

REPORT ON THE WORKSHOP: EDUCATION, PUBLICATION AND STANDARDIZATION IN THERMAL ANALYSIS

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1. EDUCATION

1.1. Status report

In most countries very little thermal analysis is taught at undergraduate level, although short sessions of a few hours only are widespread and relatively common. From institution to institution, dependent on their specific research work, different types of education occur within the bounds of general physical chemistry relevant to their research work and apparatus.

In Czechoslovakia, Finland, Japan and the U.S.A. some obligatory courses exist for students in theoretical and practical thermal analysis.

Education in special courses at the undergraduate level does not seem to be compulsory in most countries. A practical method in most countries, defined by J. Rouquerol, "Showing our research work and how helpful thermal methods are for the resolution of our scientific problems"—seems to be sufficient. This is particularly so where scientific societies (Czechoslovakia, G.D.R., U.K.) organize postgraduate courses. At postgraduate level, as mentioned previously, there is considerable activity, with many countries running regular or irregular courses of up to one week's duration, dependent on demand. Many of these are summer schools conducted in different venues where required and some involve visiting speakers (experts), even from other countries.

Three outstanding developments which might serve as models are the three full-day courses run by E. Turi of the New York Polytechnic and J. Rouquerol of the Laboratoire du Calorimetrie of the C.N.R.S., Marseille, and the course (also on tape) with accompanying instruction notes produced by B. Wunderlich of the Department of Chemistry of the Rensselaer Polytechnic Institute, Troy, U.S.A.

Thermal Analysis Highlights, 8th ICTA, Bratislava, Czechoslovakia.

1.2. Individual contributions communicated during the workshop

Reports about thermal analysis education in their countries were given by: A. Mikhail (Canada), J. Rosický and J. Šesták (Czechoslovakia), M. Leskelä (Finland), J. Rouquerol (France), W. Ludwig (G.D.R.), G. Liptay (Hungary), H. Tanaka (Japan), E. Marti (Switzerland), R.C. Mackenzie (U.K.), E. Turi and B. Wunderlich (U.S.A.). Further written comments were given by R. Blachnik and E. Wiederholt (F.R.G.) and M. Guttman (Poland).

In these countries the situation is as follows:

Canada. The status of university education is very different to that in other countries and easy to summarize. In most cases there is only a small amount of special training in thermal analysis. Modern DSC, DTA, TG or TMA apparatuses are in operation, but in many cases the operators do not have any special thermal analysis training. Special courses and meetings are organized in connection with NATAS.

Czechoslovakia. Education in thermoanalytical methods as a part of courses for the higher grades at universities and technical universities is not unified by an instructional programme, e.g. at the Faculty of Science of Charles University in Prague there is, within the scope of special studies in chemistry, a course on thermal analysis of 30 h per semester; in other universities this is part of a special course in physical chemistry. Practical laboratory exercises for students with thermoanalytical instruments are from 3 to 10 h per semester according to the extent of the lectures and the instrumental facilities of the department. The Faculty of Science of Charles University and the J.E. Purkyně University in Brno are testing a programme on thermal analysis in the 7th semester of a new curriculum for sciences, which, after its approval, will be obligatory for all faculties of science in Czechoslovakia. This programme includes instrumentation techniques for thermal analysis, calibration of instruments and evaluation of the resultant curves.

Finland. At the Helsinki University of Technology a special graduate course on thermoanalytical methods is part of the student's education. Thermal methods of investigation are used in connection with most research projects. They have been found particularly useful in monitoring various high-temperature reactions, such as the production of oxysulphides and ferric pigments, as well as for systematic studies on the rare earth sulphates. The methods include TG, DTA, DSC, EGA and kinetic analysis.

France. Education on thermal analysis in France is also very different. At the undergraduate level only a little is taught on the basis of general physical chemistry. Methods of calorimetry dominate, which have a long tradition in the activities of the AFCAT. For twelve years J. Roquerol has organized courses in calorimetry with 30–40 experiments for students. Interest in this course is very high.

G.D.R. At every university and technical university there are different fields of research and therefore there is a wide range of education and practical

exercises in thermal analysis from phase diagrams, glass transition, melting, dehydration and kinetic interpretations in the chemical departments of the universities of Jena, Berlin, Leipzig and Greifswald, to material sciences, silicates, salts, ceramics, raw materials, polymers and metals at the technical colleges of Freiberg, Merseburg, Dresden and Weimar. There are also special courses in the fields of physical chemistry, fundamental methods of measuring and their application to analytical problems, composed of lectures and exercises. Education at post-graduate level is organized by the TA-group of the Chemical Society of the G.D.R., which organizes 1-week "Autumn Schools" every two years to study the physical problems of thermal analysis for its 60 members (1977, kinetic aspects; 1979, theoretical aspects; 1981, dynamic calorimetry and phase diagrams; 1983, dynamic calorimetry and noncalorimetric methods; 1985, quantitative aspects).

Hungary. A systematic training scheme in thermal analysis, founded by L. Erdey, is organized by the Technical University of Budapest. Training in both the theoretical background and the practical applications of thermal analysis is provided. Undergraduate and postgraduate students work on projects in thermal analysis, while thermal and thermoanalytical methods can be taken up as optional disciplines for one semester. In postgraduate courses there is also some systematic training in thermal analysis. At the other Hungarian universities thermal analysis is part of the chemistry courses. As a result of the successful activity of the Hungarian Thermoanalytical School, in 1967 the Thermoanalytical Group of the Hungarian Society of Chemists was founded. A working committee for Thermal Analysis of the Hungarian Academy of Sciences organizes and controls thermoanalytical research all over Hungary. As a result of the progress of thermal analysis in Hungary, in 1969 the Journal of Thermal Analysis was founded, the first international journal in this field. For more than 25 years the Derivatograph has been produced by MOM and exported around the world.

Japan. The wide field of thermal analysis in Japan covers all aspects of thermal analysis including calorimetry, thermometry and thermomechanical methods. The methods are widely used in research and industrial applications. In all universities and technical colleges undergraduates and postgraduates receive some training in the methods of thermal analysis. At the Faculty of School Education of Hiroshima University they are beginning a programme of special lectures and training courses for undergraduate chemistry [1].

Switzerland. There is no special education in thermal analysis at the universities and technical colleges of Switzerland, but it is taught in lectures, seminars and practical exercises and as part of the research work of postgraduate studies. Regular conferences and meetings, organized by the Swiss Society of Thermal Analysis and Calorimetry, are used to relay information on the developments and new aspects of thermal analysis. "Applied thermodynamics" is a regular postgraduate course for scientists

and engineers, open to students and given by a professor of the University of Fribourg in 3 half-day seminars.

United Kingdom. Apart from Salford University and some of the Polytechnics, there appears to be little teaching of thermal analysis at the undergraduate level in the U.K. At Aberdeen it amounts to two lectures plus some laboratory instruction. In addition to this, several universities and polytechnics have held special two-day courses of instruction and the Thermal Methods Group has a week-long school every two years with both lectures and practical work.

U.S.A. The status of university education in the special field of thermal analysis in the United States is easy to summarize. In most universities there is no special training in this field. Only a few schools, known for their research in thermal analysis, have special enrollment courses with low numbers of students. Most thermal analysts who operate or supervise the thousands of modern DSC, TG or TMA installations have not had any special education in thermal analysis, neither is there any indication that this situation will be changed in the near future. In this field of primary and continuing education, the Rensselaer Institute has developed modern, innovative courses via audio tapes [2] and through personal computers [3]. More than 300 students have worked through these courses either on or off campus. Each course is equivalent to a full semester of graduate study, starting with a classical background at about the bachelors level (4 years university training in science or engineering). Other shorter courses include, in particular, the week-long, annual course at New York Polytechnic Institute [4] and occasional short courses through the American Chemical Society and local thermal analysis groups [5].

F.R.G. In most universities in the first semester of general chemistry there is some special training in thermal analysis applied to simple inorganic and organic systems. Advanced lectures and practical exercises in the fields of physical and inorganic chemistry deal with phase diagrams. Extensive practical education in thermal analysis takes place (physics of metals, metallurgy) during appropriate postgraduate courses. To teach thermal analysis a simple apparatus has been developed and tested [6].

Poland. Thermochemistry and calorimetry were initiated by W. Swietoslawski and St. Bretsznajder. Studies in this field are carried out at the institutes of the Polish Academy of Sciences. These topics, however, are relatively less advanced in Polish academic schools. Nevertheless, some specialists are working at the universities in Toruń, Łódź, Cracow, Warsaw and Wrocław. During the fourth year of study students select their specialization. Subsequently, they carry out various practical exercises during one semester and attend specialized lectures. This permits them to gain a knowledge of calorimetry, thermochemistry and thermal analysis. They start by studying the literature and finish with a report setting out the aim, method, results and their interpretation of the exercise. During the fifth year, students

perform experiments for their M.Sc. degree in their specialized field. This is possible only in academic schools with specialized calorimetry and thermal analysis laboratories. Therefore, the supply of new young investigators is rather scarce. It would be better to make study in this field obligatory for all students and moreover to increase the number of hours for specialized practical exercises and lectures.

1.3. Conclusions and recommendations

The workshop members agreed with B. Wunderlich's argument that a classical education in thermodynamics should be given to every student of chemistry, chemical and materials engineering and those in physics to provide a sufficient basis to become a qualified thermal analyst. Thermodynamics has developed over the years in such a way that it covers only a few of the aspects of thermal analysis. Thermal analysis represents a unique blend of equilibrium and irreversible thermodynamics, the discussion of thermal conductivity and the application of statistical thermodynamics and kinetics. It must clearly separate time-dependent from equilibrium data and equilibrium from non-equilibrium processes.

Education in thermal analysis must take two avenues for the resolution of this problem, briefly this is outlined in the form of a recommendation to ICTA for action.

First, the education of new students should include thermal analysis as a general topic including classical thermodynamics, as outlined above. This will not only remedy conceptual problems, but also direct student's interest to thermal analysis at an early stage, which is of increasing importance for material analysis.

Second, to cope with the present problem of poor preparation in the field of thermal analysis, continued education courses must be encouraged. Such courses should, however, be cognizant of the fact that a full education in thermal analysis cannot be achieved in a day or week-long course, but is a full field of study which may take several semesters.

Further, a basic textbook is necessary for initial instruction and background for those starting in the area of thermal analysis.

Thermal analysis could be encouraged by holding summer schools for students or high-school teachers in the country where the ICTA conference is being held. The subjects of these schools could be "What is Thermal Analysis?", "What can we obtain from Thermal Analysis?", "The principle and application of TG, DTA, DSC", and include demonstrations of the instruments. The lecturers at these schools could be scientists or university teachers who are attending the ICTA conference and scientists or engineers from companies which are exhibiting equipment.

Last, but not least, co-operation between special centres on a world-wide scale is indispensable for raising the level of academic teachers who train

students and perform research work in countries with less education in thermal analysis.

2. PUBLICATION

R.C. Mackenzie contributed to this subject as editor of Thermal Analysis Abstracts. The TAA have not been used widely enough, and therefore the cost has been relatively high. The merits of TAA in hard copy form were discussed in relation to complementary computer literature searches. If this were accomplished, it would also solve the distribution problem for underdeveloped countries since computer terminals and network costs are far less than for storage of printed copies in areas of low density of research personnel. On the other hand, most institutions, on the basis of cost, cannot afford both Chemical Abstracts and Thermal Analysis Abstracts.

Advantages of TAA were seen to be due to:

- (i) the specialist nature of the abstractors,
- (ii) the better coverage of earth science topics, and
- (iii) the extensive use in the many countries where computer search facilities are not available.

Enthusiasm was expressed for a revision and expansion of the ICTA publication "For Better Thermal Analysis", which is now to be undertaken by a newly formed committee.

J.H. Flynn, in his workshop on current problems of kinetic data reliability, formulated topics and goals to be considered by the ICTA committees including the setting up of tables of derivations of common equations for kinetic models, classification of techniques and methods to be referred to by authors and the development of nomenclature, symbols, recommended practice and terms for the description and classification of processes (see the report, this volume page 101).

3. STANDARDIZATION

The Standardization Committee of the ICTA has completed testing and preparing a set of temperature standards for DTA and TG which are marketed through the U.S. National Bureau of Standards. The very able leadership of the ICTA Standardization Committee, by Dr. H.G. McAdie (Canada) and Prof. P.D. Garn (U.S.A.), was gratefully acknowledged.

It was stated by the newly elected chairman of the Committee, Dr. P.K. Gallagher (U.S.A.), that there are still steps to be undertaken towards promoting international cooperation.

K. Heide (G.D.R.) spoke in support of the continuation of the valuable work of the Standardization Committee and stressed the importance of high-temperature, enthalpy and glass transition standards. Special aspects,

such as phase diagrams, also need the attention of the Committee.

V. Balek and J. Šesták (Czechoslovakia) stated that in Czechoslovakia a set of DTA temperature standards analogous to that of the NBS/ICTA has been tested. Further cooperation in this task with scientists of the G.D.R., Hungary and U.S.S.R. is anticipated.

EDITORIAL NOTE ON STANDARDIZATION BY VLADIMÍR BALEK AND JAROSLAV ŠESTÁK

The newly elected Chairman of the ICTA standardization committee indicated the goals and activities of this committee after the 8th ICTA Conference at Bratislava, in a letter addressed to all ICTA members. It stated that a number of temperature standards for DTA and TG tested in the preceding period, are now marketed through the U.S. National Bureau of Standards.

The potentially worthwhile tasks listed below were outlined by the Standardization Committee for future investigation.

- (1) Higher temperature (i.e. above 1000°C) standards for DTA.
- (2) Standards for dilatometry, particularly at elevated temperatures.
- (3) Standards for enthalpy.
- (4) Standards for high-temperature glass transitions.
- (5) Standards for evolved gas analysis, and primarily mass spectrographic determinations.
- (6) Standards for heat capacity.
- (7) Purity analysis by DSC.

The ICTA Standardization Committee invites all scientists who are interested in participating in the testing and establishment of standard materials and methods to contact the chairman (Dr. P.K. Gallagher, AT&T Bell Laboratories, Rm. 6D-311, Murray Hill, NJ 07974, U.S.A.).

REFERENCES

- 1 H. Tanaka and K. Kawabata, Proc. 8th ICTA '85, Bratislava, *Thermochim. Acta*, 92 (1985) 219.
- 2 Audio Course 72.6684, *Thermal Analysis (Introduction, Thermometry, DTA, Calorimetry, TMA and Dilatometry, Thermogravimetry)*, ten C-90 audio cassettes and 300 page textbook, Rensselaer Polytechnic Institute, Troy, NY, 1978, 1982.
- 3 Computer Course 72.6694, *Introduction to Differential Scanning Calorimetry (Instrumentation, Melting Transition, Glass Transition, Heat Capacity)*, four C-90 audio cassettes, six 5-1/4" floppy discs for ATART 80C-series personal computers, Rensselaer Polytechnic Institute, Troy, NY, 1984.
- 4 *Thermal Analysis in Research and Production*, 10th Annu. Conf., Polytechnic Institute of New York, Brooklyn, May 1985.
- 5 For announcements and discussions see the NATAS Notes (issued quarterly by the North American Thermal Analysis Society).
- 6 E. Wiederholt, *Differenzthermoanalyse (DTA) im Unterricht*, Praxis Schriftenreihe Chemie, Bd. 37, Aulis Verlag Köln, B.R.D., 1984.