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Note

DERIVATOGRAPH-C: A MICROCOMPUTER AUTOMATED EQUIPMENT FOR SIMULTANEOUS TG, DTG, DTA, EGA AND TD

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Derivatograph-C is suitable for the simultaneous recording of thermogravimetric (TG), derivative thermogravimetric (DTG), thermogastitrimetric (TGT) and either differential thermoanalytical (DTA) or thermodilatometric (TD) curves [1]. Thermogastitrimetry (TGT) is one of the evolved gas analytical (EGA) techniques [1–3]. If necessary, the device can also display graphically the DDTG, DDTA, DTGT and DTD curves, i.e. curves to be obtained by deriving the primary curves.

The results obtained by another foreign apparatus can also be processed by the equipment simultaneously, because one of its channels can be made free for the reception of a signal moving between 0 and 5 V.

The measuring domains of the semi-microelectronic and automatic balance of the equipment can be chosen between zero and 5, 10, 20, 50 or 100 mg, respectively. The balance operates over a raw and a fine taring system, both operating automatically.

Experiments can be carried out in different kinds of gas atmosphere. The apparatus is equipped with furnaces with Kanthal and graphite heating elements. These can be heated up to 1200 and 1600°C, respectively. The graphite furnace can be operated by continuous flushing with high-purity argon gas.

The temperature of the furnaces can be regulated by three heating techniques of different character (linear, isothermal, and quasi-isothermal). In the case of linear heating any heating rate can be chosen up to the 20°C $\min^{-1}$ limit. In the framework of a single isothermal heating program eight isothermal periods of any temperature and length can be realized. These isothermal sections can be connected by programmed periods of various heating or cooling rates. In the case of the quasi-isothermal heating programme the rate of transformation can be regulated by the signal of either the DTG, the DTA or the DTD curves. Accordingly, the examination can be carried out in such a way that the rate of change in either the weight [3–5], enthalpy [4] or linear size of the sample [3,6] should be very small, strictly constant and of a defined magnitude.

In the course of the examination, balance-control, regulation of heat, periodical sampling of the signals observed, bunching of their values and loading into the memory, as well as the continuous drawing of the curves on the display are all performed by the microprocessor.

After measurement, the data can be stored on a disk plate from which they can be recalled at any time.

For data processing and data handling a rich software program is designed with the help of which, e.g, weight change, linear changes in size, titrant consumed, etc., can be stated as a function of time or temperature. The curves can be multiplied, divided, increased or diminished by constant values, like chemical factors. Further possibilities are: addition or subtraction of curves of identical character obtained in two different examinations (e.g. TG and TGT); conversion of TG and TGT curves into percent or mole number; determination of on-set, off-set and peak temperatures as well as peak area and peak symmetry of the DTA curve; deriving and smoothing of curves; on-line correction of the baseline of DTA and TG curves, necessary in the case of very small samples; calculation of virtual kinetic parameters; curves can be shifted along the x and y axes; selected sections of the curves can also be separately enhanced according to choice in the directions of both the x and y axes.

The curves transformed by means of the necessary operations mentioned can be ordered side by side so that the diagram can be constructed on the display before it is drawn by the plotter to the desired size. After drawing the curves the plotter would automatically draw the scale division and scale values of the x and y axes. By means of the plotter, inscriptions of numbers or text can be made on any part of the diagram.

By means of the printer the data sheet can be printed. The measuring data of any curve can be prepared between the desired limits and the requested fullness of detail.

Before every measurement, every circuit can be separately calibrated and the factors obtained can be marked on the data sheet. The bunched data multiplied by the factors can be loaded into the memory.

The double floppy drive, in addition to tracing and handling, also facilitates deletion of data stored on the disk.

The running of the apparatus is simple. The software helps the user to establish the program of the experiment by a question–answer format. Handling of the apparatus does not require any previous computer-technical experience.

Figures 1 and 2 show the original diagrams taken of the decomposition process of a gibbsite examined by the quasi-isothermal measuring technique [3–5] as a function of time (Fig. 1) and as a function of sample temperature (Fig. 2), respectively. The transformation took place at a rate of 0.1 mg min^{-1}. In the constant weight interval the temperature rose at a rate of 1.5 °C min^{-1}. The weight of the sample was 104.3 mg. Sample and reference

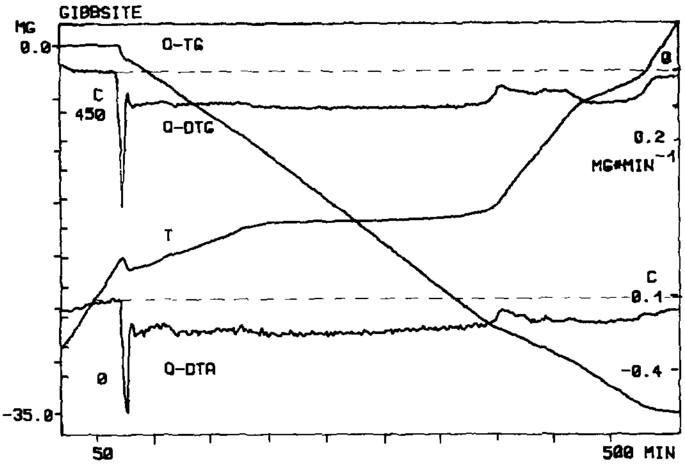

Fig. 1. Decomposition of gibbsite as a function of time.

material (Al_2O_3) were located in two closed so-called conical crucibles [4].

The decomposition of gibbsites had earlier been investigated [7] by means of the quasi-isothermal–quasi-isobaric measuring technique and by X-ray diffraction. It had been found that the transformation is composed of three decomposition processes:

$$\alpha\text{-Al(OH)}_3 \rightarrow \alpha\text{-AlOOH} + H_2O \qquad (1)$$

$$2\alpha\text{-Al(OH)}_3 \rightleftharpoons \chi\text{-Al}_2O_3 + 3H_2O \qquad (2)$$

$$2\alpha\text{-AlOOH} \rightleftharpoons \gamma\text{-Al}_2O_3 + H_2O \qquad (3)$$

The reactions (1), (2) and (3) in the Q-TG curve of Fig. 2 the non-isother-

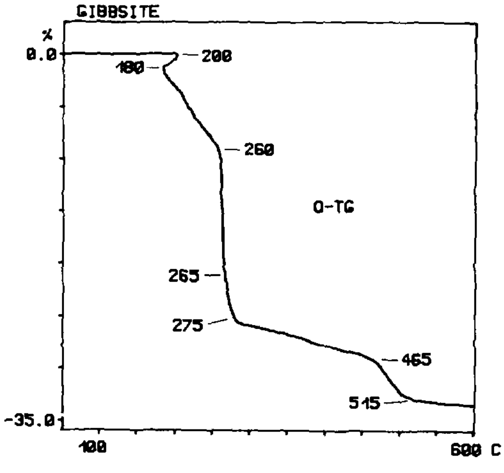

Fig. 2. Decomposition of gibbsite as a function of temperature.

mal period between 180 and 260°C, the vertical isothermal interval between 260 and 275°C and the non-isothermal section between 465 and 515°C correspond in numerical order.

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