

You can rely on METTLER TOLEDO

Temperature data:			
Temperature range	Room temperature – 1100 °C / 1600 °C		
Temperature accuracy	±0.25 °C (SF),	±0.3 °C (LF1100)	±0.5 °C (LF1600)
Temperature reproducibility	±0.15 °C (SF)	±0.2 °C (LF1100)	±0.3 °C (LF1600)
Heating rate Room temperature –1100 °C	5 min (SF)	8 min (LF1100)	10 min °C (LF1600)
Cooling rate 1000–100 °C	20 min (SF)	22 min (LF1100)	25 min (LF1600)
Balance data:			
Measurement range	1 g or 5 g		
Resolution	1.0 µg or 0.1 µg (without range switching)		
Noise (RMS)	<1 µg (SF)	<1.5 µg (LF1100)	< 2 µg (LF1600)
SDTA™ (Single Differential Thermal Analysis) data:			
SDTA™ resolution	0.005 °C		
SDTA™ noise (RMS)	0.01 °C		
SDTA™ sensor type	RTyp thermocouple (Pt-Pt/Rh 13%)		
SDTA™ signal time constant	15 s (without crucible)		
Scanning:			
Scanning rate	max. 10 values per second		
Approvals:			
Electric safety	S+, CSA, EN61010-1, C22.2 No. 1010.1-92		
Electromagnetic compatibility	EN55011, FCC Part 15J, EN50082-1		
Disturbances in supply systems	EN 60555-2, EN60555-3		
Conformity mark	CE		

SF: Small furnace, LF1100: Large furnace up to 1100 °C, LF1600: Large furnace up to 1600 °C

Ask for our product information on material characterization

For the determination of thermal values such as melting, boiling, dropping and cloud points, we offer a large selection of system combinations. Various measuring cells (DSC, TGA, TMA, TOA) and software for thermal analysis are available.

UserCom

Regular information concerning TA-Tips, News and applications.



Quality certificate

Development, production and testing following ISO9001.



Worldwide service

Our large service network is one of the best in the world and ensures the maximum availability and service life of your product.



METTLER TOLEDO on the Internet

You will quickly find easily surveyed, essential information on our products and services and about our company under ...<http://www.mt.com>.



Because we care.

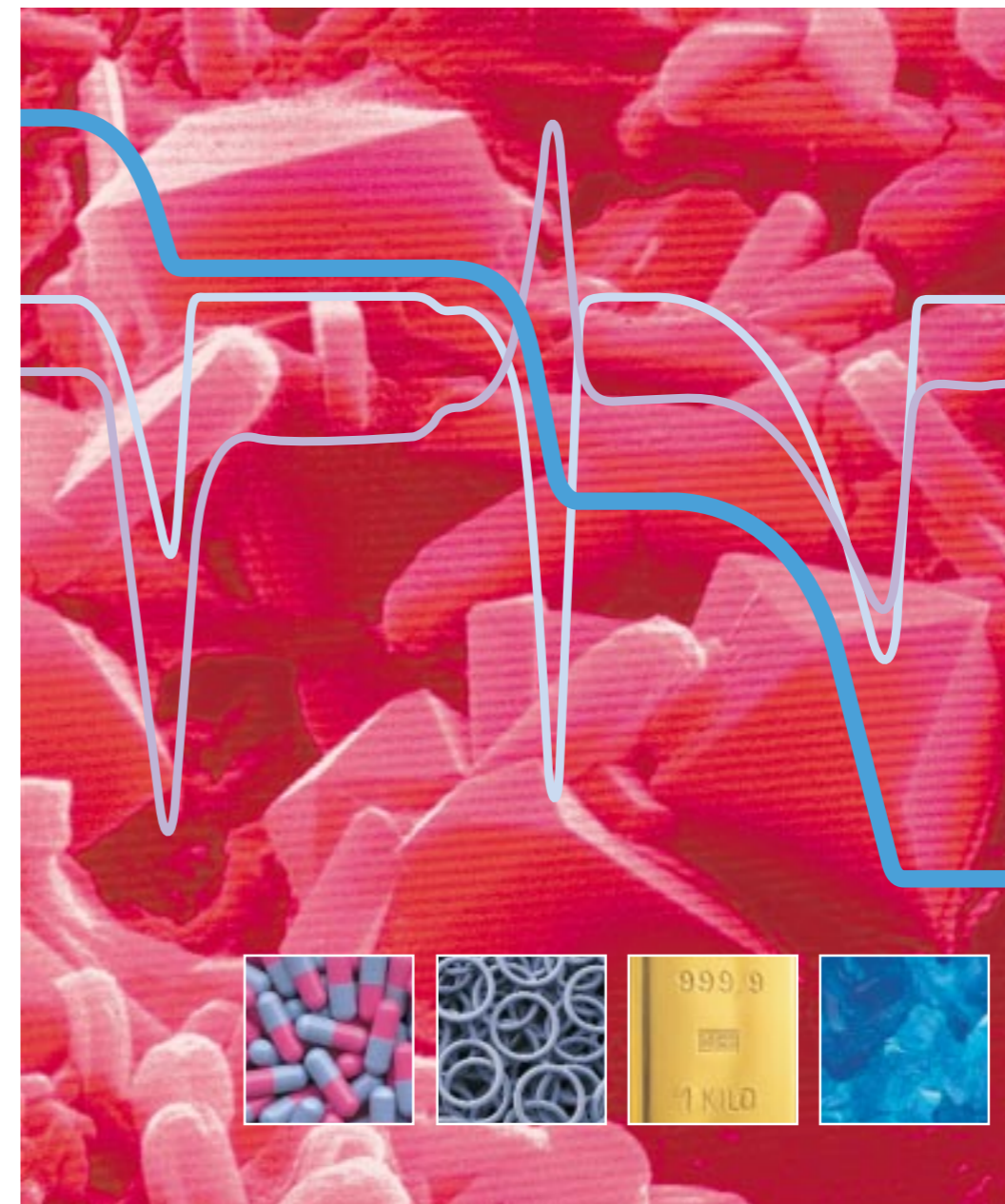
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Subject to technical changes

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Thermogravimetry for exacting requirements

TGA/SDTA851^e Module



METTLER TOLEDO

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Outstanding performance, simple and reliable operation

Thermogravimetry is a technique for the measurement of mass changes as a function of temperature. It is used primarily for determination of the composition of a substance and finds application in the fields of rubber and plastics analysis, the analysis of minerals and ceramics and in the chemical and pharmaceutical industries.

- Wide measurement range (1 or 5 g)
- High resolution (0.1 or 1.0 µg)
- Temperature range (room temperature to 1600 °C)
- High temperature accuracy (± 0.25 °C)
- The parallel guided balance ensures accurate, position independent weight measurements
- Controlled measurement environment thanks to gas-tight measuring cell
- Physical transitions observable on the SDTA™ curve
- Analysis of decomposition products with EGA (Evolved Gas Analysis)
- Modular design is readily upgradeable

Its modular construction makes the TGA/SDTA851^e eminently suited for manual or automatic operation in research and development, through to quality assurance and production.



Outstanding measurement performance

Measuring 1 million, 5 million or 50 million points over the entire weight range, no other TGA achieves this resolution. With samples capacity up to 5 g, this means determining weight changes to 0.1 µg. You can analyse small and large samples with the same resolution without switching the weight range. With noise less than 1 µg, you can really rely on your results.

Parallel guided balance

Thanks to the parallel guided balance, positioning of the sample has no influence on the measurement. Melting of the sample causes no apparent weight change.

Minimized disturbing influences

Thanks to the horizontal furnace arrangement, unavoidable disturbing influences (gas flow perturbations and thermal buoyancy) are minimized.

SDTA™ operation

(Single Differential Thermal Analysis) The DTA curve can be superimposed on the TG curve. This is even possible with only one sample crucible as the sample temperature is measured and the reference temperature calculated using a well defined mathematical model. Simultaneous measurement of the TG and DTA curves facilitates the interpretation appreciably.

Simple operation, reliable running

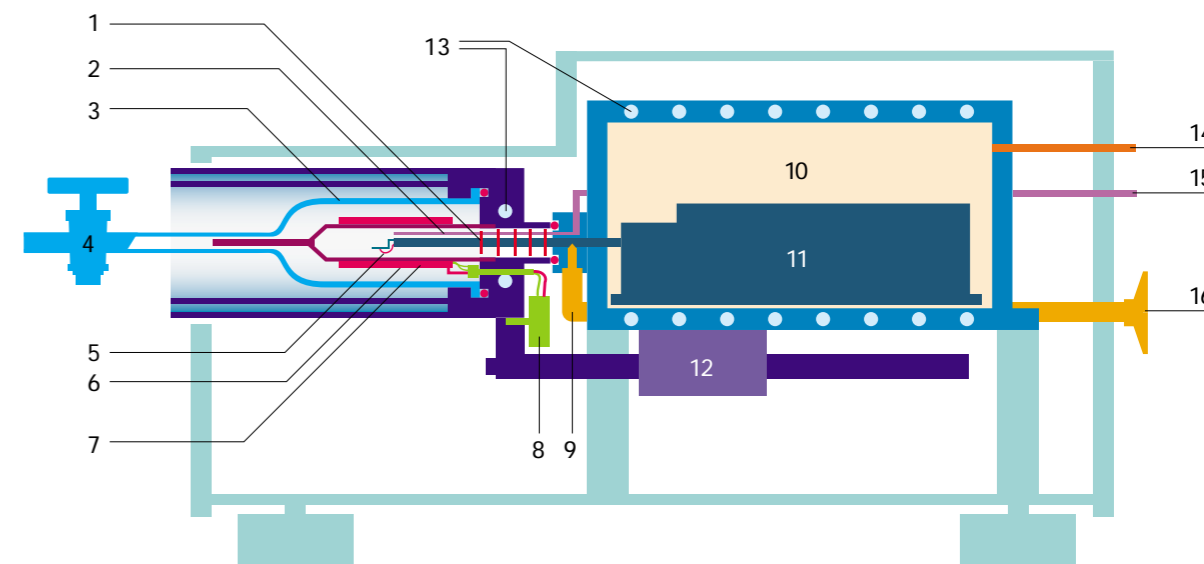
Automation starts with loading: Open the sample chamber with a keystroke, insert sample, close and you are ready to run. Routine operation with a sample robot is also possible and even more convenient.

High temperature accuracy

Temperature fluctuations of ± 0.25 °C can be detected by the sample temperature sensor directly attached to the sample holder. Calibration utilizes the accurate melting point of pure metals instead of the Curie temperature formerly employed.

Clearly definable atmosphere

The tightly sealed measuring cell can be evacuated and then purged with high purity gas with any particular gas. Such a closed system permits unambiguous results under exactly defined conditions. This opens up new applications (e.g. avoidance of oxidation thanks to oxygen-free atmosphere).



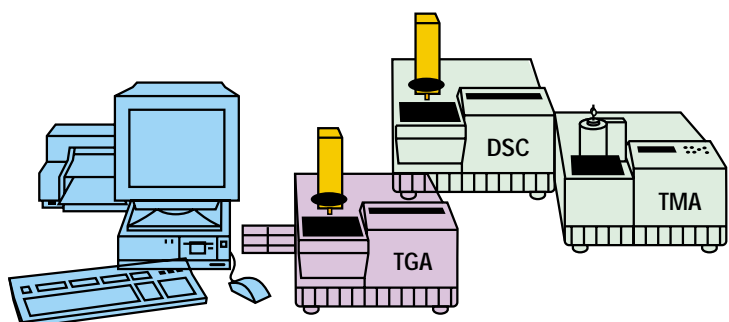
Key to illustration:

- 1 Baffles
- 2 Reactive gas capillary
- 3 Fused silica jacket
- 4 Gas outlet stopcock
- 5 Sample temperature sensor
- 6 Furnace heater
- 7 Furnace temperature sensor

- 8 Electrical connections
- 9 Vacuum and purge gas tubing
- 10 Thermostated balance chamber
- 11 Parallel guided ultramicro balance
- 12 Furnace motor for sample chamber opening
- 13 Cooling
- 14 Protective gas inlet
- 15 Reactive gas inlet
- 16 Vacuum connection and purge gas inlet

Expand and automate

Make your application more efficient



Equipped for the future

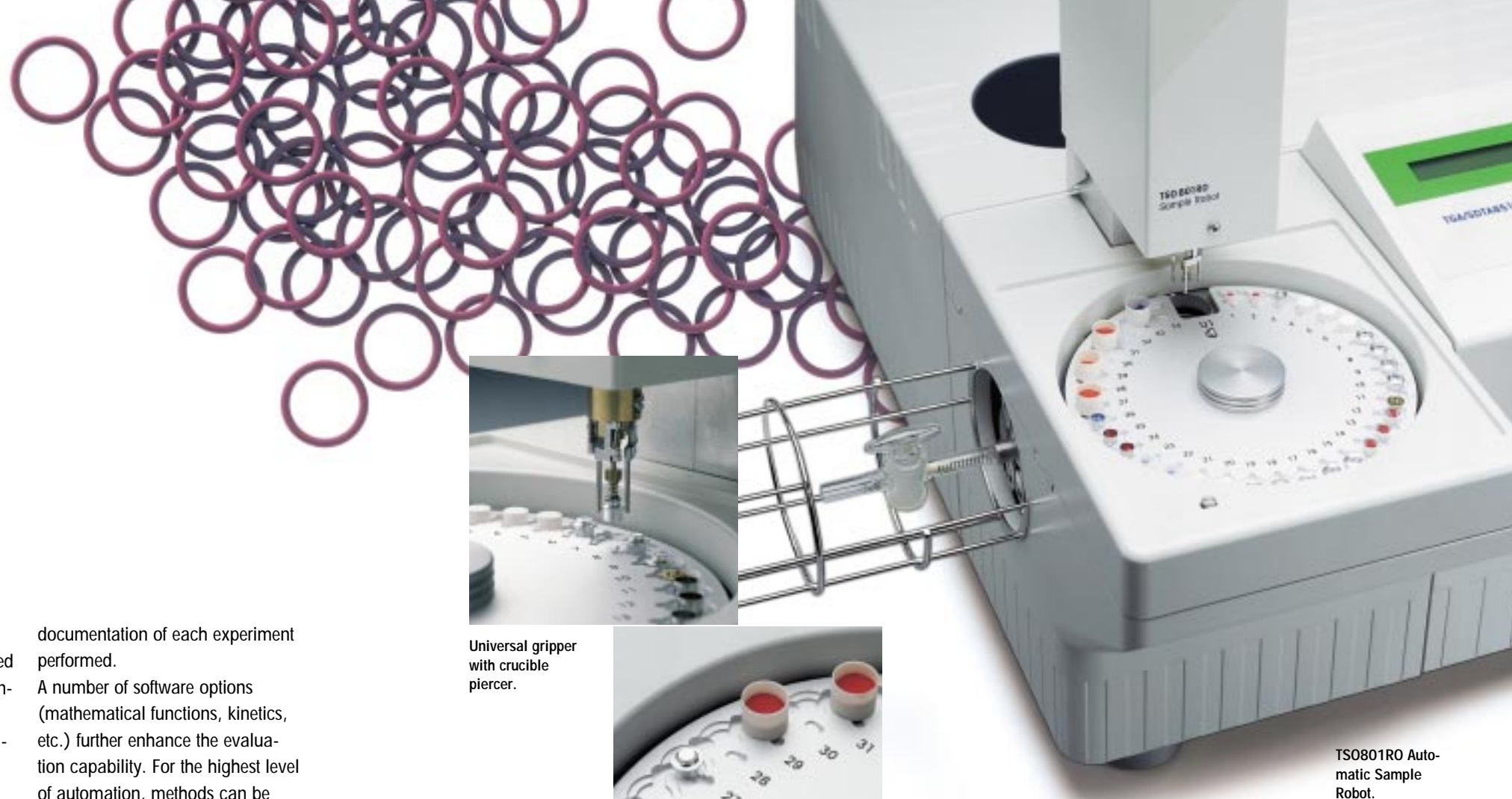
It is best to start with the instrument configuration which covers your current needs. You can choose from among three balance and three furnace alternatives to create your optimal system. Expansion with options and practical accessories, such as the Sample Robot or gas controller, is possible at any time.

Computer control

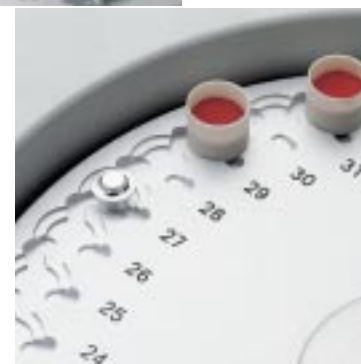
Measurement methods are designed using state-of-the-art computer control, and stored in a dynamically linked database. Experiment definitions, including the sample name and method, are transferred to the instrument and the experiment performed. The data and links to the method and experimental information are automatically stored in the same database, ensuring complete

documentation of each experiment performed. A number of software options (mathematical functions, kinetics, etc.) further enhance the evaluation capability. For the highest level of automation, methods can be created which automatically load the sample, perform the measurement, evaluate the data and print out the results without operator intervention. Individual measurement and evaluation programs can be defined for each sample.

For exacting demands, the TGA/SDTA851[®] module with small furnace.



Universal gripper with crucible piercer.



Sample turntable with crucibles of different size.

TSO801RO Automatic Sample Robot.

Automatic and efficient

All TGA/SDTA851[®] types can be automated with the TSO801RO Sample Robot. This allows the automatic processing of up to 34 samples, each by a different method and with a different crucible. A unique feature is the possibility of either removing or, with hermetically sealed aluminum crucibles, puncturing the crucible lid with the sample robot before the measurement. As a result, the exchange of moisture between weighing in and measurement is very much restricted. Sample weighing can also be fully automated. Simply place the empty crucibles on the sample

turntable, press automatic weighing and you can turn to other tasks. In less than one hour, all 34 crucibles have been weighed. Add sample to the crucibles and press the start key and the experiment series begins. Crucible and sample weights can also be manually entered into the computer for maximum flexibility. As the universal gripper permits use of crucibles with different diameters, you can select exactly the right crucible for each application. With TGA/SDTA measurements we recommend platinum, gold or aluminum crucibles. Aluminum oxide crucibles are also available.

Modular means expandable at any time

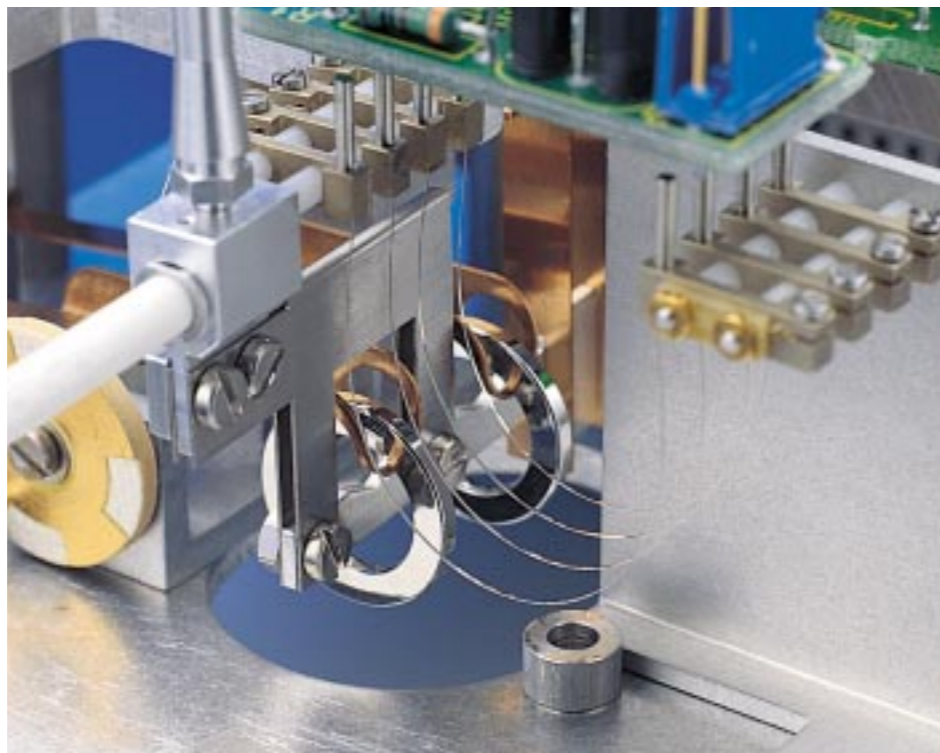


The optimum balance to meet your demands

When you purchase a TGA/SDTA851^e you have a choice of three balance types to match your application. With the MT1 you can measure samples weighing up to 1000 mg with a continuous resolution of 1 µg. To increase capacity the MT5 has been specially designed for heavy samples up to 5000 mg. With the MT5 weight is measured over the entire weighing range with a resolution of 1 µg without switching the measurement range. The UMT5 Ultramicro Balance is even more sensitive; you can mea-

sure 5000 mg with a continuous resolution of 0.1 µg. A feature common to all balances is the internal, automatic calibration. Motorized, built-in calibration weights are loaded during pauses in measurement when the balance is in the standby mode without any action on your part. As is usual with METTLER TOLEDO balances, no weight compensation is needed, a necessary feature for automation if light and heavy weights are to be measured in succession.

Inside the balance you can see the ring weights for automatic calibration.



Coupled measurement technique for more informative results

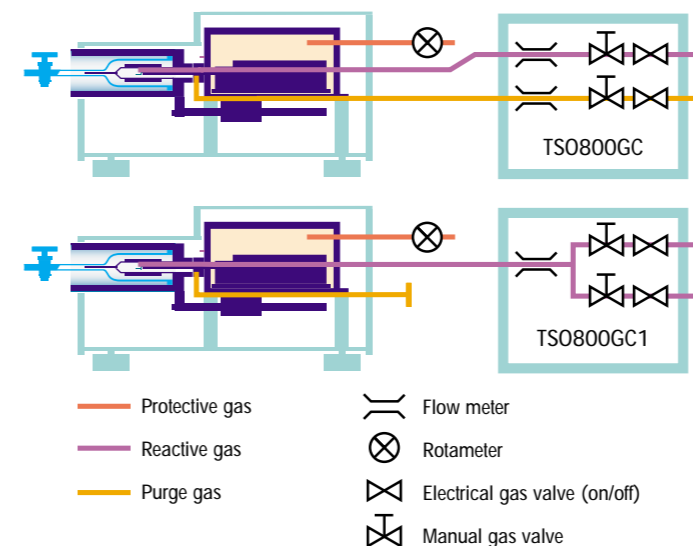
The TGA/SDTA851^e Measuring Cell can be coupled with a mass spectrometer, FTIR spectrometer or gas chromatograph. Several reactions can occur at a single TG step and be identified through their different products. Identification of the decomposition products provides valuable additional information on the sample and allows a dependable and unequivocal interpretation of the experimental curve. The peripheral option includes a trigger signal to start the external device and the TGA experiment simultaneously. The subsequent data correlation is thus extremely simple.



We offer you all important accessories: crucibles, tutorial substances and tweezers.

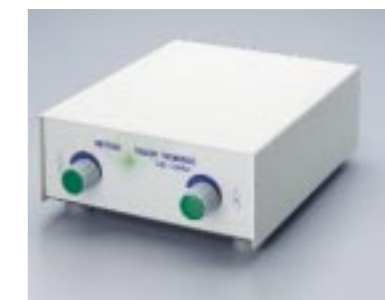


Application documentation helps you convert our know-how into state of the art solutions to problems.



Programmable gas switching

Automatic operation can be suitably supported with the gas controllers. The gas controllers measure the mass flow of the gas and, depending on the type of controller, one channel or two channels can be turned on and off using the software.



TSO800GC Gas Controller measures, switches and monitors.

For large and small sample volumes

As heterogeneous samples necessitate a large sample weight and correspondingly large sample volumes, use the large furnace with crucibles of volume up to 900 µl (max. diameter 12 mm). In the case of highly exacting temperature demands with extremely

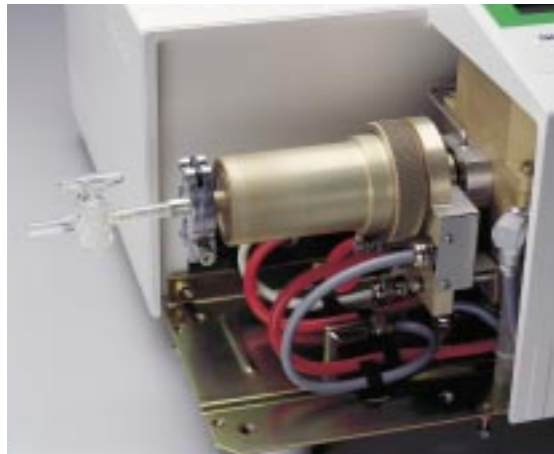
low temperature gradients, we recommend the small furnace for the measurement of sample volumes up to 100 µl (diameter 7 mm). You have a choice of 3 furnace types:

- small furnace up to 1100 °C (100 µl)
- large furnace up to 1100 °C (900 µl)
- large furnace up to 1600 °C (900 µl)



Large sample holder with 900 µl aluminum oxide crucible.

Thermal isolation of the extremely sensitive balance is implemented by a water-cooled flange. All furnaces are gas tight and can be evacuated and purged. A reactive gas can be introduced near the sample. The sample holder is provided with a thermocouple as standard to permit measurement of the sample temperature and calculation of the SDTA™ curve.



Large watercooled furnace.

Thermogravimetry application possibilities

Within a short time and using small samples, information is obtained concerning the composition of the sample as well as regarding its stability, oxidation behavior or the kinetics of a chemical reaction.

The amount of weight lost during a TGA experiment provides quantitative insight into the composition of a sample. Additionally, the tempera-

tures of thermal and oxidative degradation are measured. A wide variety of material behaviors and products can be investigated, including volatilization, thermal degradation in an inert atmosphere (pyrolysis), reactions with oxygen or other reactive gases, and amount of residual materials (ash, filler) or EGA (Evolved Gas Analysis), e.g. mass spectrometry (TG-MS) provide

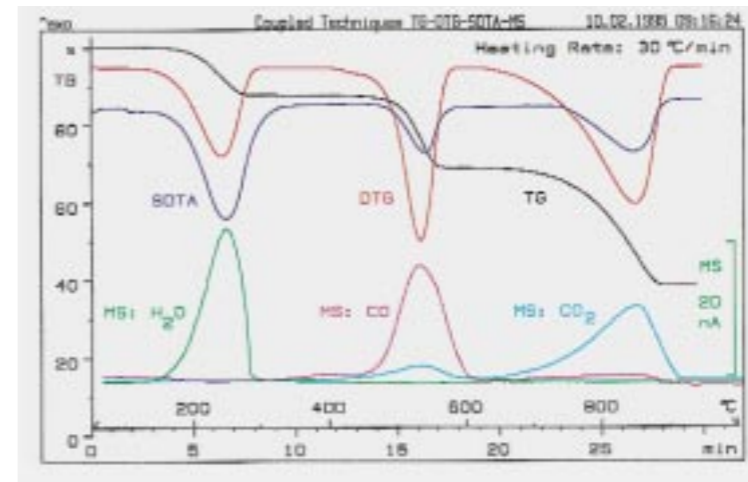
additional thermal or chemical information which is very useful for the interpretation of complex thermochemical processes. The main applications of thermogravimetry in industry, education and research concern investigations of polymers, inorganic and organic chemicals, pharmaceuticals, building materials, minerals, ceramics and composites.

All combination possibilities at a glance

Option → required option	Small furnace 1100 °C SF	Large furnace 1100 °C LF	Large furnace 1600 °C LF	MT1-TGA	MT5-TGA	UMT5-TGA	Peripheral	On/off switching
TGA/SDTA851 ^e (1100 °C SF)	●			●	●	●		
TGA/SDTA851 ^e (1100 °C LF)		●		●	●	●		
TGA/SDTA851 ^e (1600 °C LF)			●	●	●	●		
Gas controller							●	
Sample robot*								● 1
Cryostat cooling								● 1
Coupling (MS/GC/FTIR)								● 2

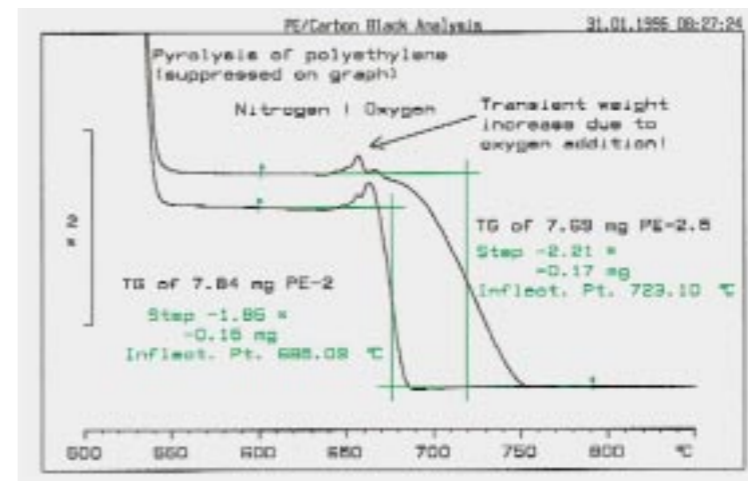
● Included as standard
● Either/or
● Recommended
● A must

SF: Small furnace LF: Large furnace
 1) Only with long idle times
 2) FTIR only with the large furnace
 * No additional options required

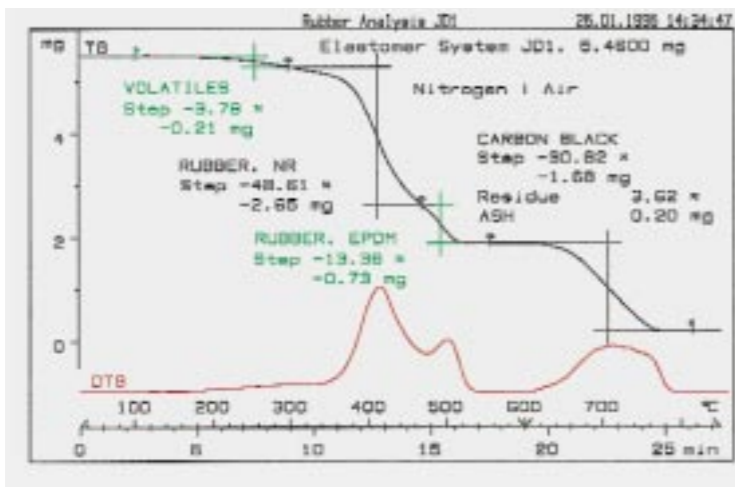
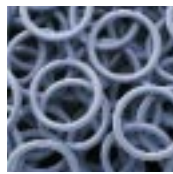


Calcium oxalate monohydrate exhibits three weight steps. As the SDTA™ curve attests, the three reactions are all endothermal if measured in argon. The mass spectrometer Balzers MS-Cube™ is coupled to the TGA/SDTA851^e by a heated capillary. The three simultaneously measured ion current curves for H₂O, CO and CO₂ explain the course of the splitting reactions:

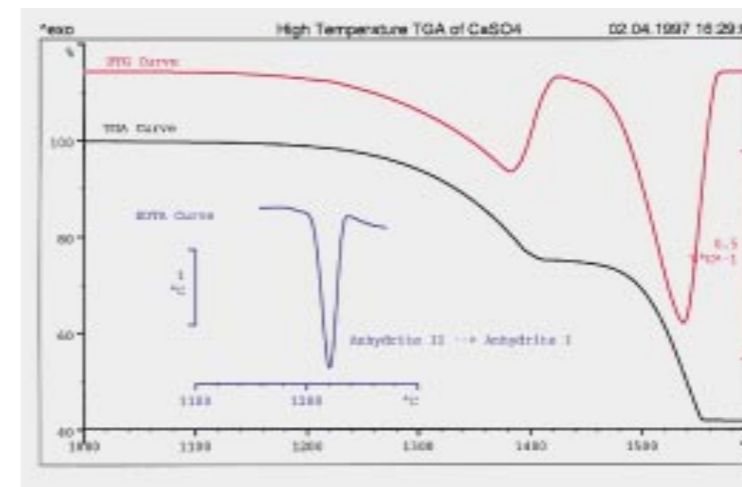
- water of crystallization
- carbon monoxide forming CaCO₃
- carbon dioxide.



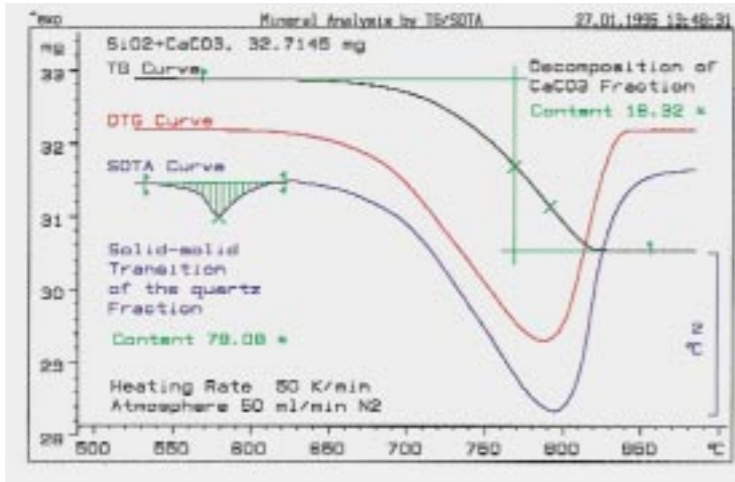
Carbon black added to polyolefines influences the bulk properties considerably. Therefore, an accurate determination of carbon black is essential. The main component – the polymer – is flashed off under pure nitrogen in the gas tight TGA/SDTA851^e. At 650 °C an automatic switch is made to oxygen and the remaining carbon black burns away. The burning profiles attest different activities of carbon black (slow combustion = low activity).



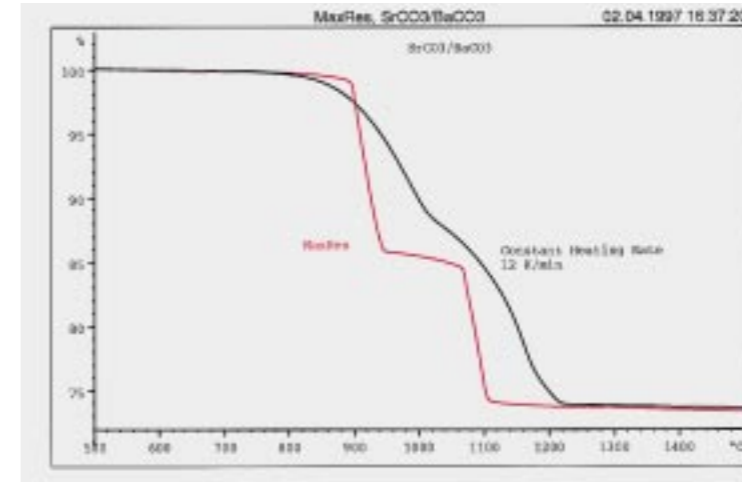
Compositional analysis determines the main components of elastomer systems. The first step represents plasticizers evaporating between 200 and 300 °C. Afterwards the individual elastomers pyrolyze often at different temperatures. The DTG-peaks serve for good separation of overlapping decomposition steps. At 600 °C the atmosphere is automatically changed to air to combust the carbon black. Ash and fillers remain as residue.



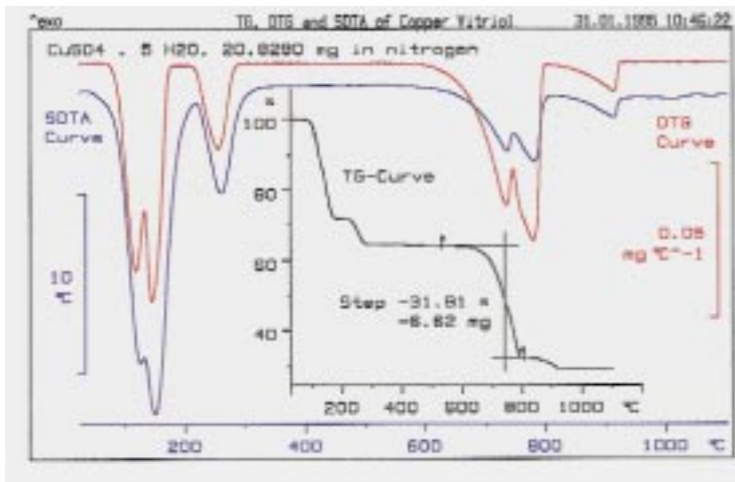
Thermal behavior of anhydrite, CaSO₄. Above 1200 °C the SDTA™ curve shows an endothermic peak that is due to the solid-solid transition of anhydrite II to anhydrite I. At higher temperature the anhydrite decomposition occurs in two steps evolving SO₂ and O₂. The DTG peaks indicate the two individual reactions. CaO remains as residue.



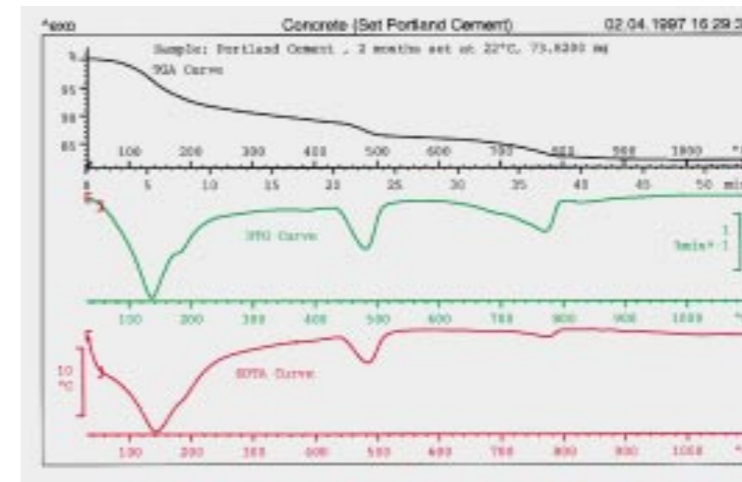
Many building materials and minerals contain CaCO₃ along with SiO₂. The integral of the SDTA™ curve during the solid-solid-transition is proportional to the quartz content (78 %). The weight loss between 600 and 800 °C corresponds to the CO₂ release of the limestone and the evaluation software calculates the stoichiometric CaCO₃ content (16.3 %). Physical and chemical transformations without mass change are visible at a glance by comparing the DTG and SDTA™ curves.



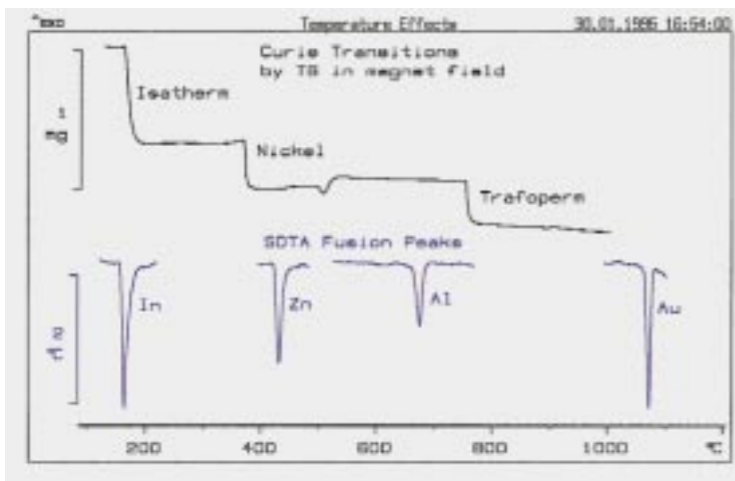
MaxRes allows rapid TGA measurements with simultaneously high resolution. Based on the rate of weight change, the heating rate is automatically lowered or raised stepwise. Conditions: the sample is a mixture of approx. 20 mg SrCO₃ and 20 mg BaCO₃ in alumina pan, atmosphere 60 ml/min nitrogen. The obtained MaxRes curve is compared with the classical TGA curve whose heating rate has been calculated to get the equal experiment time of 85 min.



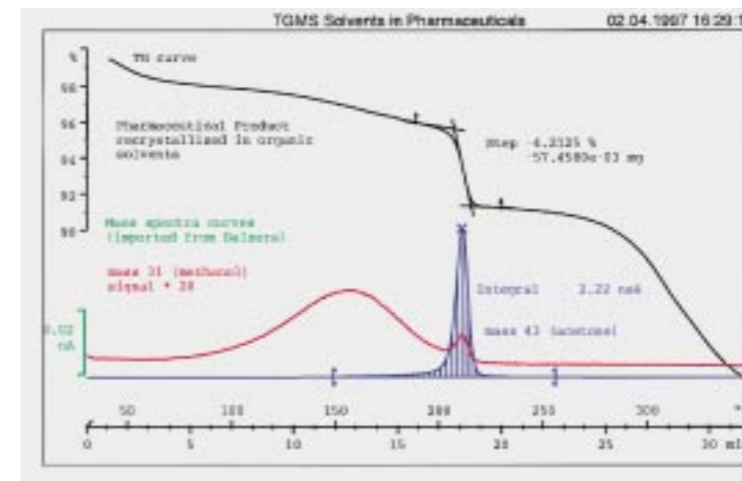
On heating, inorganic salts first split off their water of crystallization. Copper sulfate heated with 50 °C/min gives a first TG step that corresponds to 4 H₂O. The DTG curve shows that the step consists of two overlapping single reactions. Around 250 °C the most strongly bound water is liberated. The sulfate ion decomposes between 600 and 800 °C. Again, DTG and SDTA™, indicate two individual reactions. At 900 °C the remaining CuO forms Cu₂O and oxygen.



The main components of the cement are tricalcium silicate, dicalcium silicate and tricalcium aluminate. After the cement has been mixed with water, various hydrates are formed as the concrete sets. Conditions: 74 mg concrete set two months at 22 °C, heating rate 20 K/min, atmosphere air. Adsorbed water evaporates first, then the hydrates of calcium silicate and the hydroxides of calcium, magnesium and aluminum decompose until 570 °C. Finally the calcium carbonate releases CO₂.



The upper curve is obtained from three ferromagnetic samples exposed to a non-homogeneous magnet field. The small peak at 500 °C is due to the Curie transition of the heater wire and disappears after blank subtraction. Curie transitions allow temperature calibration. The more accurate temperature calibration is based on SDTA™ melting curves of pure metals. As the other examples attest, many different thermal effects appear up on SDTA™ curves of course.



During production of pharmaceuticals often the product has to be recrystallized in different solvents. To check that only small amounts of these solvents may stay in the final product a combined measurement of TG and MS is performed (Evolved Gas Analysis: TGA/SDTA851[®] and Balzers ThermoStar). This analysis shows the weight losses in the TG curve and the corresponding gases detected as molar masses 31 and 43. They represent acetone and methanol which were used for recrystallization and acting as a solvat now.