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**Book Review**

# Thermal Analysis: Fundamentals and Applications to Polymer Science

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T. Hatakeyama, F.X. Quinn, J. Wiley and Sons, 1994

This publication is an introduction to the methods of thermal analysis (TA) with applications to polymers and is mainly intended for beginners. All essential methods are presented. The first 12 pages deal with DTA and DSC and the following 19 pages with calibration and sample preparation. Thermogravimetry (TG) is dealt with on 26 pages and the chapter "Applications" comprises 40 pages. Another 30 pages briefly present a great number of other methods. Furthermore, the book consists of a glossary of TA terms, tables of standard reference materials, physical constants, conversion tables (here, instead of the former thermochemical calorie (4.184 J), the international calorie which equals 4.1868 J should have been listed), and a chemical formula index.

The introductory chapter which briefly presents the characteristics of the various TA methods, stresses the important fact that non-equilibrium methods are considered and underlines the significance of parameter influences. In Fig. 1.1, the DTA signal is unfortunately given as $\Delta T/\Delta t$ ($\Delta T$ is correct; see Chapter 2). The imbalance between the chapter on DTA/DSC and that on TG is striking. For polymers, DTA/DSC is of great importance so a more comprehensive, better description of DTA/DSC is desirable. The description of classical DTA is far too short; modern commercial facilities are not mentioned but a custom-made high-pressure DTA is described, which is not very useful in an elementary book. Errors and lack of clarity impair the understanding of the principles of DSC measurements. In heat-flux DSC, the measured temperature difference is not proportional to the change in the heat flux but to the differential heat flow rate to sample and reference sample. It is most unusual that the heat capacity of the heat-sensitive plate is measured by adiabatic calorimeters to allow enthalpies of transition to be estimated using heat-flux DSCs: heat-flux DSCs are generally subjected to calibration.

The statement of a maximum sensitivity is misleading (the sensitivity would have to be stated in $\mu V\ mW^{-1}$); what the authors mean is the heat flow rate detection limit which is linked with the noise of the measuring system. In the following passages, sweeping statements are made which — though generally correct — are

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of no use to the beginner and user, as only brief hints are given without the problems being made clear. For DSC, a special device is presented (triple-cell quantitative DTA), which is of no interest at all to the beginner.

In the chapter on power compensation DSC, the authors say that the energy input is proportional to the heat capacity of the sample. This could be misunderstood; it would have been correct to say that the energy input difference per unit time supplied to the sample is proportional to the heat capacity of the sample. The DSC-7 (Perkin-Elmer) which has been available for many years is not discussed; only the previous model, the DSC-2, is mentioned.

References to further introductory literature or literature for the advanced reader are not given at all.

The chapter "Calibration and Sample Preparation" gives useful hints and clearly stresses the influence of sample parameters but the directions about how best to proceed are not sufficient. Above all, the calibration and the role of uncertainties in the measurement should have been described in more detail to provide a better basis for the chapter "Applications". More recent publications on the calibration of DTA/DSC are not mentioned. It is a mystery why a maximum operation temperature of 400 K is given for stainless steel crucibles (Table 3.2): neither is it understandable why for a large amount of sample at a slow scanning rate a silver sample vessel is recommended owing to its high thermal conductivity. Firstly silver oxidizes quickly and secondly, especially at a low heating rate and crucible material is not very important: the large amount of sample itself determines the dynamics of the temperature fields.

The hints for sample preparation (especially for polymers) are useful and reflect the experience of the authors.

The TG chapter has a much better structure and gives a much better description than the DTA/DSC chapter. The measuring system (weighing instrument), the furnace, the calibration, and the influence of the sample, atmosphere, heating rate, etc., are described very well, providing the beginner with an idea of the capabilities and limits of TG.

The chapter "Applications of TA" deals with a number of evaluation methods for determining sample- or reaction-specific quantities, i.e. transition temperatures, enthalpy, kinetics, purity, glass transition, heat capacity, crystallinity, etc. Here it would have been desirable to present the methods for the quantities which can be determined directly from the curves of measurement values (temperatures, heat flow rates, heat, mass change) in more detail — including information on how to determine the uncertainties. These quantities form the basis for further evaluations (kinetics, purity, etc.). The references in this chapter, do not include the latest developments e.g., no reference is made to the work of M. Richardson.

When applying the measurement procedures described, for example for determining the heat capacity (chapter 5.5), to obtain meaningful results, it would be better to proceed in accordance with the operating instructions for the device than to follow the procedure described here in which the heat capacity measurement is carried out without a reference crucible. Furthermore, the corresponding Fig. 5.14

shows a glass transition for the calibration material (sapphire) — an observation which so far has only been made with any certainty by the authors. The quantities $T_i$, $T_e$, $I_s$ and $I_r$ used in the text do not appear in the figure.

The chapter "Applications" is followed by the final chapter "Other TA Methods" but, unfortunately, the concerted use of many TA methods to solve a problem is not illustrated by examples. The lack of literature references offering further information on the methods is again a serious shortcoming.

To whom is this publication (164pp.; £35.00/US$56.00) of use? Only the chapter on TG meets the minimum didactic requirements to be fulfilled by the structure and contents of a specialist chapter. The chapter on DTA/DSC is not worth reading. The chapter "Applications" lacks the necessary fundamental metrological information, e.g. determination of uncertainties, and thus does not allow the capabilities and limits of the TA methods themselves and the interpretations of the measurement results to be evaluated. The chapter "Other TA Methods" specifies a great number of individual methods but scarcely shows how these can be integrated in an overall interpretation.

Should another edition be planned, it is advisable to have the book thoroughly revised. Otherwise, its purchase cannot be recommended.

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