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The use of Differential Scanning Calorimetry in Chemical Education Part 1, A general description

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Abstract
In this paper a description is given of the possibilities of DSC in chemical education. First, well-suited instrumentation is described, thereafter a special instruction package is explained.

1. INTRODUCTION

As pointed out earlier [1], in chemical education a clear need exists for the implementation of Thermal Analysis techniques into the curriculum.
Main problems obstructing a rapid introduction of those techniques into chemical education are:
1. the absence of well-suited instrumentation.
2. the absence of special educational tools, allowing a rapid implementation of thermal analysis, both for students and teachers.
In this contribution a description is given of the way in which these problems were eliminated.

2. INSTRUMENTATION

A Differential Scanning Calorimeter has been developed especially suited for use within chemical education. The development backgrounds of this instrument were described earlier [1] and the instrument is available at Mettler as DSC12E. Though developed for educational purposes, the instrument also has many possibilities in the industrial market, mainly as a result of its specifications which are on a high level. They are listed in Table 1.
The DSC12E is currently being used at various universities and technical colleges in the Netherlands both in the graduate and in the under-graduate programs. As an example, the Technical University of Delft uses four DSC12E in the freshman practical training. From all users, the reliability, flexibility and user-friendliness of the instrument are mentioned frequently. Also the availability of a MS-Windows based software package to a great extent increased the popularity of the DSC12E.

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Table 1
DSC12E, Specifications

Temperature Range	-40 - +400 °C
Temperature accuracy	< 0.4 °C over the total range
Temperature reproducibility	< 0.1 °C
Heating Rates	1 - 20 °C/min (integer values)
Thermal Noise	approx. 15 μW (peak-peak, 200 °C)
Signal time constant	approx. 4 sec.
Baseline drift	<4 mW (50-400 °C)

3. EDUCATIONAL TOOLS

To achieve a rapid introduction of Thermal Analysis techniques into the curriculum of educational institutes, the availability of well-suited instrumentation alone is not sufficient.

Therefore, a textbook has been written, entitled "the practice of thermal analysis" [2]. This book is divided into two parts:
In part 1, "Definitions and applications", after a general introduction, in which the definition and history of thermal analysis is given, a number of well-known and less-known thermal analysis techniques are comprehensively described. Thereafter, the construction of thermal analysis instruments is outlined and some basic thermodynamics, necessary for a good understanding of thermal analysis, is presented. In a large chapter, a great number of applications of thermal analysis in various fields of chemistry is given.
Part 2, "DSC Tutorial", first presents the background theory of DSC. After that, sample preparations techniques are outlined and finally, 16 DSC experiments are comprehensively described. These experiments are very illustrative and can be performed by one or more students in a limited amount of time. (in general less than three hours). The experiments selected are listed in Table 2.

Table 2
Educational DSC experiments

1. Calibration of a DSC	2. Phase diagram of Pb and Sn
3. Thermal behaviour of PET	4. Glass transitions
5. $CaSO_4$-H_2O equilibrium	6. Dehydration of $CuSO_4.5H_2O$
7. Polymorphism	8. Curing of an epoxy resin
9. Crystal transitions of NH_4NO_3	10.Oxidative stability of Polyethylene
11.Melting	12.Identification of Polymers
13.Explosives	14.Melting behaviour of a strong PE fiber
15.The Curie transition	16.Liquid crystal transitions

Of each experiment, first the underlying theory is given, thereafter, the experiment is thoroughly described, the measured curves presented and explained. A number of questions are asked.

Next to this book, a sample kit is available, containing most of the samples needed to perform the experiments.

Further, for teaching purposes a set of twenty overheads is available, containing the backgrounds of thermal analysis. Finally, a poster has been designed on which the basics of DSC are displayed.[3]

As an illustration of the large information content of DSC and the insight which can be obtained into various chemical and physical processes, one experiment, the dehydration of $CuSO_4.5H_2O$ will be described here into detail.

4. DEHYDRATION OF COPPERSULPHATE

In this experiment, the student is first asked to heat some coppersulphate carefully on a watch glass. The blue colour disappears and colourless crystals are formed. After adding a drop of water, the blue colour reappears. The reversibility of this (equilibrium) reaction is easily shown in this way.

Thereafter, a sample of approx. 10 mg. $CuSO_4.5H_2O$ is heated at 10 °C/min in an open crucible. The obtained curve is given in Figure 1.

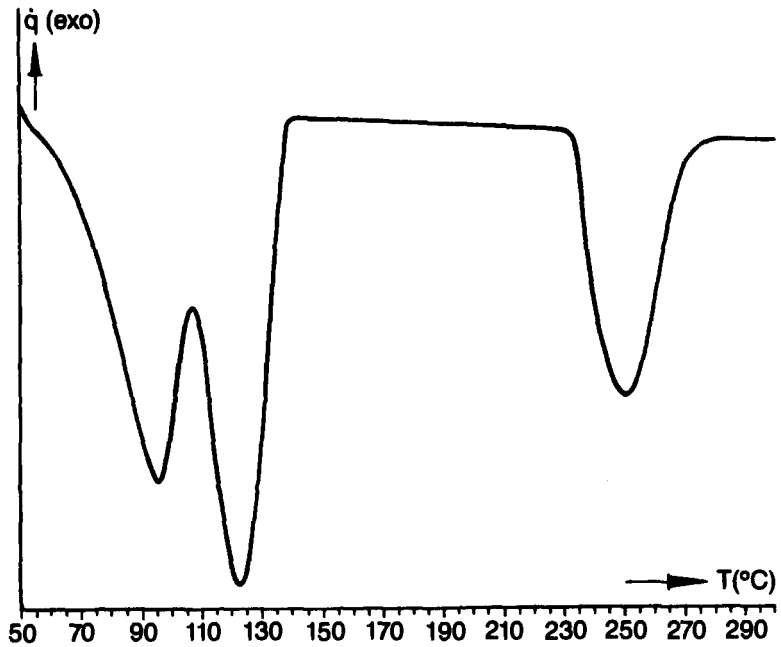

Figure 1. $CuSO_4.5H_2O$ in open crucible

This curve shows the well-known three steps in which $CuSO_4.5H_2O$ looses its crystal water. By measuring the relative peak areas it can be shown easily that the endothermal effect is not only caused by the evaporation of water, but also by the bonding forces of the individual molecules.

When a similar sample of $CuSO_4.5H_2O$ is measured at 10°C/min in a crucible with a small hole in its lid, a curve as given in Figure 2 is obtained.

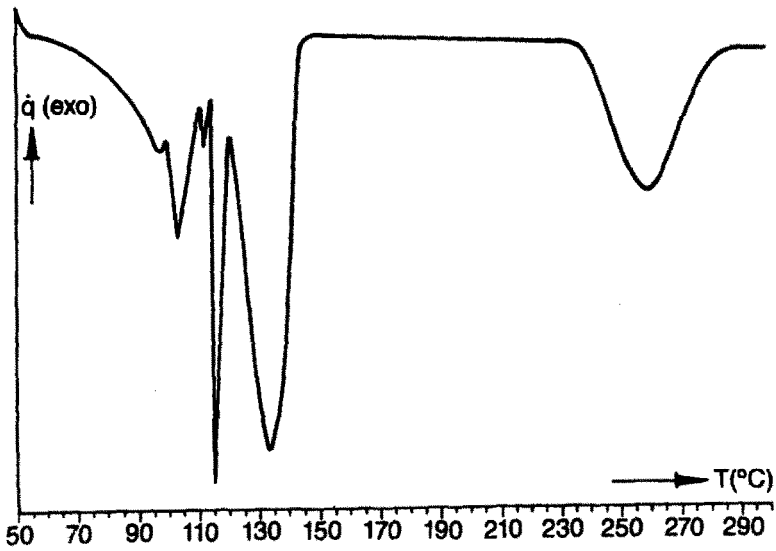

Figure 2. $CuSO_4.5H_2O$ in crucible with small hole in lid

The great difference between Figures 1 and 2 is striking.
It can easily be shown that all peaks are shifted towards a higher temperature, due to the higher relative vapour pressure of water. As the first peak is below 100 °C, water in the liquid state is formed, which boiles at 100 °C, giving rise to the second peak. The pattern between 100 and 120 °C is difficult to reproduce as it strongly depends on the sample preparation, in particular particle dimensions. Grinding the sample strongly influences this pattern and it can be shown easily that when the dimensions of the crystals increase, the peaks shift towards a higher temperature. This, of course is due to diffusion of water through the crystals, a process which can be demonstrated here in a very nice way.
Rather often, at temperatures of approx. 200 °C, an exothermal effect can be seen which seems hard to be explained. This effect is due to the solid state reaction between Al and $CuSO_4$. Adding finely divided Al powder increases this effect, measuring in an inert crucible causes the peak to disappear.
In this way, reactions between solids can be treated nicely.

Summarizing, it is shown here that also simple reactions have a large information content and prove the great potential of DSC in chemical education.

5. REFERENCES

1. G. van der Plaats et al., Thermochim. Acta, 151(1989) 319-322
2. G. van der Plaats, The practice of Thermal Analysis, Mettler-Toledo, 8606 Greifensee, Switzerland, 1991 (ME 724412, english version, ME 724411, german version)
3. Proficient Pac, Mettler-Toledo, 8606 Greifensee, Switzerland, 1991