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THE DERIVATOGRAPH-PC A NEW TYPE OF PERSONAL COMPUTER-CONTROLLED DERIVATOGRAPH

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The Derivatograph-PC is an instrument for simultaneous TG, DTG, DTA, TD and EGA investigations, using non-isothermal, isothermal and quasi-isothermal heating techniques. This is the latest type in the series of derivatographs. It is operated by means of an IBM-compatible personal computer.

The method of simultaneous thermoanalytical measurements dates back to nearly four decades [1, 2]. The Derivatograph was the first in series produced instrument operating on the above principle [3]. The newest development of this series is the Derivatograph-PC.

This instrument is suitable for the simultaneous recording of thermogravimetric (TG), on line derivative thermogravimetric (DTG), and differential thermoanalytical (DTA) [1, 2], furtheron the thermogastitrimetric (TGT) [1, 2, 4-7] and thermodilatometric (TD) [1, 2, 5, 8] curves.

It is possible to measure the TG and TD curves simultaneously on a single sample [1, 5].

With the help of an adapter, the release of water (WD) can also be detected simultaneously, independently of the other gaseous decomposition products [9]. The device can calculate and record the DDTA, DTGT and DTD curves as first derivatives, and the DDTG, DDTGT and DDTD curves as second derivatives respectively.

A practical example can be seen in Figs 1 and 2. The curves in these Figures show the course of decomposition of calcium oxalate monohydrate,

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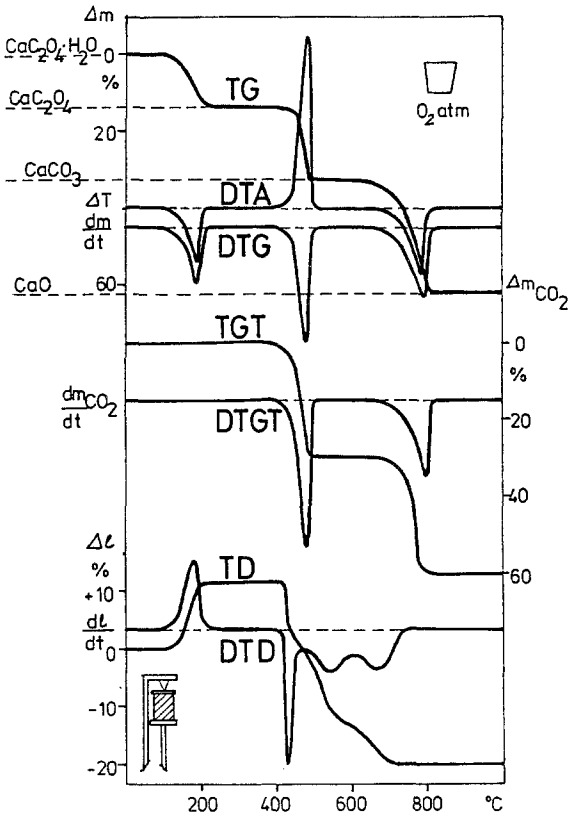

Fig. 1 Decomposition of $CaC_2O_4 \cdot H_2O$. Heating non-isothermal; sample holder: open crucible; in the case of TD: a pressed test piece; atmosphere: O_2 carrier gas

investigated by non-isothermal heating in an open crucible, and in air and nitrogen atmospheres, respectively.

Table 1 Hardware difference between the two latest type of derivatograph

	Derivatograph-C	Derivatograph-PC
Memory	64 Kbyte	640 Kbyte
Clock fr.	3 MHz	10 MHz
Floppy d.	8' - 540 Kb	5.25' - 1.2 Mb
Hard disc	---	Possibility
Monitor	Monochrom	Color

The Derivatograph-PC is also suitable for investigations under quasi-isothermal and quasi-isobaric circumstances [1, 10-12]. The course of

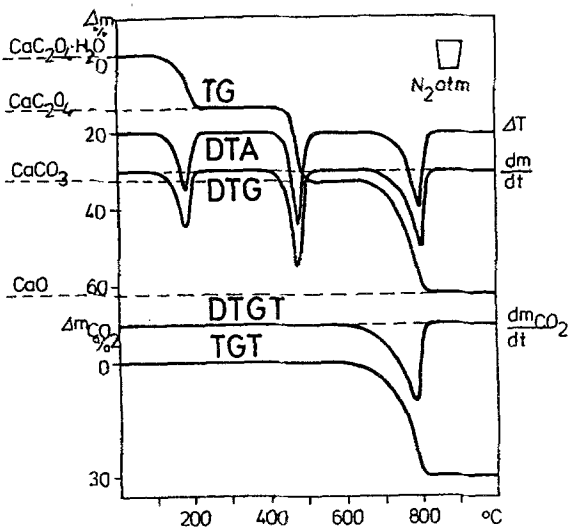

Fig. 2 Decomposition of $CaC_2O_4 \cdot H_2O$. Heating: non-isothermal; sample holder: open crucible; atmosphere: N_2 carrier gas

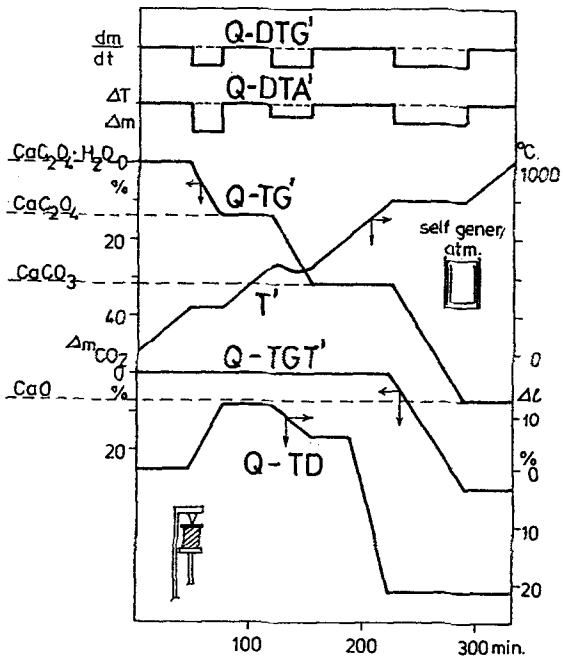

Fig. 3 Decomposition of $CaC_2O_4 \cdot H_2O$. Heating: non-isothermal; sample holder: labirinth crucible; in the case of Q-TD: a pressed test piece; atmosphere; O_2 carrier gas

decomposition of $CaC_2O_4 \cdot H_2O$ under these conditions and as a function of time are shown in Fig. 3. The simultaneous Q-TG, Q-DTG and Q-DTA curves [11, 12], the simultaneous Q-TG and Q-TGT [1, 12, 13] and the Q-TD curves [1, 12, 14] in Fig. 4 are drawn as functions of the sample temperature. In the latter two cases, the labyrinth crucible was applied, giving rise to a self-generated atmosphere [1, 5, 12].

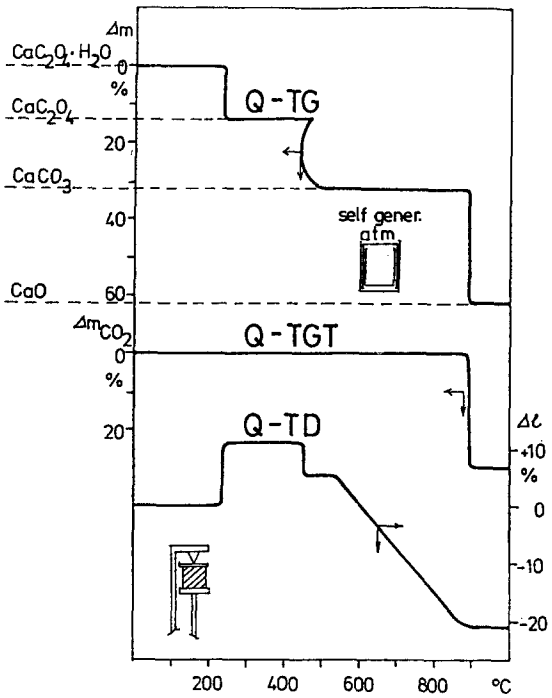

Fig. 4 Decomposition of $CaC_2O_4 \cdot H_2O$. Heating: quasi-isothermal; sample holder: labirinth crucible; atmosphere: O_2 carrier gas

If necessary, an oxygen-free, pure gas atmosphere [1] or an atmosphere with controlled composition [15] can also be attained. The performance of investigations under isothermal circumstances [16] is likewise possible.

The operating principle of the instrument is illustrated in Fig. 5.

The basic differences between the two latest types, the Derivatograph-C [16-18] and the Derivatograph-PC, are that the latter includes an IBM-compatible PC/XT (parts 23-26 in Fig. 5) computer, and the fact that the structure and function of the interface (part 27 in Fig. 5) have been changed.

The hardware differences between the Derivatograph-C and the Derivatograph-PC are summarized in Table 1. In the Derivatograph-C the software is burned into the ROM (32 Kb), while in the Derivatograph-PC it is on a floppy disc (part 25 in Fig. 5). The analog system consists of one ICL 5216 eight-channel multiplexer (Intersil) and one ICL 7104 14-bit analog - to - digital converter (monolithic) instead of the "intelligent data collector" (Parts 28 and 29 in Fig. 5). The eight analog channels are: D. S. (deviation signal for balance control), T, DTA, TD, TGT and T' (for heating control); two channels remain empty, and are suitable for connection to various other instruments. However, in this case it is necessary to use amplifiers, because the AD converter works in the range of ± 4.5 volts). Part 30 in Fig. 5 is a port driver card with 48 output and 8 input digital lines.

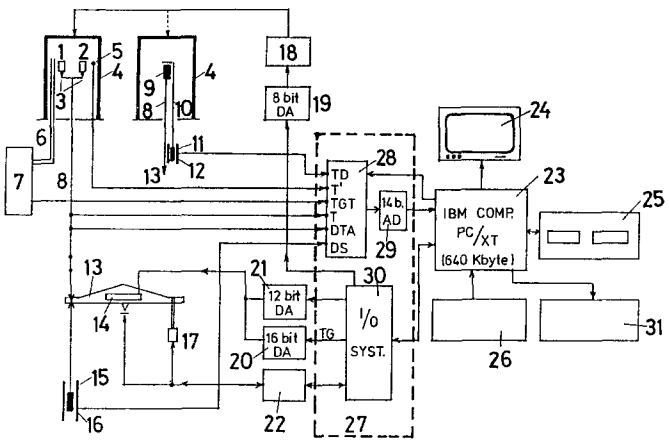

Fig. 5 Block diagramme of Derivatograph-PC, 1 - sample; 2 - reference material; 3 - counter connected thermoelements; 4 - furnace; 5 - heating controller thermoelement; 6 - gas exhoust tube; 7 - thermogastitrimetric equipment; 8 - corundum tube; 9 - compressed sample for TD examination; 10 - corundum rod; 11 - linear differential transformer; 12 - iron core; 13 - balance; 14 - coil moving in permanent magnetic field; 15 - linear differential transformer for balance control; 16 - iron core; 17 - electromechanical rough taring device; 18 - heating controller system; 19 - 8-bit DA converter; 20 - 15-bit DA converter; 21 - 12-bit DA converter; 22 - balance servis system; 23 - IBM; 26 - keyboard; 27 - interface; 28 - analog multiplexer; 29 - 14-bit monolithic analog to digital converter; 30 - I/O port driver card; 31 - printer plotter

The thermal curve can be engineered on the screen for the recording; the curves can be processed from many points of view. The possibility of the wide-ranging processing program of the Derivatograph-C [14, 16] remains

unchanged. After processing, the thermal curve can be plotted by means of a printer-plotter (EPSON FX-1050, part 31 in Fig. 5) in any size up to the limit of the chart.

Since the mechanism of the decomposition of $CaC_2O_4 \cdot H_2O$ is well known, this example was selected for sake of demonstration (Figs 1-4). The aims are to demonstrate the information-multiplying effect of the simultaneous measuring techniques [1, 3], and to prove that, with conscious changes of the experimental conditions between wide limits, this information-multiplying effect can be further increased, thereby facilitating the interpretation of the kinetics and mechanisms of complicated and complex reactions.

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On the basis of our instructions, the prototype of the equipment was developed in the Institute for General and Analytical Chemistry (Technical University of Budapest) in cooperation with the Hungarian Optical Works. The software was designed by P. Somogyvári, E. Cilják, Z. Baki and L. Hegedüs. The computer hardware was prepared by I. Németh, E. Imre and T. Tóth. The equipment is produced by the Hungarian Optical Works, Budapest, Hungary, H-1525, P.O.Box 52.

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Zusammenfassung – Derivatograph-PC ist ein Gerät für die Durchführung von simultanen TG, DTG, DTA, TD and EGA Untersuchungen unter Anwendung von nicht-isothermen, isothermen und quasi-isothermen Aufheizungsmethoden. Dieses Gerät ist der neueste Typ in der Serie der Derivatographen. Die Einrichtung funktioniert mit Hilfe von einem "IBM compatible personal Computer".