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THE NEW METTLER TOLEDO ULTRAMICRO THERMOBALANCE TGA850

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Abstract

A thermogravimetric unit is described having horizontal furnace arrangement, a temperature range of up to 1100°C and unsurpassed dynamic weighing range from 0 to 2 g at 0.1 μg resolution. Options of the system are sample robot, gas controller, high temperature furnace and hyphenated EGA techniques.

In addition to the TG, the SDTA™ curve allows the detection of endo- or exothermal effects (analogous to DTA or DSC) to a resolution of 5 mK.

Keywords: DTA, hyphenated technique (EGA), sample robot, TG, thermobalance, SDTA

The new compact TGA850 measuring cell incorporates state of the art METTLER TOLEDO weighing technology, an ultramicro balance sampling 20 million points in the range of 2 g giving a resolution of 0.1 μg. There is no

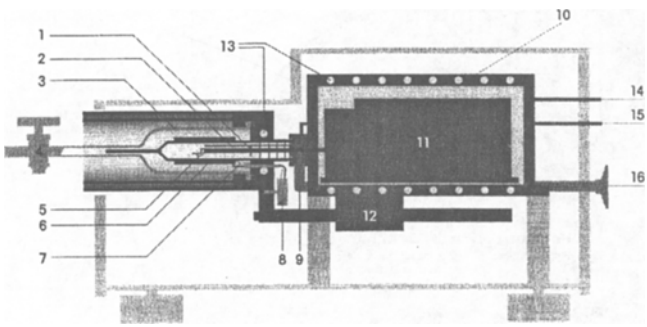

Fig. 1 Schematic drawing of the new TGA850 module. Keys to illustrate of the main parts: Balance (11) with thermostated housing (10) and protective gas purge (14), alumina furnace (6) with temperature sensor (7) and fused silica jacket (3) with baffles (1) and reflectors. Horizontal sample holder and support (5) with sample temperature sensor. Reactive gas capillary (2), gas outlet with stopcock or link to EGA (e.g. MS, FTIR, GC) (4)

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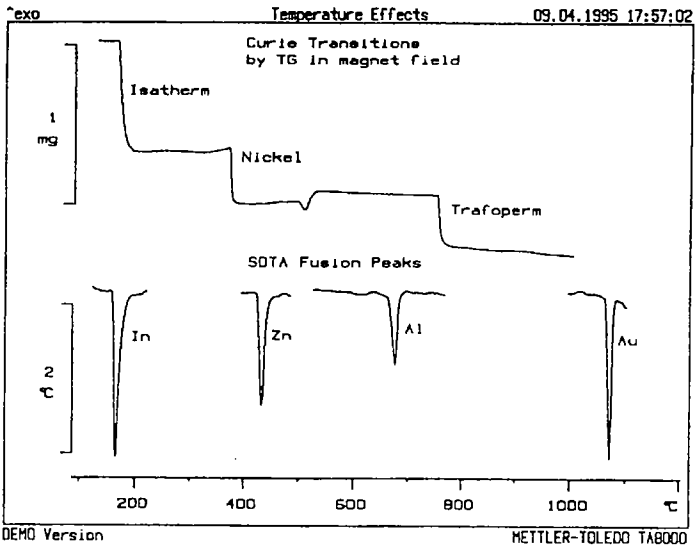

Fig. 2 Ferromagnetic samples (e.g. nickel or special alloys) exposed to a nonhomogeneous magnet field can be used for the traditional temperature calibration of TGA modules. The more accurate temperature calibration is based on SDTA melting curves of pure metals (e.g. indium, gold)

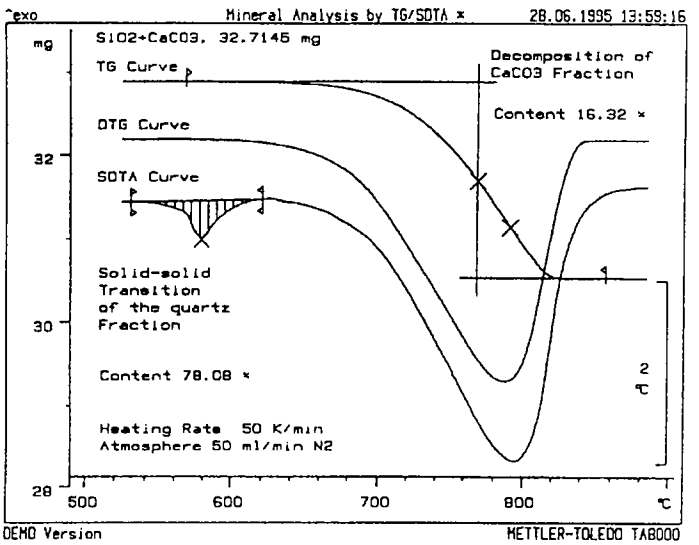

Fig. 3 A typical application of simultaneous SDTA/TGA measuring technique: Physical and chemical transformations without mass change are visible at a glance by comparing DTG and SDTA curves. Many building materials and rocks contain $CaCO_3$ besides SiO_2. The solid-solid transition is proportional to the quartz content (78%) followed by the carbonate decomposition indicating a content of 16.3%

range/resolution switching depending on the amount of sample. Thanks to the parallel guided ultra micro balance, position changes of the sample have no influence on the TG curve.

The temperature range goes from ambient to 1100°C. The sample temperature is determined directly at the crucible. In conjunction with the automatic temperature calibration using melting points of ultra pure metals a temperature accuracy of ±0.25°C is reached (Fig. 2).

A DTA curve, named SDTA™, single Differential Thermal Analysis, is obtained by means of a mathematically modelled reference temperature, but having only one sample crucible and the thermocouples for the furnace and the sample. In addition to TG, the SDTA allows the detection of endo- or exothermal effects (similar to heat flow DSC) to a resolution of 5 mK. Simultaneous measurement of the TG and DTA curves facilitates the interpretation appreciably (Fig. 3).

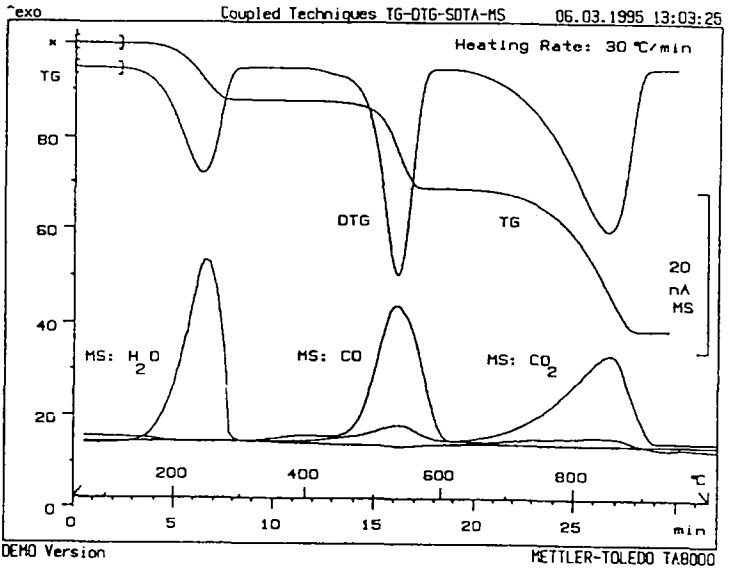

Fig. 4 Calcium oxalate monohydrate exhibits three endothermal decomposition steps if measured in argon. The mass spectrometer BALZERS MS-Cube™ is coupled to the TGA850 by a heated capillary. The three simultaneously measured ion current curves for water, carbon monoxide and carbon dioxide explain the course of the splitting reactions

The TGA850 with its horizontal furnace is vacuum tight and has a positive pressure outlet for evolved gas analysis, e.g. a coupling to a mass spectrometer for identification of decomposed products (Fig. 4).

The furnace opens and closes motor driven, a sample robot for up to 34 samples is a convenient option to run also routine analysis with the same precision and accuracy as developments in R&D. In routine, samples with mois-

ture may have to be stored during several hours on its position in the sample robot. to prevent substantial loss of volatiles, the crucibles can be fitted with a lid, which is removed or punched by the robotic system just before measurement.

The new TGA850 module is operated by the TA Station and is a new member of TA8000 family. A large number of software options (i.e. automation functions, mathematical functions, *n*-th order and the new model free kinetics, alternating techniques also for TG) will add to the already highest performance of the TGA850 module for top applications in thermogravimetry.