

Approaches to teaching thermal analysis

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Abstract

The needs of the next generation of students, researchers and industrial workers for clear, readily available information and instruction in thermal methods is considered. The selection of topics for basic and for more advanced courses is discussed and the methods which can be used to convey information are reviewed. Conclusions about the needs and methods to be employed in the future recognise that a sustained effort must be made to ensure that instruction in the techniques and applications of both traditional and newer “state of the art” methods is encouraged. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

It is a primary concern for all who use analytical chemistry that there should be a very good training for those who will succeed them. The idea of the “master” training his “apprentice” is still around, despite the social and technological changes which have taken place. To investigate the present state of training in the art and science of thermal analysis, it is useful to consider what we should teach and how we might teach it now and in the future.

Over the last few years, there have been several appropriate comments made at conferences:

- In 1992, Phil Willcocks of ICI commented “A view from industry would be that one aim should be simply to raise the level of awareness of the real potentials of an ever increasing range of Thermal

Analysis techniques”. He also commented that too often he has heard the words: “Yes, I know all about thermal analysis. We studied that one afternoon in my . . . course” [1].

- At the 30th Anniversary meeting of the TMG in 1995, Vincent Mathot from DSM, Netherlands, said that “more attention should be devoted to education to bring thermal analysis and calorimetry users up to standard and also to stimulate the market pull of users” [2].
- In 1998, Bernhard Wunderlich in the USA wrote that out of over 10 000 students only 6% took a course in thermal analysis and only 21% had heard about thermal analysis in courses on polymers, materials, physical chemistry or even analytical chemistry. He added “this in turn limits the future teachers in the field . . . and promotes low-grade thermal analysis which is a major problem” [3].

It was interesting recently to receive a letter from Singapore from a teacher who was incorporating thermal analysis into a book of A-level comprehension exercises. Surely, if good A-level students can “com-

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prehend” infrared and mass spectrometry, as well as all the other classical analytical techniques, some simple thermal analysis should not be beyond them? The essence of what should be taught falls into three main areas: theory, experimental and applications.

2. What to teach?

2.1. Theory

The basic principles of thermoanalytical methods, including the simpler versions of the theory of heat transfer, thermodynamics, kinetics and mechanics might form a part of any course on physical chemistry or materials science. Unfortunately, there is a tendency to omit more and more of the “difficult” mathematical concepts as well as much of the basic physics, because of limitations of time and talent. This leaves the student lost when he comes to read a text dealing with the fundamental theories of thermal methods such as DSC or DMA.

It is therefore most important that there is available some easily understood source explaining the principles of each of the thermal analysis methods. In each course of study, whether for an academic undergraduate, a trainee worker in industry or a novice researcher, the first question is “*What should we teach?*”. Considering the needs of chemists, pharmacists, materials scientists, polymer scientists and other users, everybody studying these subjects needs to learn at least the fundamentals of the basic methods shown in Table 1(A). How deeply the course describes and expands on these methods, and what further techniques, such as those in Table 1(B), may be thought appropriate depends chiefly on the time available and the needs of the particular course. In some cases, only 1–3 credit hours can be spared for specific teaching of thermal analysis [3].

2.2. Experimental

Thermal analysis is, and always should be, an experimental science. To know the theory of DSC without ever having performed a real DSC experiment would not be very useful. Time does not, however, allow that any person training in a basic course can run every possible experimental technique under all

Table 1
What should we teach?

(A) <i>The basic techniques, instrumentation and applications of</i>
Thermogravimetry
Differential thermal analysis and differential scanning calorimetry
Thermomechanical analysis and dynamic mechanical analysis with some mention of evolved gas analysis and simultaneous methods
(B) <i>More techniques and advanced methods</i>
Modulated DSC
Thermomicroscopy
Constrained rate techniques
Thermoelectrometric techniques
Viscometry
Techniques of product analysis

experimental conditions. In attempting to summarise the need to report all the factors likely to affect the results of a thermal analysis experiment, we coined the acronym SCRAM [4]:

S	sample
C	crucible or sample holder
R	rate of heating (or cooling)
A	atmosphere
M	mass of sample and its packing

A realistic experimental course for training in the basics, which we have used at the Thermal Analysis Schools run by the Thermal Methods Group of the Royal Society of Chemistry, contained 16 case studies from which the participants could choose about five most appropriate to their needs. Wunderlich’s survey of American Colleges showed that experiments there varied from 1 to 7 [3] and at several UK universities there is usually time for only one or two. Several manufacturers recognise this “short-fall” and incorporate “Help-Screens” into the computers connected to their instruments.

2.3. Applications

It should be remembered that the majority of users of thermal methods in industry are more interested in the information they obtain than in the technique itself. Consequently, most are drawn to the discipline from a pragmatic need, rather than idle curiosity. So, an emphasis on the applications, utility and benefits of using thermal methods is a necessary component of

“what should be taught”. Having obtained their attention the theoretical and experimental components described above are required in order that they produce good data and make realistic interpretations.

3. How to teach?

There is, therefore, a need for some means of instruction for any person wanting to use thermal analysis in his research or job, which will allow that person to have access to “expert” knowledge and comparative experimental results, properly reported, so that he will be able to validate his work. Tribute must be paid to the instrument manufacturers for their efforts in producing study aids for individual workers. Since the 1950s, they have produced excellent and informative leaflets and brochures to tell the user (or customer) what their apparatus would (or should) do. This continues today with the use of modern, multimedia and web-based instruction (see Table 2).

These may be considered in turn. *Lectures from experts* may be the best way of learning how the technique was developed and what thinking lies behind the method. It is never possible for every person to sit at the feet of the expert, and so it is useful that some lectures by the leaders in the field are available on video for class or private study [5]. In the same way, some experts, notably Bernhard Wunderlich, have transformed their teaching into Audio Courses and Training Programs, for example in the Thermal Analysis of Polymers [6]. Perhaps it is debatable whether any UK student would be dedicated

enough to work through some 20 thirty-minute lectures.

Journals are now so expensive that many places of learning cannot afford to subscribe to many and are cutting back on those which are less favoured. Possibly electronic on-line searching will eventually replace the purchased journal. It is also true that undergraduate students do not find research papers easy to read. Many are written with other experts and researchers in mind. Very few papers on thermal methods have appeared in the “popular” journals such as *Chemistry in Britain*. There is a need for papers related to thermal analysis to be contributed to the new publication “University Chemistry Education” [7].

The standard university course used to involve the “transmission of facts from the lecturer’s notes to the student’s notes without passing through the mind of either”. *Lectures to groups* of 150+ students, without the time for supportive tutorials, are not the most effective way of imparting knowledge. Even the production of illustrative hand-outs, giving equations and diagrams so that mis-copying will not occur, tends to send the students into “sleep-mode”. Leaving gaps for them to insert important facts too often results in gaps in their notes, too. It is therefore important to recognise that many students will not specialise in a particular discipline, but may need to know the basics, or to know of a source of information if they need to refer to it later. Specialist texts have been written on DSC [8] and its applications to materials [9], polymers [10,11], calorimetry [12] and pharmaceuticals [13]. “Thermal Characterization of Polymeric Materials” edited by Edith Turi has excellent reviews of instrumentation and practice as well as containing some of the most up-to-date work in the field [11].

In the 1950s it was usual for students to purchase their own copies of the *basic textbooks*. With the demands on the student grant and the range of subjects studied in some courses, the purchase of a text for any individual analytical subject is no longer possible. Even with college libraries buying several copies of a text, it is not unusual to find all are out on loan, “permanently borrowed”, or in use. Perhaps a good answer is for *standard undergraduate analytical textbooks* to include an up-to-date chapter on thermal methods. Certainly one text does contain a good chapter, but 20 pages, out of 900, cannot cover enough

Table 2
How can we teach?

(A) *Expert-based*

Lectures from originators and expert users

Journal articles by researchers

“Chalk and talk/glare and stare” Lectures from qualified practitioners

Specialist textbooks

(B) *Student-centred*

Introductory textbooks

Manufacturers’ literature and compilations

Video learning packages

Computer-based learning: Discs and CD-ROMs

Internet sites

[14]. Much the same applies to some standard American texts [15]. Some of the best introductory texts have been out of print for many years and consequently are also out-of-date. Others do provide a reasonable grounding in the fundamentals of many thermal methods. A short list is provided by the references in this paper.

4. Student-centred and computer-based learning

We must recognise the need for students to learn some of their course on their own. Of course, help should be available and if it is by means of re-playable, interactive material on their own PC or networked terminal, then they can work at their own pace, repeat as often as they need to, and save up any awkward questions for their tutor, or for a remote instructor. The manufacturers have made a most significant contribution for teachers by producing excellent descriptive brochures of instruments and applications, compilations of results and teaching aids. This continues with informative computer-based materials. Some examples will illustrate the usefulness of this contribution.

4.1. Floppy discs

The construction, functioning and operation of instruments is well illustrated in the manufacturers' brochures, and in computer presentations such as those by Rheometric Scientific [16] and Mettler [17]. The theory of operation is also included in such presentations and there is a particularly full description of MTDSC by Bernhard Wunderlich available on disc from T.A. Instruments [18].

4.2. Booklets

The operation of instruments and the conditions for experimental work are most fully described in booklets from Netzsch [19], Mettler [20], SETARAM [21] and Shimadzu [22] for polymers, foods and pharmaceuticals.

4.3. CD-ROM

The wide-ranging collection of TA Instruments (and the earlier Du Pont) Thermal Analysis Literature has

now been transferred to CD-ROM for easier access [23].

4.4. Video

As well as the lecture material available on video [5], three videotapes have been produced on DTA/DSC, TG and EGA and TMA/DMA [24]. These allow students to observe the instrumentation, and to follow the chemical and physical changes studied, supplemented by thermomicroscopic images.

4.5. Internet and web sites

Besides the general site offered by the Royal Society of Chemistry [25], there are specialist web sites dealing with the chemical industry [26] and education [27]. Wunderlich's course on the "Thermal Analysis of Materials" is now available as well as a database of heat capacity information [28]. Again, tribute must be paid to the manufacturers for their useful web sites.

5. What next?

As practising thermal analysts we must be active in educating our colleagues and managers in the benefits of these techniques. The difficulty found by staff in obtaining release and funding to attend conferences or courses must be recognised. It is essential that the information is widely and easily available. Suggestions for improving the accessibility of thermal analysis information include more training workshops, the establishment of "centres of excellence" which may be consulted when problems arise, plus the production of more videos, discs, CD-ROMs and other information material. Perhaps one thing that might be investigated is the extent and effectiveness with which thermal analysis is taught in all the relevant departments of the UK universities and colleges.

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