Thermal Analysis Excellence

TGA/DSC 3+

STAR[e] System

Innovative Technology

Versatile Modularity

Swiss Quality

Thermogravimetry

For Unmatched Performance

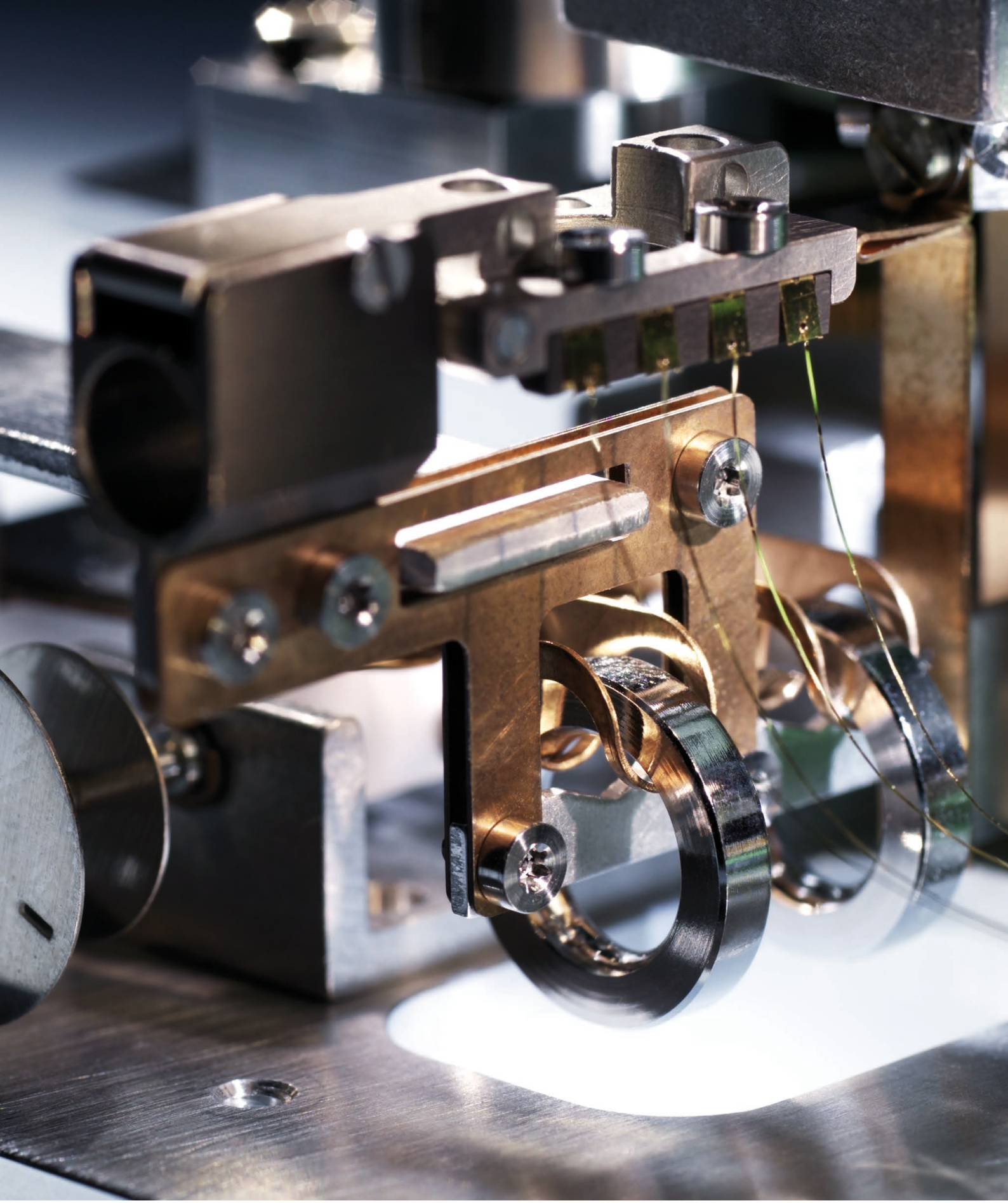
METTLER TOLEDO

Fast and Accurate TGA Results
METTLER TOLEDO Balance Inside

Thermogravimetry (TGA) is a technique that measures the change in weight of a sample as it is heated, cooled or held at constant temperature. Its main use is to characterize materials with regard to their composition. Application areas include plastics, elastomers and thermosets, mineral compounds and ceramics as well as a wide range of analyses in the chemical and pharmaceutical industries.

Features and benefits of the METTLER TOLEDO TGA/DSC 3+:

- **METTLER TOLEDO ultra-micro balance** – rely on the leader in balance technology
- **Very low minimum weight on 5-gram-balances** – measure samples accurately and precisely
- **Unparalleled performance** – sub-microgram resolution over the entire weighing range
- **Wide temperature range** – analyze samples from ambient temperature to 1600 °C
- **DSC heat flow measurement** – for simultaneous detection of thermal effects
- **Built-in gas flow control** – investigate your samples in defined atmospheres
- **Automated TGA-FTIR, TGA-MS and TGA-GC/MS systems** – perform accurate evolved gas analyses using FTIR and MS
- **Modular concept protects your investment** – fits to your current and future needs
- **Robust endurance-tested sample robot** – operates efficiently and reliably around the clock



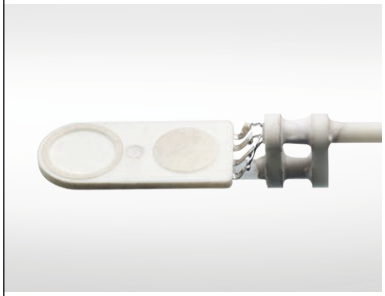
TGA equipped with a top-of-the-line METTLER TOLEDO ultra-micro balance with unique built-in calibration weights ensures unbeatable accuracy.

www.mt.com/ta-acc

MultiSTAR™ TGA/DSC Sensors
Simultaneously Measure Heat Flow (DSC)

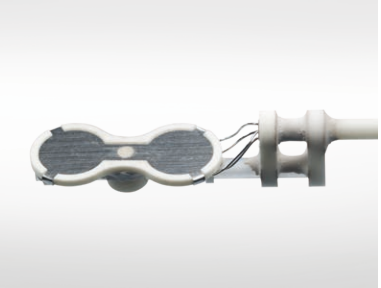
The DSC sensor is based on the unique MultiSTAR sensor amplification technology. The six thermocouples generate a larger measurement signal, improving the signal-to-noise ratio. With all three types of sensors, the heat flow is determined from the calculated or measured temperature difference. The horizontal furnace design helps to minimize possible effects caused by thermal turbulences and the purge gas.

DSC Sensor



Six thermocouples located directly below a protective ceramic support that measure the sample and reference temperatures.

DTA Sensor



The platinum support measures both the sample reference temperatures. The differential measurement improves the signal-to-noise performance of the sensor.

SDTA Sensors

A platinum support with a thermocouple that measures the sample temperature. These large SDTA sensors, allows the use of crucibles up to 900 µL for the large and high temperature furnace options.

Thermocouples are attached to the crucible holder to detect the smallest thermal effects. Temperature calibration and adjustment is performed using precise melting points, resulting in high temperature accuracy.

www.mt.com/ta-crucibles

Reliable Automation
24 Hours a Day, Like a Swiss Watch

Automatic and efficient, our sample robot is a sophisticated automation option allowing for reliable operation 24 hours a day though the whole year. Together with the STAR^{e} software, the power of the automation is increased by our unique FlexCal™ calibration concept always selecting the correct adjustment parameters, and the possibility to automatically evaluate results.

Features and benefits of the sample robot:

- **34 sample positions** – dramatically increases efficiency
- **Simple and rugged design** – guarantees reliable results
- **Unique lid piercing capability** – no change in sample weight during the wait cycle
- **Automatic crucible lid removal system** – reduces unwanted loss of sample mass
- **Universal gripper** – can handle all types of METTLER TOLEDO crucibles

Fully automatic weigh-in

Samples can be weighed-in semi or fully automatically using the internal TGA balance in combination with the sample robot. Simply place the empty crucibles for automatic weighing, and then insert a sample in each crucible. The robot will take care of the rest.

No weight change before measurement

The sample robot can remove the protective crucible lid from the crucible or pierce the lid of hermetically sealed aluminum crucibles immediately before measurement. This unique feature helps to prevent any mass change between weigh-in and the measurement.

Extensive range of crucibles

We have the right crucible for every application. The crucibles are made of different materials with volumes ranging from 20 to 900 µL. All of the different types can be used with the sample robot. Available crucible materials can be found here:

www.mt.com/ta-crucibles

All TGA/DSC 3+ models can be automated. The sample robot can process up to 34 samples even if every sample requires a different method and a different crucible.

www.mt.com/ta-automation

Hyphenated Techniques
Provides More Material Insight

To obtain more information from a single experiment, a TGA/DSC 3+ can be coupled to a humidity generator, a mass spectrometer, an FTIR spectrometer, a GC/MS-system or a Mirco GC/MS. This enables you to interpret measurement curves with greater certainty.

Features and benefits of the TGA Hyphenated techniques systems:

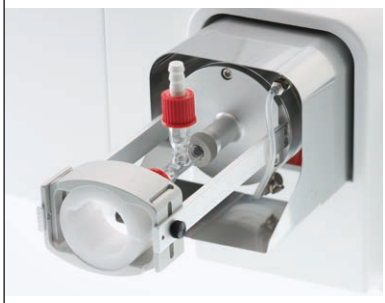
- **Automated TGA-EGA systems** – save time by acquiring more information from a single run
- **Simultaneous TGA-EGA** – enables quantitative compositional analysis and material identification
- **Reference databases** – help to identify gaseous decomposition products
- **Unique flexibility** – a highly sensitive thermoanalyzer combined with full sorption analysis capability
- **Defined environment** – the effect of moisture and temperature on material properties can be easily investigated

Sorption Interface

The TGA can be converted to a TGA Sorption analyzer in just a few minutes. This allows materials to be analyzed under precisely defined conditions of relative humidity and temperature.

www.mt.com/ta-moisture

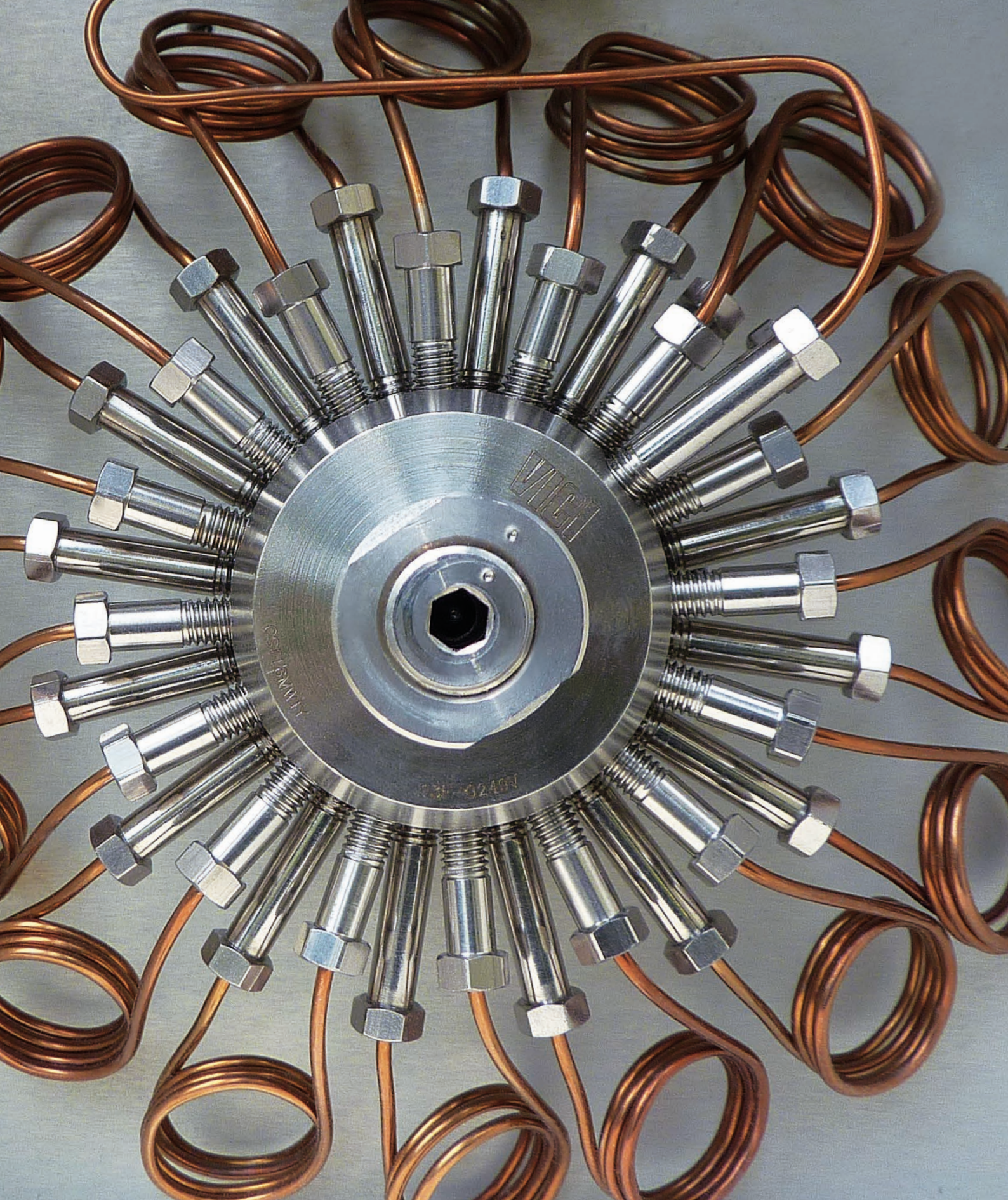
TGA-MS Interface



The TGA-MS interface is able to expand the instrument to the extremely sensitive techniques of Mass Spectrometry (MS). Similarly, you can couple a TGA to a Micro GC/MS.

TGA-FTIR Interface

FTIR can be used to characterize or identify a substance or class of molecules. The identification of the evolved gas is simplified by the ability to import the curves into S**TA**R^{e} Software.



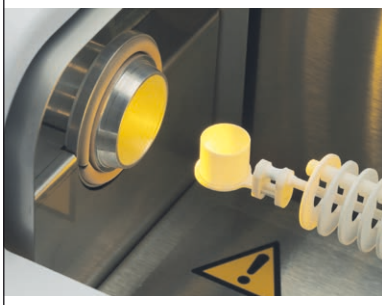
The TGA-IST16-GC/MS is an instrument extension for characterization by thermogravimetric analysis. The combined system provides valuable information, whether it is used in quality control or for industrial and academic research.

www.mt.com/ta-ega

Versatile Modularity
Fits Every Need, Even if They Change

Ever since its inception in 1964, METTLER TOLEDO's TGA technology has continued to evolve, spawning innovation in industries and new materials. Trying to fit the needs of every customer, there are countless options and built in features to make the TGA/DSC 3+ a world-class instrument.

Furnace options



Choice of different furnace sizes and different temperature ranges allow for a wide range of possible applications. The small furnace has the highest temperature accuracy, due to its small volume. The large furnace allows measurement of samples in crucibles up to 900 µL. The high temperature furnace can reach a temperature of 1600 °C.

Optimal atmosphere

Built-in mass flow controllers allow for precisely defining the furnace atmosphere. This allows accurate and repeatable investigation of material properties under a variety of atmospheres (including vacuum) and switching of reactive gas during an experiment. The gastight furnace with defined conditions is essential to obtain unambiguous information and quality results.

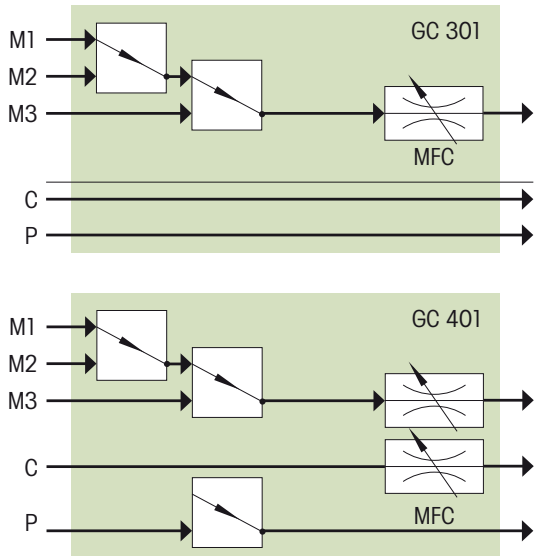
Balance type

Four different balance types are available for the TGA/DSC 3+, with a measurement range up to 5 g and resolutions as low as 0.1 µg. These balances are produced in-house by the market leader in precision balances.

Option Matrix: A TGA/DSC 3+ for every need and configuration can be found in the below table.

	Balances				EGA (MS, FTIR, GC/MS, Micro-GC/MS)	Sorption	GC 301	GC 401
	XP1	XP1U	XP5	XP5U				
TGA/DSC 3+ (SF 1100 °C)	•	•	•	•	•		standard	optional
TGA/DSC 3+ (LF 1100 °C)	•	•	•	•	•	•	standard	optional
TGA/DSC 3+ (HT 1600 °C)	•	•	•	•	•	•	standard	optional
Peripheral control					recommended	recommended		
Sample Robot	no additional options required							

● = Selectable



Defined furnance atmosphere, programmable gas flow and gas switching.
The furnace chamber can be purged with a defined gas flow. The software-controlled mass flow gas controller measure and regulates the gas flow between 0 and 200 mL/min and can automiatically switch up to 3 gases. Regulate and switch gases such as air, nitrogen, oxygen, argon, CO_2 and inert hydrogen (96% Ar, 4% H_2).

Sensors	SF (1100 °C)	LF (1100 °C)	HT (1600 °C)
SDTA	•	•	•
DTA		•	•
DSC		•	•

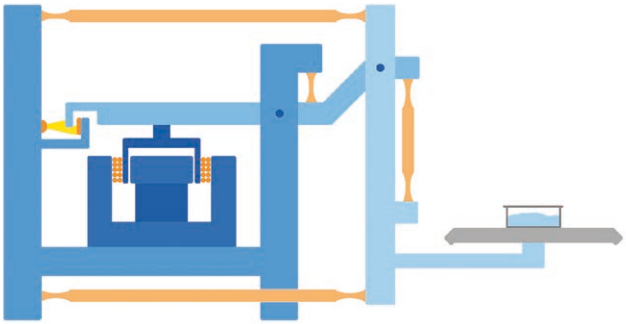
Build the TGA that fits your requirements. Different furnace sizes, sensors and balances (micro or ultra-micro) are available to help solve all possible applications and needs.

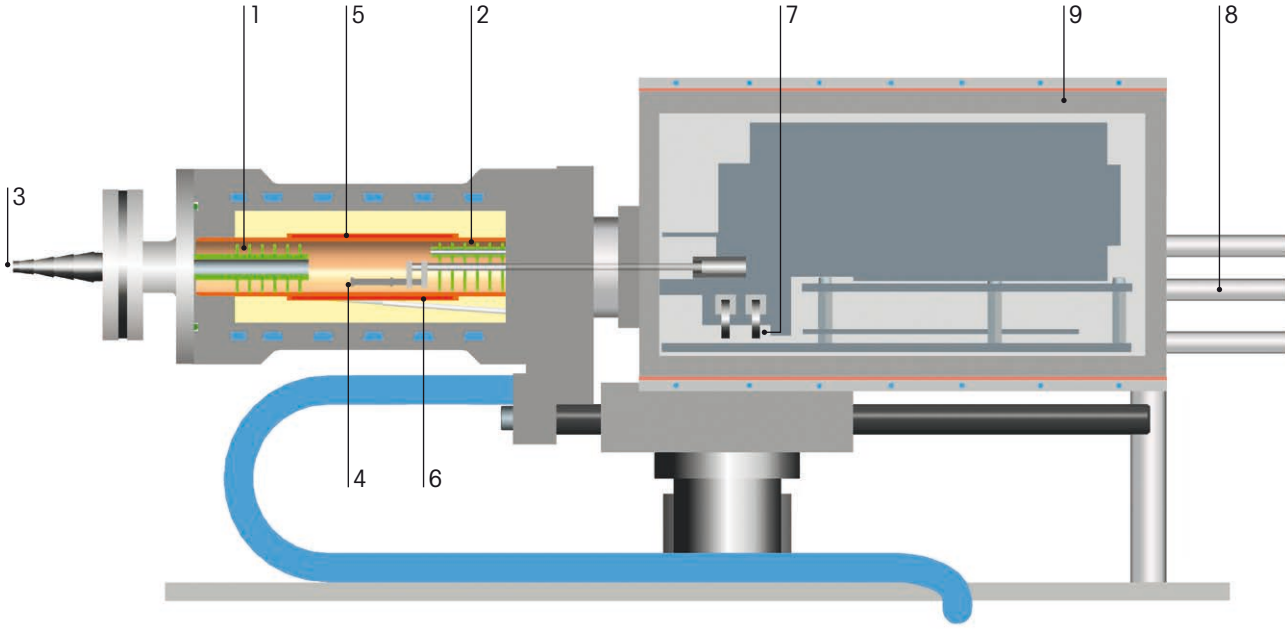
Balance	Measurement Range (g)	Resolution (µg)
XP1	1	1
XP1U	1	0.1
XP5	5	1
XP5U	5	0.1

Excellent Performance
Over the Entire Weighing Range

Outstanding weighing performance. No other TGA can measure up to 50 million resolution points continuously – weight changes of a 5-gram sample are determined to 0.1 µg. A low minimum weight ensures that small samples close to the low end of the weighing range are also weighed accurately.

Modern weighing technology: The parallel guided balance ensures that the position of the sample does not influence the weight measurement. Built-in automatic buoyancy compensation eliminates the need for time consuming baseline measurements.





1. Baffles
2. Reactive gas Capillary
3. Gas outlet
4. Temperature Sensor
5. Furnace heater
6. Furnace temperature sensor
7. Adjustment ring weights
8. Protective and purge gas connector
9. Thermostated balance chamber

Inserting samples manually is made easy with the well-designed ergonomic support. The display gives a clear picture of the instruments status, and in is OneClick™ enabled, allowing predefined methods to be started with ease.

www.mt.com/ta-tgadsc

Extremely Wide Application Range
For All Kinds of Materials

Simultaneous Thermogravimetic Analysis and Differential Scanning Calorimetry yields valuable information about the mass change and heat flow of the sample. Together these two can be used to determine the temperature of different effect with great certainty.

The TGA/DSC is an exceptionally versatile tool for the characterization of physical and chemical material properties and chemical material properties under precisely controlled atmospheric conditions. This simultaneously measurements provide insights for research, development and quality control in numerous fields such as plastics, building materials, minerals, pharmaceuticals and foodstuff.

Examples of thermal events and processes that can be determined by TGA/DSC

TGA

- Quantitative content analysis (moisture, fillers, polymer content, materials, etc.)
- Adsorption and desorption of gases
- Kinetics of decomposition processes
- Sublimation, evaporation and vaporization
- Thermal stability
- Oxidation reactions and oxidation stability
- Identification of decomposition products, solvents and solvates
- Sorption and desorption of moisture
- Reaction and transition enthalpies
- Pseudopolymorphism

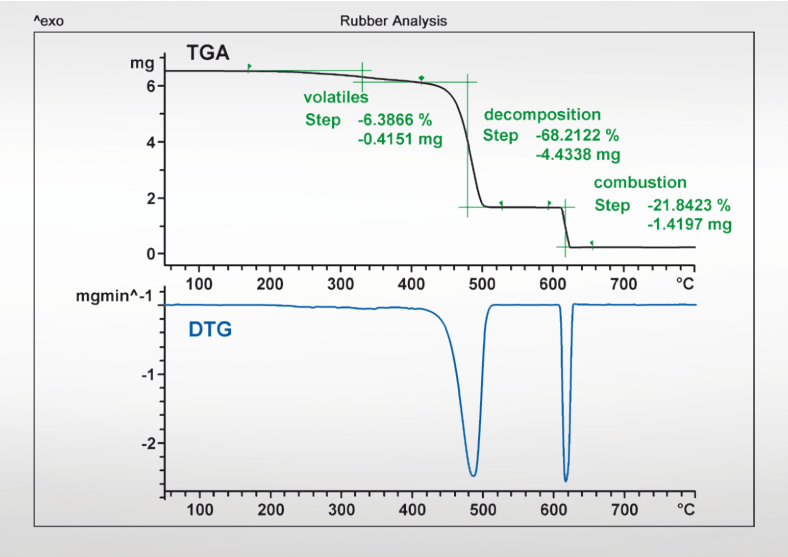
DSC

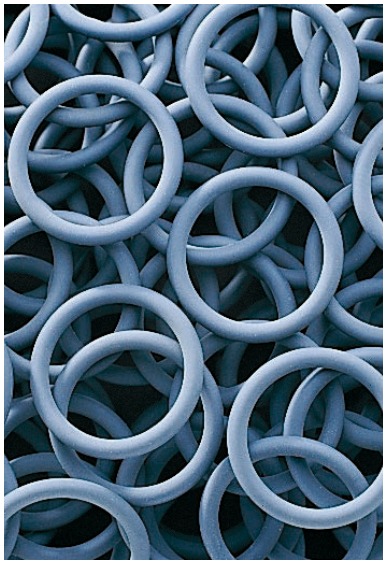
- Determination of Curie temperatures
- Melting behavior
- Crystallization
- Polymorphism
- Phase diagrams
- Glass transitions
- Reaction kinetics
- Heat capacity

Thermogravimetry provides quantitative information on the composition and thermal stability of many different types of materials. The method is fast and can be used with very small samples.

www.mt.com/ta-applications

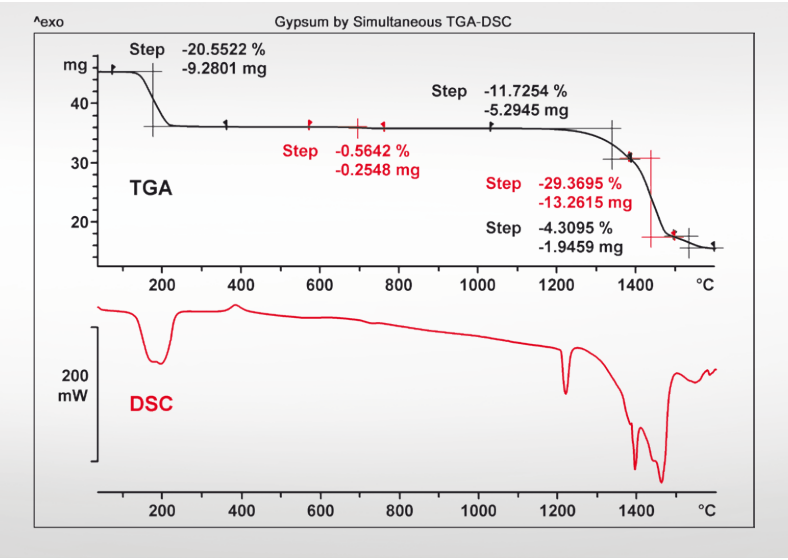
Rubber analysis of SBR





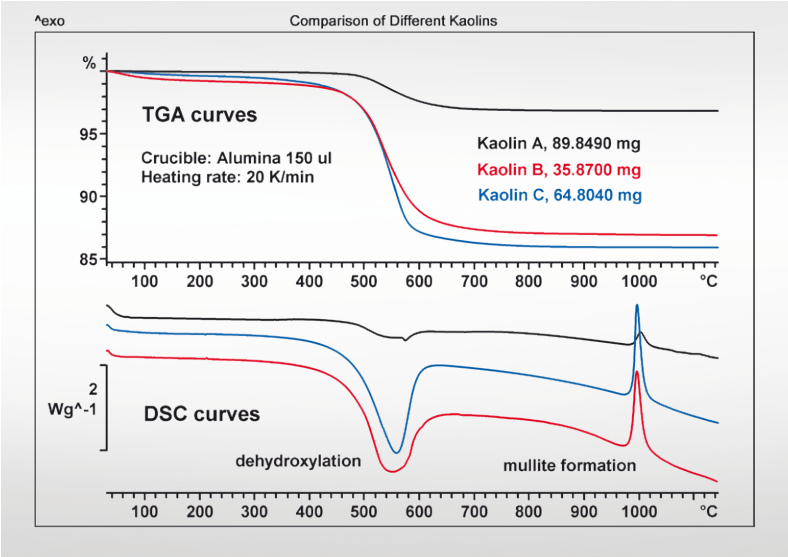
The sample is first heated to 600 °C under inert conditions, during this heating, the volatile components (plasticizers, often oils) vaporize and pyrolysis of the polymer begins shortly afterward at about 400 °C. At 600 °C, the atmosphere is switched from inert to oxidative, resulting in the combustion of the carbon black additive. Inorganic components remain behind as a residue. The SBR sample analyzed in this example contains 6.4% plasticizer, 68.2% polymer, 21.8% carbon black, and 3.6% residual components (mainly zinc oxide).

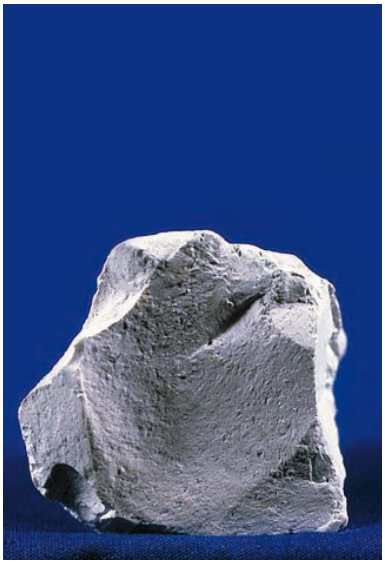
Thermal analysis of gypsum



Gypsum ($CaSO_4 \cdot 2H_2O$) loses its water of crystallization below 300 °C. The calcium carbonate, present as an impurity, decomposes at about 700 °C. Decomposition of the calcium sulfate occurs in several steps from about 1200 °C onward. The simultaneously recorded DSC curve shows two effects due to solid-solid transitions at about 390 and 1236 °C: γ-$CaSO_4$ (anhydrite III) to ß-$CaSO_4$ (anhydrite II), and β-$CaSO_4$ to α-$CaSO_4$ (anhydrite I). The latter melts slightly below 1400 °C and is observed as a sharp endothermic peak.

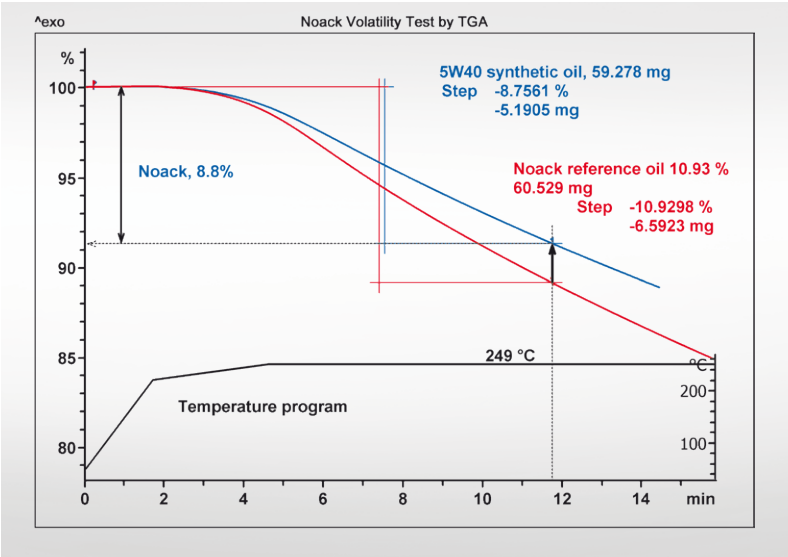
Kaolinite





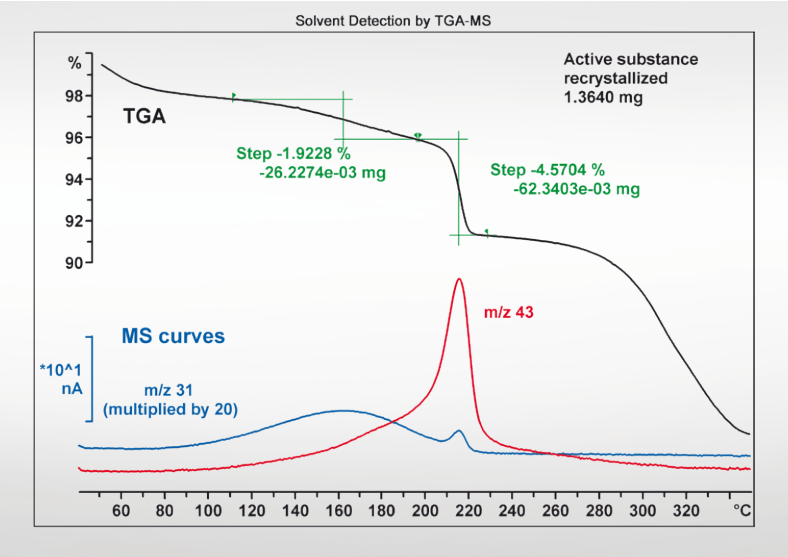
Kaolin is a white mineral used in the paper industry as a filler in plastics and for the manufacture of porcelain. The main constituent of kaolin is kaolinite, $Al_2Si_2O_5(OH)_4$, which dehydroxylates between 450 °C and 600 °C. This is the reason for the weight loss in the TGA curves. The example shows the measurement of three kaolin samples with different contents of kaolinite. The DSC curve for Kaolin A shows a small peak at about 575 °C. This peak is characteristic for the solid-solid transition of α-quartz to β-quartz. The exothermic peak at about 1000 °C is due to the formation of mullite.

Volatility of oils



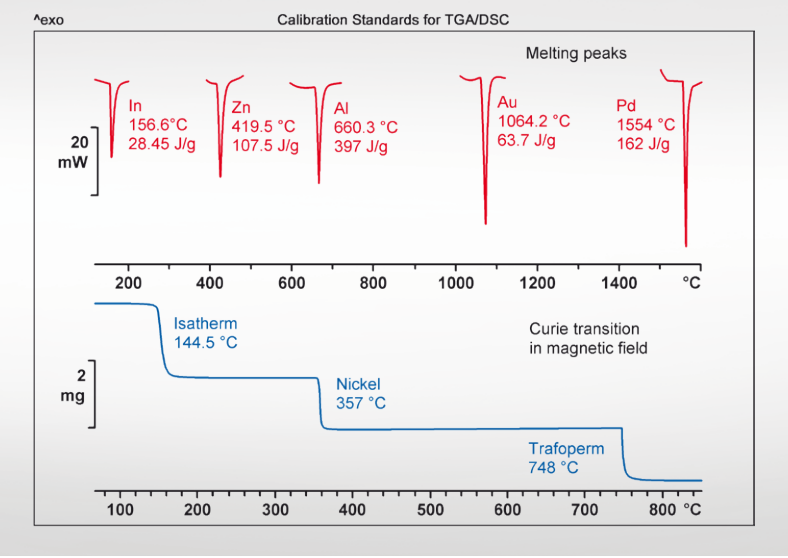
The Noack Test according to ASTM D 6375 is used to assess the volatility or evaporation loss of a lubricating oil in comparison with a reference oil at a particular temperature. The procedure is summarized with the temperature program (black curve). The reference oil takes 11.9 min to lose the specified mass loss of 10.93%. The oil under examination loses 8.8% of its mass up until this time. Therefore, its Noack volatility is 8.8%. The method allows rapid and reliable characterization of oil volatility.

Residual solvents in pharmaceutical substances



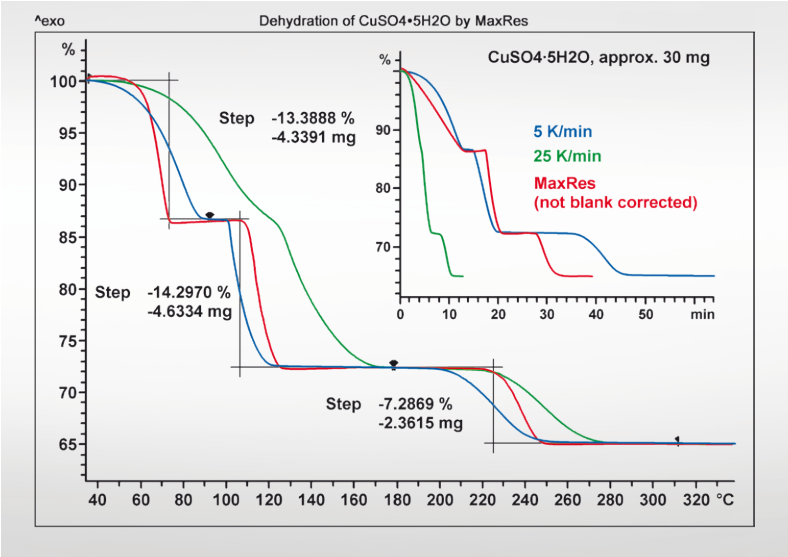
Many pharmaceutical substances are recrystallized from solvents. As a result, residues of solvents often remain in the product. Hyphenated techniques such as TGA-MS are ideal to detect and identify such undesired residues. In the example, methanol and acetone were used to recrystallize the active substance. The presence of these two substances is confirmed by the peaks in the m/z 43 and m/z 31 fragment ion curves. The results indicate that the weight loss step at 200 °C is almost entirely due to the elimination of acetone.

Adjustment of temperature and heat flow



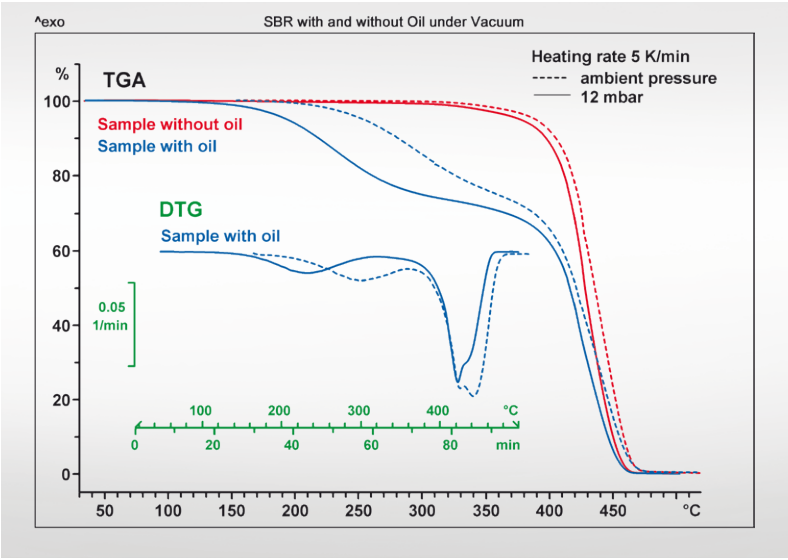
Adjustment of temperature and heat flow is normally performed with certified pure metals. Gold and palladium can be used to calibrate and adjust the temperature and heat flow up to the maximum temperatures specified for the TGA/DSC 3+ furnace (1100 or 1600 °C). The Curie temperatures of ferromagnetic metals can also be employed for temperature adjustment. This is however, not recommended because Curie temperatures are not clearly defined, in contrast to melting points of pure metals.

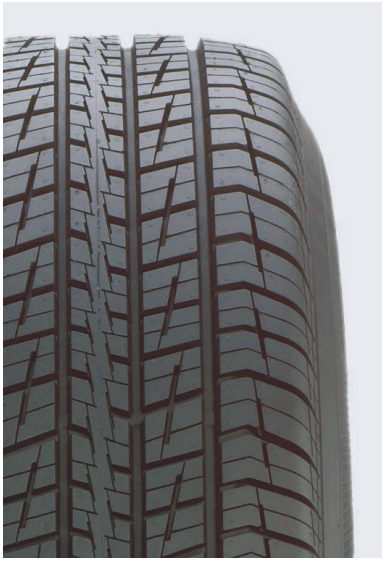
MaxRes: high resolution despite short measurement times



With MaxRes™, the heating rate changes automatically depending on the rate of change of weight. This enables overlapping weight loss steps to be optimally separated in the shortest possible time. This example shows the dehydration of copper sulfate pentahydrate. At 25 K/min, the first two weight loss steps are not properly separated. Using MaxRes, the separation is more clear than using a slower heating rate of 5 K/min, even though the overall measurement time is shorter.

Determination of the plasticizer content in elastomers



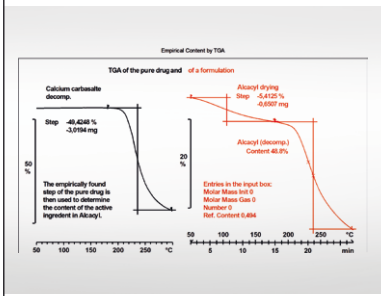


Oils are often used as plasticizers in elastomers. Usually, the oil vaporizes in the same temperature range as the elastomer decomposition begins, making it difficult to quantify the oil content. In such cases, the elastomer samples are measured at reduced pressure to separate the two effects. The example shows the weight loss curves of SBR samples with and without oil at normal pressure and at 12 mbar. Pressure hardly influences the measurement curve of SBR without oil. In contrast, when SBR with oil is measured at reduced pressure, the vaporization of the oil and decomposition of the elastomer are almost completely separated.

Simple, Intuitive Operation
Straightforward, Efficient and Secure

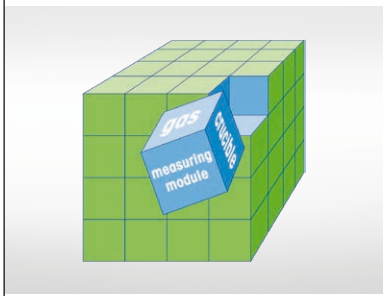
STAR^e software has been expanded to include new features that help you prepare your TGA/DSC 3+ instrument for specific experiments, develop methods for advanced analyses and perform flexible result evaluations. Complex measurement programs are set up within minutes and the vast range of available tools permit curves to be evaluated accurately and efficiently.

TGA Evaluation



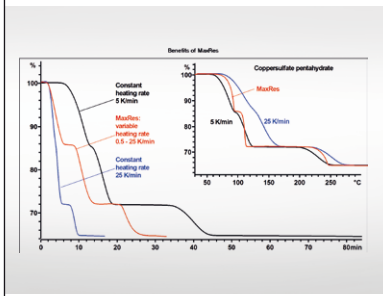
Detailed analysis of the weight loss steps requires the evaluation methods included in the TGA Evaluations option. The evaluation possibilities include normalization to sample weight, percentage content, stoichiometric content, empirical content and conversion.

FlexCal™



With FlexCal, a standard feature on STAR^e Software, users can be confident in their results. The correct adjustment and calibration parameters are selected for heating rate, temperature, purge gas and crucible types.

MaxRes™



When selecting parameters for a TGA or TMA experiment, the user often has to choose between two conflicting goals (shortest possible experimental time or optimum resolution). MaxRes automatically adapts the heating rate to achieve the best possible resolution and separation of closely lying thermal effects.

A complete thermal analysis system consists of the basic six complementary measuring techniques, each bringing fast and accurate results. Additional knowledge can be obtained by means of several hyphenated techniques.

www.mt.com/ta-software

World-Class Service and Support
Provide Results You Can Trust

METTLER TOLEDO's portfolio of services is designed to ensure the continuous performance and reliability of your thermal analysis systems. Factory-trained in Switzerland, our worldwide teams bring the professional expertise and know-how needed to provide you with the highest level of after-sales support, as well as the experience necessary to optimize services for your own particular needs.

Large library of overview and how-to videos

Increase your productivity by learning from our video libraries and more than 600 applications.

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We offer effective classroom training sessions.

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For self-training purposes, you may purchase the tutorial kit consisting of 23 well-chosen application examples and their corresponding test substances.

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Bi-annual application magazine

Every year, thermal analysis generates a large number of scientific results and discoveries. Interesting examples from different application fields and industries are published in our UserCom magazine.

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Sign up to receive quarterly news on the latest trends in thermal analysis, including applications, webinars, courses and how-to-videos.

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We offer Thermal Analysis e-Trainings. An e-Training consists of an e-Learning course and a live webinar. Take part remotely and work together with our experts to enhance your skills and understanding of thermal analysis.

www.mt.com/ta-etraining

TGA/DSC 3+ Specifications

Temperature data	Small furnace (SF)	Large furnace (LF)	High temp. furnace (HT)
Temperature range	RT to 1100 °C	RT to 1100 °C	RT to 1600 °C
Temperature accuracy [1]	± 0.25 K	± 0.3 K	± 0.5 K
Temperature precision [1]	± 0.15 K	± 0.2 K	± 0.3 K
Furnace temperature resolution	0.001 K	0.001 K	0.002 K
Heating time	5 min (RT to 1100 °C)	10 min (RT to 1100 °C)	10 min (RT to 1600 °C)
Cooling time	20 min (1100 to 100 °C)	22 min (1100 to 100°C)	27 min (1600 to 100 °C)
Cooling time with helium	≤ 10 min (1100 to 100 °C)	≤ 11 min (1100 to 100 °C)	≤ 13 min (1600 to 100 °C)
Heating rate [2]	250 K/min	150 K/min	100 K/min
Cooling rate [2]	–20 K/min (≥ 150 °C)	–20 K/min (≥ 150 °C)	–20 K/min (≥ 200 °C)
Sample volume	≤ 100 µL	≤ 900 µL	≤ 900 µL

Special modes			
Automation	optional		
MaxRes			
TGA-MS, TGA-FTIR, TGA-GC/MS, TGA-Micro GC/MS			
Vacuum	> 10 mbar	> 10 mbar	> 10 mbar
TGA sorption	no	optional	

Balance data	XP1	XP1U	XP5	XP5U
Measurement range	≤ 1 g	≤ 1 g	≤ 5 g	≤ 5 g
Resolution	1.0 µg	0.1 µg	1.0 µg	0.1 µg
Weighing accuracy	0.005%	0.005%	0.005%	0.005%
Weighing precision	0.0025%	0.0025%	0.0025%	0.0025%
Repeatability	< 0.001 mg	< 0.0008 mg	< 0.002 mg	< 0.0009 mg
Typical Minimum Weight [3]	0.19 mg	0.16 mg	0.22 mg	0.17 mg
Typical Minimum Weight USP [3,4]	1.9 mg	1.6 mg	2.2 mg	1.7 mg
Internal ring weights	2			
Blank curve reproducibility	better than ± 10 µg over the whole temperature range			

Calorimetric data				
Sensor data (typical values)	**Sensor type**	**SDTA**	**DTA**	**DSC**
	Surface material	platinum	platinum	ceramic
	Number of thermocouples	1	2	6
	Signal time constant at 900 °C	15 s	14 s	14 s
	Sensitivity	0.5 mW	0.2 mW	0.1 mW
	Temperature resolution	0.005 K	0.0001 K	0.00003 K
Enthalpy reproducibility (standard deviation)		better than 5%		

Sampling	
Sampling rate	maximum 10 values/second

Approvals

IEC/EN61010-1:2001, IEC/EN61010-2-010:2003
CAN/CSA C22.2 No. 61010-1-04 & -2-010
UL Std. No. 61010-1 (2nd Edition)
EN61326-1:2006 (Industrial environments)
EN61326-1:2006 (class B) AS/NZS CISPR 11, AS/NZS 61000.4.3

[1] based on metal standards
[2] depends on instrument configuration
[3] depends on instrument environment and condition
[4] USP = United States Pharmacopeia

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For more Information

METTLER TOLEDO Group
Analytical Division
Local contact: www.mt.com/contacts

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