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NOMENCLATURE COMMITTEE†

**NOMENCLATURE FOR THERMAL
ANALYSIS—II & III**

(Recommendations 1979)

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NOMENCLATURE FOR THERMAL ANALYSIS—II & III

INTRODUCTION

It is noted with great satisfaction by the Commission that the first rules approved in 1973 and published in Pure and Applied Chemistry, 37, 439 (1974) have been completed by further reports prepared by the Nomenclature Committee of the International Confederation for Thermal Analysis (ICTA).

Since compiling the first report, the ICTA Committee have actively been considering other aspects of thermal analysis that require attention. They have produced two further reports which appeared in Talanta, 19, 1079 (1972) and in J. Therm. Analysis, 8, 197 (1975).

Both reports have been duly considered at various meetings of the present Commission which endorses the following integration of the above reports and recommendations made by the ICTA.

1. AMPLIFICATION OF FIRST REPORT

Because of the variety of opinions expressed on, and the different interpretations of, the term pyrolysis, the Committee considered the time inopportune to promulgate on thermal decomposition and related terms - see First Report, Section I (f).

Some confusion appears to have arisen over the term isobaric weight-change determination as defined in the First Report, Section IIIB, Sub-section 1. The Committee consider that this confusion could be obviated by the following statement.

In the context of the report published in Talanta, 16, 1227 (1969) Section IIIB, Sub-sections 1 and 2 headed, respectively, Static and Dynamic, these terms refer to environmental temperature. It should be noted that the same terms are also used with reference to environmental atmosphere.

The definition of differential scanning calorimetry in the First Report applies only when power-compensation instruments are used and no definition has been proposed to cover the use of heat-flux instruments: in French the two have been clearly distinguished. The Committee also recognize that there has over the past few years been increasing use of differential thermocouples for measurements under isothermal external conditions. It is therefore recommended that the following two terms and definitions be added to those in the First Report:

Quantitative differential thermal analysis (quantitative DTA).

This covers those uses of DTA where the equipment is designed to produce quantitative results in terms of energy and/or other physical parameters. The record should be plotted in the same manner as a normal DTA curve.

Differential thermal analysis (DTA) in an isothermal environment.

A variant of DTA in which the temperature difference between a substance and a reference material is continuously recorded vs time as the two specimens are maintained in a nominally isothermal environment. The record should be plotted in the same manner as a normal DTA curve.

The term isothermal DTA is incorrect and the abbreviation QDTA is considered not to be warranted.

In view of recent developments, the brief comments on multiple techniques, simultaneous and combined, in the First Report require clarification and the following names and definitions have been approved.

Simultaneous techniques. This term covers the application of two or more techniques to the same sample at the same time - e.g. simultaneous thermogravimetry and differential thermal analysis.*

Coupled simultaneous techniques. This term covers the application of two or more techniques to the same sample when the two instruments involved are connected through an interface† - e.g. simultaneous differential thermal analysis and mass spectrometry.

Discontinuous simultaneous techniques. This term covers the application of coupled techniques to the same sample when sampling for the second‡ technique is discontinuous - e.g. discontinuous simultaneous differential thermal analysis and gas chromatography, when discrete portions of evolved volatile(s) are collected from the sample situated in the instrument used for the first‡ technique.

II. DTA AND TG APPARATUS AND TECHNIQUE

In considering the terms available, certain arbitrary choice have had to be made - e.g. between specimen and sample - but the only term in fairly common use rejected is inert material.

A. DTA

The sample is the actual material investigated, whether diluted or undiluted.

The reference material is a known substance, usually inactive thermally over the temperature range of interest.

The specimens are the sample and reference material.

The sample holder is the container or support for the sample.

The reference holder is the container or support for the reference material.

The specimen-holder assembly is the complete assembly in which the specimens are housed. Where the heating or cooling source is incorporated in one unit with the containers or supports for the sample and reference material, this would be regarded as part of the specimen-holder assembly.

A block is a type of specimen-holder assembly in which a relatively large mass of material is in intimate contact with the specimens or specimen holders.

* In writing, the names of simultaneous techniques should be separated by 'and' when used in full and by a hyphen when abbreviated acceptably - e.g. simultaneous TG-DTA. Unless contrary to established practice, all abbreviations should be written in capital letters without full-stops.

† A specific piece of equipment that enables two instruments to be joined together.

‡ In coupled simultaneous and discontinuous techniques, the first technique to be mentioned is that in which the first, in time, measurement is made - e.g. when a DTA instrument and a mass spectrometer are connected through an interface, DTA-MS is the correct form, not MS-DTA.

The differential thermocouple* or ΔT thermocouple* is the thermocouple* system used to measure temperature difference.

B. TG

A thermobalance is an apparatus for weighing a sample continuously while it is being heated or cooled.

The sample is the actual material investigated, whether diluted or undiluted.†

The sample holder is the container or support for the sample.

C. DTA and TG

The temperature thermocouple* or T thermocouple* is the thermocouple* system used to measure temperature; its position with respect to the sample should always be stated.

The heating rate is the rate of temperature increase, which is customarily quoted in degrees per minute (on the Celsius or Kelvin scales). The heating or cooling rate is said to be constant when the temperature/time curve is linear.

In simultaneous DTA-TG, definitions follow from those given for DTA and TG separately.

III. DTA AND TG CURVES

The Committee, in reaffirming their decision to use the terms differential thermal curve or DTA curve, thermogravimetric curve or TG curve and derivative thermogravimetric curve or DTG curve, recommend disuse of the other terms which have appeared in the literature, such as thermogravimetric analysis curve, thermolysis curve, thermoweighing curve, thermogravigram, thermoponderogram, thermogram, differential thermogravimetric curve, differential thermogram, derivative thermogram, polytherm, etc.

Certain conventions and reporting procedures for DTA and TG curves have already been specified in Analyt. Chem., 39, 543 (1967), and in the First Report. The following definitions are to be read in conjunction with these recommendations.

A. DTA

In DTA it must be remembered that although the ordinate is conventionally labelled ΔT the output from the ΔT thermocouple will in most instances vary with temperature and the measurement recorded is normally the e.m.f. output, E - i.e. the conversion factor, b , in the equation $\Delta T = bE$ is not constant since $b = f(T)$. A similar situation occurs with other sensor systems.

All definitions refer to a single peak such as that shown in Fig. 1: multiple peak systems, showing shoulders or more than one maximum or minimum, can be considered to result from superposition of single peaks.

The base line (AB and DE, Fig. 1) corresponds to the portion or portions of the DTA curve for which ΔT is approximately zero.

A peak (BCD, Fig. 1) is that portion of the DTA curve which departs from and subsequently returns to the base line.

* Should another thermosensing device be used, its name should replace thermocouple.

† Samples used in TG are normally not diluted, but in simultaneous TG-DTA diluted samples might well be used.

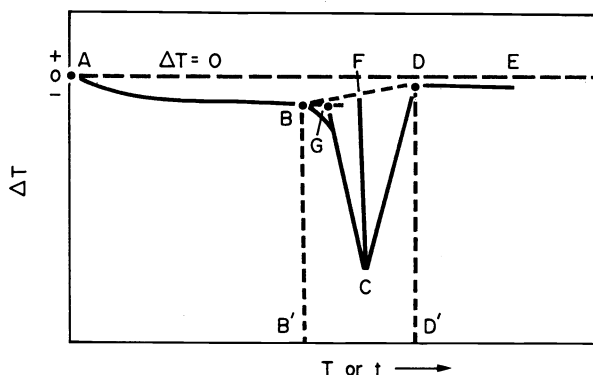


Fig. 1 Formalized DTA curve

An endothermic peak or endotherm is a peak where the temperature of the sample falls below that of the reference material; that is, ΔT is negative.

An exothermic peak or exotherm is a peak where the temperature of the sample rises above that of the reference material; that is, ΔT is positive.

Peak width ($B'D'$, Fig. 1) is the time or temperature interval between the points of departure from and return to the base line.*

Peak height (CF , Fig. 1) is the distance, vertical to the time or temperature axis, between the interpolated base line* and the peak tip (C , Fig. 1).

Peak area ($BCDB$, Fig. 1) is the area enclosed between the peak and the interpolated base line.*

The extrapolated onset (G , Fig. 1) is the point of intersection of the tangent drawn at the point of greatest slope on the leading edge of the peak (BC , Fig. 1) with the extrapolated base line (BG , Fig. 1).

B. TG

All definitions refer to a single-stage process such as that shown in Fig. 2: multi-stage processes can be considered as resulting from a series of single-stage processes.

* There are several ways of interpolating the base line and that given in Fig. 1 is only an example. Location of points B and D (Fig. 1) depends on the method of interpolation of the base line.

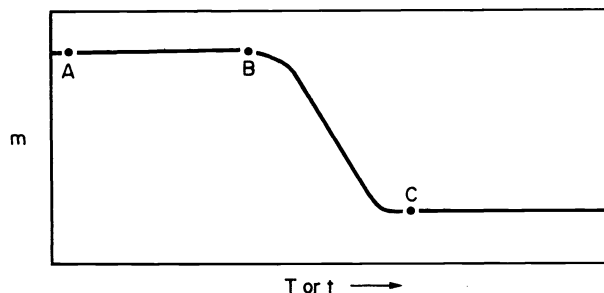


Fig. 2 Formalized TG curve

A plateau (AB, Fig. 2) is that part of the TG curve where the mass is essentially constant.

The initial temperature, T_i , (B, Fig. 2) is that temperature (on the Celsius or Kelvin scales) at which the cumulative mass change reaches a magnitude that the thermobalance can detect.

The final temperature, T_f , (C, Fig. 2) is that temperature (on the Celsius or Kelvin scales) at which the cumulative mass change reaches a maximum.

The reaction interval is the temperature difference between T_f and T_i as defined above.