is continuously updated on the screen. Once the system is running and storing data from two analysers concurrently, it takes only 2 or 3 seconds for the operator to redirect instructions from one analyser to the other. Built-in service diagnostics facilitate fault-finding and guarantee minimum downtime. For further information, please contact Dr. T. Lever, Perkin-Elmer Ltd., Post Office Lane, Beaconsfield, Bucks. HP9 1QA. 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

C80 Calvet Heatflux Calorimeter, two Differential Scanning Calorimeters, the High Sensitivity Bio-DSC Scanning Calorimeter, mentioned in the previous Newsletter, as well as several equally sensitive high and low temperature Calvet Microcalorimeters and thermo and microthermo-analysers.

Setaram have recently introduced the Model 1885, a modular Thermogravimetric Analyser. It can be extended or rendered more specialized by means of a wide range of highly effective accessories and like all other Setaram equipment can be interfaced with a data processing system. The 885 Balance has an absolute sensitivity of 10 micrograms and the ICC 85 Temperature Programmer and Controller offers unequalled programming possibilities.

Also new to the Setaram range is a TG/DSC111 Analyser enabling TG/DSC/DTG to be analysed simultaneously on the same sample over a temperature range of -123°C to 827°C achieving sensitivities of 10 microwatts (TO) and 15 microwatts (DSC).

Application files on cements and plasters, elastomers and thermic hazards evaluations are readily available on request and also 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 Ltd., Lysons Avenue, Ash Vale, Near Aldershot, Hampshire GU12 5QR, Tel. No. 0252—514711.

Stanton Redcroft

Stanton Redcroft Ltd. announce the introduction of two new differential scanning calorimeters: The STA-785 simultaneous TG-DSC unit is the most recent addition to the STA-780 family of simultaneous thermal analysers. It features a suspended heat-flux DSC head, with a chromel sensor plate incorporating positive crucible location. The sample is heated by a silver block heater with integral liquid nitrogen cooling as a standard feature, enabling programmed heating and cooling to be carried out over the entire operating range of -125 to 625°C . The design of the low swept volume sample chamber enables good atmosphere control to be maintained and gas changeover to be effected without purging of the balance system.

The latter is based on the standard STA-780 system 5 g capacity electronic microbalance with a microprocessor based control unit enabling any weight range from 2—200 mg full scale deflection to be selected. Constant calorimetric sensitivity is provided by means of a microprocessor based linearizer module which also enables the sample temperature to be displayed and recorded to 0.1%. The unit thus combines the advantages of heat-flux DSC with those of a high sensitivity microbalance.

The DSC 1500 operates over the range from ambient to 1500%. The DSC head is of the heat flux type, the sensor plate being constructed from platinum-rhodium alloy, and located in a high purity alumina cup. Sample and reference materials are contained in platinum crucibles 6 mm diameter and 4 mm high, positively located on the plate. A special lid also permits the use of quartz crucibles 30 mm long for samples that creep or bubble. Provision is made for gas flow directly through the DSC cup. An optional head fitted with a chromel plate is available for high sensitivity measurements in the range ambient -700%. Samples are heated by means of a water cooled platinum-rhodium wound furnace. The furnace is provided with a motorized handling system for precise and reproducible location over the measuring head and for automatic cooling of the furnace on completion of the run using the in-built fan. As with the STA-785 the DSC output is given directly in milliwatts and the sample temperature is measured directly and displayed to 0.1%.

For further information on these units please contact Mike Stevens, Sales Office Manager, Stanton Redcroft Ltd., Copper Mill Lane, London SW17 0BN, Tel. No. 01-946-7731.

RECENT MEETINGS

Applications of Thermal Analysis - 5-6th April 1984, University of Leeds

Some 50 people attended the lecture sessions with 30 residents in the Charles Morris Hall of the University. The programme consisted of the following lectures:

1. Physical Ageing - A General Property of Glassy Polymers: Studies of Polyaryl Ether Ketone
.J. Keimnish and J.N. Hay, University of Birmingham
2. The Use of Thermal Analysis in Determining the Fusion Behaviour of PVC Compounds ;
Marianne Gilbert, Loughborough University of Technology
3. Examination of Pharmaceutical Film Coats by Thermal Methods
P. York and A.O. Okhamafe, University of Bradford
4. Lifetime Prediction by Thermal Analysis
J. Leckenby, Du Pont
5. The Work of the Thermal Analysis Committee of the Petroleum Institute
S.R. Wallis, Castrol
6. Oxidative Temperatures of Lubricating Oils
A. Cash, University of Leeds
7. Analysis of DSC Exotherms
T. Boddington, University of Leeds
8. Thermal Stability Assessment: The Application of Thermal Methods
M.R. Ottaway, Columbia Scientific Industries
9. Low Temperature Behaviour of Water—Oil Microemulsions: Thermal and Dielectric Analysis
G.G.T. Guarini, O. Senatra and G. Gabrielli, University of Florence
10. Microcomputers in On—line Data Acquisition and Processing
P.A. Barnes, Leeds Polytechnic
11. The Role of Organic Binders in Pyrotechnic Reactions
E.L. Charsley and Jenifer Rumsey, Stanton Redcroft
12. The Heat Capacity of Fossil Fuels
M.J. Richardson, National Physical Laboratory
13. Thermal Analysis of Some Ammonium—sulphur Salts
P.E. Adams, J.T. Pearson and J.A. Wood, Huddersfield Polytechnic
14. Thermal Analysis and Liquid Crystals J.Lydon, University of Leeds

FUTURE MEETINGS

ESTAC 3 - 9-15th September at The Congress Centre Casino, Interlaken, Switzerland

Applications of Thermal Methods In Catalysis - 21st November 1984 at The Scientific Societies' Lecture Theatre, Savile Row, London W1.

This is a joint meeting with the Surface Reactivity and Catalysis Group of the Faraday Division of the Royal Society of Chemistry. The first notice of the meeting has been circulated to members. Registration will cost about £35 (~20 for students) and will include morning coffee, buffet lunch and afternoon tea. Further details and an additional call for papers will be given in the second notice.

All intending participants should notify Dr. L.F. Jones, Catalyst Department, Johnson Matthey Chemicals Ltd., Orchard Road, Royston, Herts. Tel. 0763—44161 Ext. 3595.

The Use of Thermal Methods in Assessing the Hazards and Safety of Chemical Reactions — 25th January 1985 at The Scientific Societies' Lecture Theatre, Savile Row, London W1.

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

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

The title of this meeting is tentative and details have still to be finalised.