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DERIVED DIFFERENTIAL THERMAL CURVES*

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Under certain routine conditions where, for example, original grain size of particles, the presence of other mineral constituents, and certain chemical impurities are known, differential thermal techniques can be applied in a semi-quantitative manner. Greatest accuracy is obtained by measuring the area under portions of the curve representing certain kinds of reactions: loss of absorbed or other kinds of water, decomposition, inversion from one polymorphous form to another, and recrystallization. Ordinarily, the loss of water (and perhaps atomic oxygen) as well as decomposition reactions do not start at a sharply defined temperature, but are first represented by a gradual departure of the differential thermal curve from baseline. This gradual drift from baseline often grades imperceptibly into the portion of the curve representing the important part of the reaction which is used for quantitative measurement. If the operator cannot accurately select the point where the important part of the reaction started, it is impossible to accurately position a baseline from which to delimit the area under the curve. Small changes in the positioning of the baseline result in large changes in the measured area under the curve so, under these conditions, the technique degenerates to only a rough, semi-quantitative procedure.

Baseline drift can often be controlled, when carbonate minerals or rocks are being investigated, by the use of a CO₂-atmosphere in the furnace (Rowland and co-workers, 1951 and 1952). This technique, however, does not help in systems where carbonate minerals are not present: when clay and other silicate systems are being investigated.

The purpose of this note is to call attention to a technique long used by the metallurgist to help resolve this difficulty. The metallurgist employs heating or cooling curves to help construct equilibrium diagrams and, in particular, to locate inversion temperatures in steels and other

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systems. They also have applied the differential thermal method to their problems.

The first derivative of a differential thermal curve often provides very useful information. Slight changes in the slope of a gradually drifting curve, or reversals in slope, are represented by sharp changes in the direction of a curve representing the first derivative of the differential thermal curve. To avoid using the word "differential" twice, this second curve might best be called a derived differential thermal curve. The use of derived differential thermal curves was proposed by Walter Rosenhain and is described in detail by Foote, Fairchild and Harrison (1921, pp. 200-203).

In Fig. 1, *B* represents the differential thermal curve for a typical kaolinite; curve *A* is the first derivative or derived differential thermal curve for the same kaolinite specimen.

A derived differential thermal curve can be graphically constructed in approximately 10 minutes after a little practice. If a galvanometer

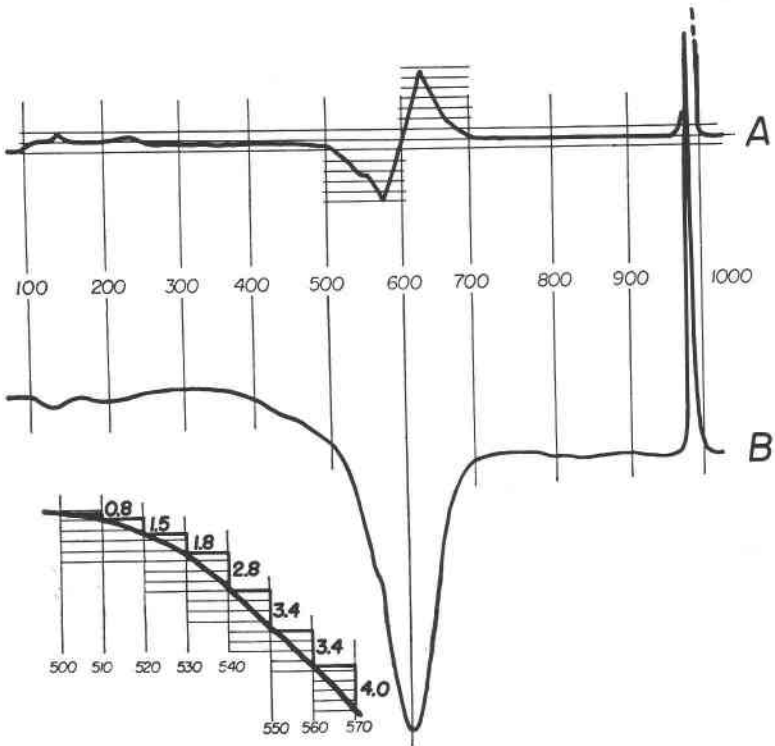


FIG. 1. Differential thermal curve (*B*) of a sample of kaolinite; (*A*) represents the first derivative or the derived differential thermal curve for the same sample.

record is obtained (instead of one traced by an electronic recorder on graph paper) it is most convenient to first transfer it to some convenient graph or ruled paper.

The method of obtaining the derived curve consists of observing successive values of change of temperature ($T - T'$) for equal increments of T . A convenient increment is 10° C. This is expressed as $\Delta(T - T')/\Delta T$ where ΔT is taken as 10° C. The derived differential is the graph obtained by plotting the temperature differences against $T: d(T - T')$ vs. T .

The inset in Fig. 1 represents an enlargement of a portion of curve B . The differences in temperature (represented by any convenient unit) between the kaolinite sample and the standard, on heating from 500 to 570° C., are $0.8, 1.5, 1.8, 2.8$, etc., units for each 10° increase in temperature. These values are plotted against $500, 510, 520, 530^\circ$ C., etc., respectively.

The slight "bump" on the large endothermic peak (curve B) plots as a straight horizontal line (between 550 and 560° C.) in the derived curve. The base line under the two peaks of the derived curve can be objectively drawn with precision. Apparently the important part of the reaction starts at 500° C. The area to be measured under this peak can be delimited by extending the straight-line portion of the derived curve (from 500 – 550° C.) back to the base line.

Two independent operators can select a base line and duplicate the measured areas under the curve within 3 per cent. Usually the differential curve obtained from two portions of the same sample check rather closely, consequently, when suitable samples are chosen to construct an area vs. quantity-of-mineral-present curve, the derived differential technique greatly improves the accuracy of the method.

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INTRODUCTION

Between Northfield and Cannon Falls, Minnesota, some 40 miles south of St. Paul, on State Highway 19, mesas rise to heights of 60 feet or more above the preglacial Cannon River Valley flood plain (1). These prominences, when traced by aerial photographs, are seen to pass southward