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# THE DELTATHERM V DIFFERENTIAL THERMAL ANALYSIS AND THERMOGRAVIMETRY SYSTEM

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## ABSTRACT

An inexpensive, microcomputer controlled DTA/TG system, suitable for educational or small industrial laboratory applications, is described. The system consists of a microcomputer based controller to which a DTA or TG module can be attached. A convenient peak area integration mode in the software permits use of the DTA module for DSC emulation by quantitative $\Delta H_f$ determination as well as for other measurements. A digital baseline readout function similarly assists TG and heat capacity measurements.

## INTRODUCTION

The Deltatherm V system consists of a Model D8600 microcomputer controller coupled to either a Model D8100 DTA or Model D8900 TG module. The controller is based on a Commodore VIC-20 microcomputer and includes a built-in plotter as well as the programming ROMs for the module furnace and other functions. This system, which is designed primarily for educational and instructional use, and for use as a thermal analysis unit for the small industrial laboratory, is exceptional for its low-cost and ease of use. All of the module experimental parameters can be set before use by a user prompt interaction with the microcomputer which establishes the heating rate, DTA $\Delta T$ sensitivity, chart speed, temperature limits, memory for data storage, and so on. Data is stored with full dynamic range so nothing is ever "lost" during a run. A unique replot feature permits changing the $\Delta T$ sensitivity and/or baseline position to keep the experimental curve on the plotter chart.

We wish to describe this system here, using applications that are useful for educational institutions and small industrial laboratories.

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## DISCUSSION

The Deltatherm V system, with the attached DTA module, is shown in Fig. 1. The DTA or TG module is attached to the right side of the controller unit by a multiple-pin electrical connector. The controller unit contains a built-in Commodore 8-bit microcomputer programmed in Forth and a plotter that prints out on 4.5 in. wide paper. All commands to establish the instrument parameters are sent via the keyboard with menu prompts read from a printout of the plotter.

The specifications for the system are given in Table 1 while the heater and sample holder are illustrated in Fig. 2.

The heater consists of a 2.5 cm diameter by 3.6 cm in length Inconel steel block wound with a sheathed resistance heating element, which is usable to a maximum temperature of 1000 ° C. Sample and reference material temperatures are detected by chromel–alumel thermocouples welded to small nickel platforms, holding cups that are typically 5 mm in diameter by 2.5 mm in height. The cups are located symmetrically in the heater block. An insulated furnace jacket is placed around the heater unit during the thermal run to prevent heat loss to the ambient surroundings. For ease of sample handling, the sample is contained in stainless-steel or aluminum cups placed on the platform of the sample holder. Likewise, a reference material such as alumina can be placed into the reference holder. The furnace may be flooded with nitrogen or any other non-corrosive gas atmosphere through inlet and outlet connectors at the base of the module.


Fig. 1. Photograph of the Deltatherm V system with DTA/DSC module.

TABLE 1

Specification for the Deltatherm V system. Model D8600 controllers with D8100 DTA/DSC module or Model 8900 TG module

| Parameter | Description |
|---|---|
| *DTA/DSC Module* | |
| Temperature range | $-80$–$1000\,^{\circ}$C, automatic shut-off available in $1\,^{\circ}$C increments |
| Heating rate | 1–20 °C $\mathrm{min}^{-1}$ in 1 °C steps |
| Sensitivity | 1/4–16 °C $\mathrm{in.}^{-1}$ in 2:1 steps (0.1–6.4 °C $\mathrm{cm}^{-1}$) |
| Chart width | 4 in. (10 cm) usable, 4.5 in. (11.5 cm) actual |
| Chart speeds | 3–48 in. (7.6–122 cm) $\mathrm{h}^{-1}$, 2:1 steps |
| Time limit | 1 min increments to 24 h |
| Atmosphere | Control available at ambient pressure |
| Memories | Three provided for up to 16 in. (40 cm) of chart data and full dynamic data range |
| Data report | Can be made at any sensitivity and offsets up to 2 chart widths of suppression in either direction |
| Area under cover | Digital measurement with $\pm 1\%$ or better for 1 $\mathrm{in.}^2$ or larger |
| *TG module* | |
| Mass range | Up to 10 g capacity; limited by sample holder to about 1 g |
| Recorder range | 1–64 mg $\mathrm{in.}^{-1}$ (0.4–10.24 mg $\mathrm{cm}^{-1}$) in 7 steps of 2:1 for total recorder range of 4–256 mg full scale |
| Atmosphere | Control available at ambient pressure |
| Mass change | Digitial measurement with $\pm 1\%$ or better for 10 mg or more |

Two typical DTA curves, one for tin metal and the other for $CuSO_4 \cdot 5H_2O$, are illustrated in Fig. 3.

The fusion endothermic peak for tin is shown in Fig. 3A. This peak area was integrated by the "area-under-curve" command and used as a calibration point in the $\Delta H$ calibration curve. The heating rate was 10 °C $\mathrm{min}^{-1}$; temperature markers can be plotted on the chart at intervals of 10–100 °C, as set by the user. In the curves shown here, the markers are indicated at 50° intervals. For curve area integration applications, a 10 °C interval is recommended.

The DTA curve for the dehydration of $CuSO_4 \cdot 5H_2O$ to anhydrous $CuSO_4$ is also shown in Fig. 3. Three endothermic peaks were observed corresponding to the four well-known reactions

$$CuSO_4 \cdot 5H_2O(s) \rightarrow CuSO_4 \cdot 3H_2O(aq) + 2H_2O(l)$$

$$2H_2O(l) \rightarrow 2H_2O(g)$$

$$CuSO_4 \cdot 3H_2O(s) \rightarrow CuSO_4 \cdot H_2O(s) + 2H_2O(g)$$

$$CuSO_4 \cdot H_2O(s) \rightarrow CuSO_4(s) + H_2O(g)$$

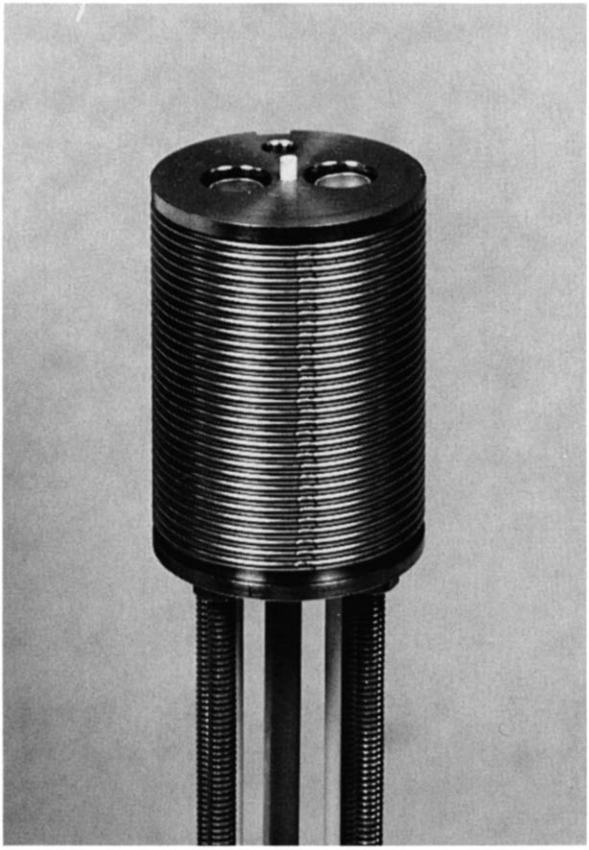

Fig. 2. Furnace and sample holder for the Deltatherm V system.

Peak areas for the three peaks could easily be integrated by the microcomputer.

For quantitative calorimetric measurements with the system, as well as for other DTA systems [1], the calorimeter response of the furnace and sample holder must be known. To do this, a series of reference samples (usually metals), with known thermal transition, were studied on the system. The calibration constant $K$, which is defined by the relationship

$$KA = \Delta H_t m$$

or

$$K = \frac{\Delta H_t m}{A}$$

where $\Delta H_t$ is the thermal transition (J), $m$ is the reactive sample mass (g) and $A$ is the peak area (in.$^2$). The constant $K$, has the units J in$^{-2}$. As small samples are involved, usually 2–5 mg, the units used here were mJ and mg.

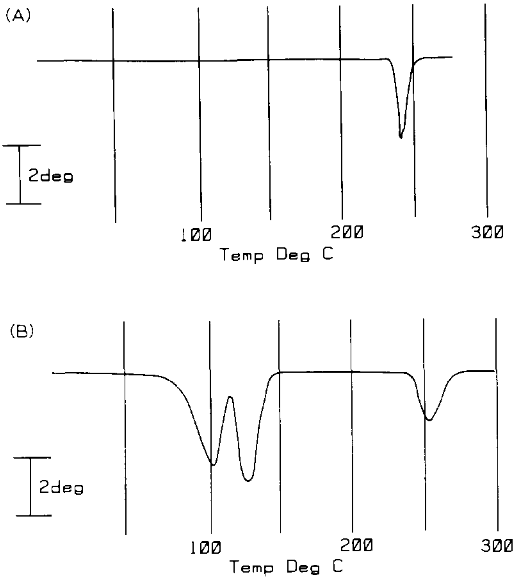


Fig. 3. DTA curves of: (A) tin metal; and (B) $CuSO_4 \cdot 5H_2O$. Heating rate of 10 °C $min^{-1}$ in a dynamic $N_2$ atmosphere.

Since the calorimetric response is dependent on temperature, three reference materials were used to calibrate the system, indium (m.p. 156 °C), tin (m.p. 232 °C) and lead (m.p. 327 °C). A typical calibration curve, $K$ versus temperature, is shown in Fig. 4. A linear relationship over the temperature range 50–400 °C was observed.

To illustrate the use of the quantitative measurement capability of the system, $\Delta H_t$ for the dehydration of $BaCl_2 \cdot 2H_2O(s)$, whose DTA curve is

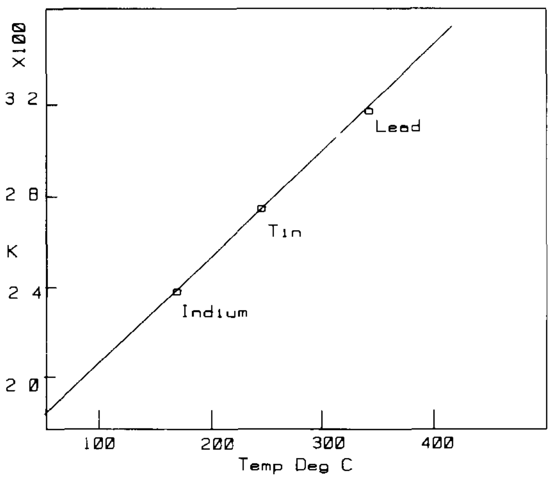


Fig. 4. A typical calorimetric calibration curve of $K$ versus temperature.

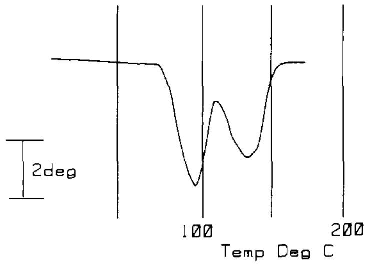


Fig. 5. DTA curve of $BaCl_2 \cdot 2H_2O(s)$; furnace heating rate of 10 °C $min^{-1}$ in a $N_2$ atmosphere.

shown in Fig. 5, was determined. Due to the two endothermic curve peaks overlapping, both peaks were integrated as a unit for the purposes of the determination. A $\Delta H_t$ value of 486 mJ $mg^{-1}$ or (486 J $g^{-1}$) was found; a value of 487.9 J $g^{-1}$ has been reported in the literature [2]. It is estimated that the precision and accuracy of the area measurements on the system are about ± 1%, for an area of 1 $in.^2$ or larger. This result appears to confirm this estimate.

## CONCLUSIONS

The Deltatherm V system is capable of a high level of performance over the temperature range − 80–1000 °C and instrument design parameters. Not only can the system be used for qualitative identification purposes, but due to the peak area mode, quantitative determinations such as $\Delta H_t$, mass of reactive material, and so on, can be made. The latter is an unusual feature for an inexpensive instrument. Also, the instrument is easy to use with system parameters being set by the user with the keyboard before each run. DTA data can never be "lost" because of the replot feature, which permits the replotting of data at different $\Delta t$ sensitivities. For educational purposes, the instrument is very simple for the student to use, and may find applications in undergraduate analytical instrumentation and physical chemistry courses. For the small industrial laboratory, the instrument is useful for quality control of raw materials or finished products, as well as analysis of samples containing DTA reactive materials.

## ACKNOWLEDGMENT

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