KEP Technologies

THEMYS

Thermal Analysis Platform

TGA, HP-TGA, STA, DTA/DSC, EGA, TMA

REIMAGINE MATERIAL CHARACTERIZATION

setaram
KEP TECHNOLOGIES

CUSTOMIZED SOLUTIONS

Modular design allowing for upgraded and tailored functionality
Access to all previous non-proprietary custom requests
Open access to engineering development team

ULTRA-HIGH TEMPERATURE CAPABILITY
to 2400°C with a single furnace

VARIETY OF ATMOSPHERE CONDITIONS
multiple carrier and reactive gas options

HIGH ACCURACY & VERSATILE
hang-down symmetrical beam balance designed for TGA applications

MODULAR ADAPTATIONS
up to 2400°C : *TGA, DTA, TG-DTA, TMA*
up to 1600°C : *DSC, TGA-DSC,*

EXTERNAL COUPLING CAPABILITY
to increase your research options

ACCURATE & SENSITIVE MEASUREMENT
tri-couple DTA technology

PRESERVATION OF SAMPLES
in TMA up to 2400°C

BALANCES

Because the core of a thermogravimetric analyzer is its balance, SETARAM Instrumentation designs balances **exclusively for thermal analysis applications.**

Three models of balance are offered with THEMYS to cover the whole range of applications. They are all based on the hang-down design, providing the highest level of stability and the best detection limit.

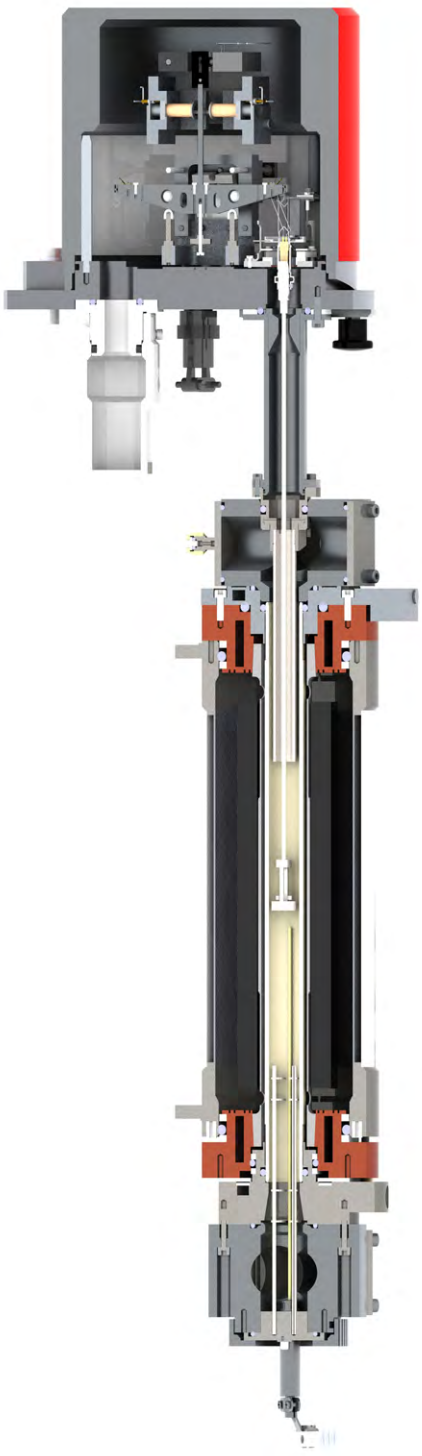
The **HIGH SENSITIVITY** balance is designed for the accurate study of very small mass variations. Its 35g loading capacity still allows the characterization of heavy samples. It has the lowest background noise level, best detection limit and the best isothermal drift . This balance is typically the ideal choice for long term corrosion kinetics.

The **HIGH CAPACITY** balance has a +/- 3 000 mg mass variation range, which makes it perfect for experiments that lead to the full decomposition of large samples, as with heterogeneous materials.

The **HIGH VERSATILITY** balance is equipped with the AUTO-TARE system. It is the perfect choice to take full advantage of the THEMYS platform modularity, when frequent changes of samples types, crucibles, or other experimental conditions are required. The HIGH VERSATILITY balance also benefits from an excellent signal drift under temperature scanning conditions and from within +/- 200 mg and +/- 2 000 mg mass variation ranges.

FURNACE

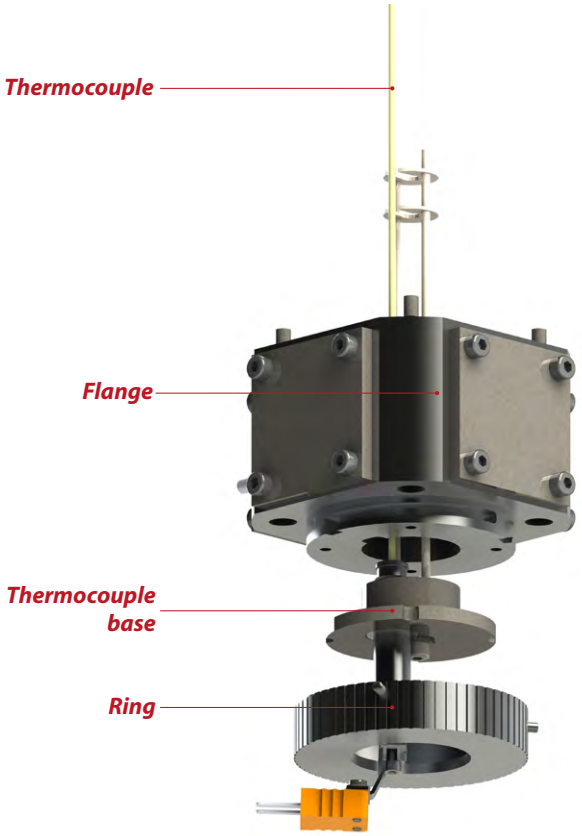
Heat transfer specialists know that a high temperature **furnace with small dimensions means large temperature gradients.** This is why THEMYS is designed around a single, robust and high performance graphite furnace protected by an 18mm internal diameter alumina tube. This ensures a l**arge homogeneous temperature zone** as needed by diverse experiments: small or large samples, small TGA crucibles or large tricouple DTA rods, etc. This also avoids the inconvenience of changing furnaces when experiment conditions change.
This furnace principle is consistent with SETARAM's focus to provide systems with the **lowest operational costs in the market**.



SCHEMATICS OF THE THEMYS BALANCE, FURNACE AND TEMPERATURE CONTROL

TEMPERATURE CONTROL

A choice of high sensitivity thermocouples to cover various temperature ranges is provided with a simple TWIST AND LOCK system.
It is fast, does not require tools, and the thermocouple's temperature range is detected automatically.
For challenging samples and atmospheric conditions, sleeved thermocouples (alumina or silica) are available to enhance corrosion resistance.



SCHEMATICS OF THE **TWIST AND LOCK** SYSTEM

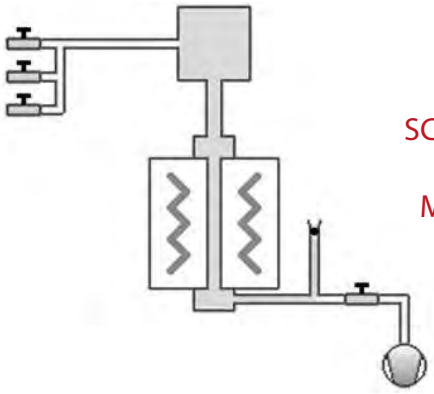
ATMOSPHERE CONTROL

As most TGA or STA applications are linked with solid-gas reactions, THEMYS provides accurate control of the furnace atmosphere, with different options including mass flow controllers, gas switching and blending devices, vacuum pumps and gauges.

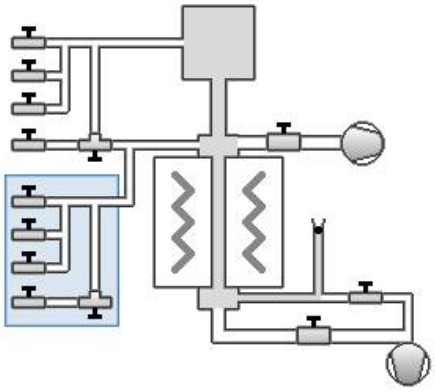
GAS FLOW MANAGEMENT

The gas flow management system is designed to be the most flexible and easy to use.

Three options, **PureGas, GasBlend** and **MultiGasBlend**, are available with an increased capacity to generate and control complex atmospheres. They all have in common an intelligent software control system for sequentially changing the gas types, flowrates, or blend ratios during the experiment or sample pretreatment. It also warns the user in the case of an incompatible or hazardous gas blend.



SCHEMATIC REPRESENTATION OF THE PUREGAS (LEFT) AND MULTIGASBLEND (RIGHT) OPTIONS



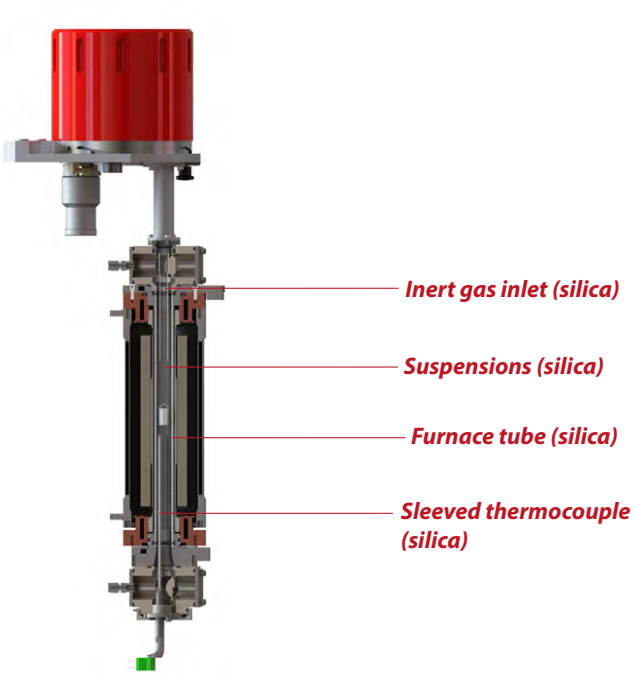
		OPTION		
FEATURES		**PureGas**	**GasBlend**	**MultiGasBlend**
Carrier gas (generally inert)	Connected to the TGA platform	Up to 3 gases	Up to 3 gases	Up to 3 gases
	Flowing in the furnace	1 amongst the 3	1 amongst the 3	1 amongst the 3
Auxiliary gas (generally active)	Connected to the TGA platform	-	1	Up to 5
	Flowing in the furnace	-	1	1 pure OR a blend of up to 3 of the connected gases
Mass flow controllers		1 inside structure	2 inside structure	2 inside and 2 outside structure (external gas panel)
Sequential programming		Yes	Yes	Yes

VACUUM

Primary, Forced Primary or Secondary Vacuum options are available with a selection of adapted vacuum pumps. New pre-programmed procedures for stepwise sample evacuation are available with Calisto software.

CORROSIVE GASES

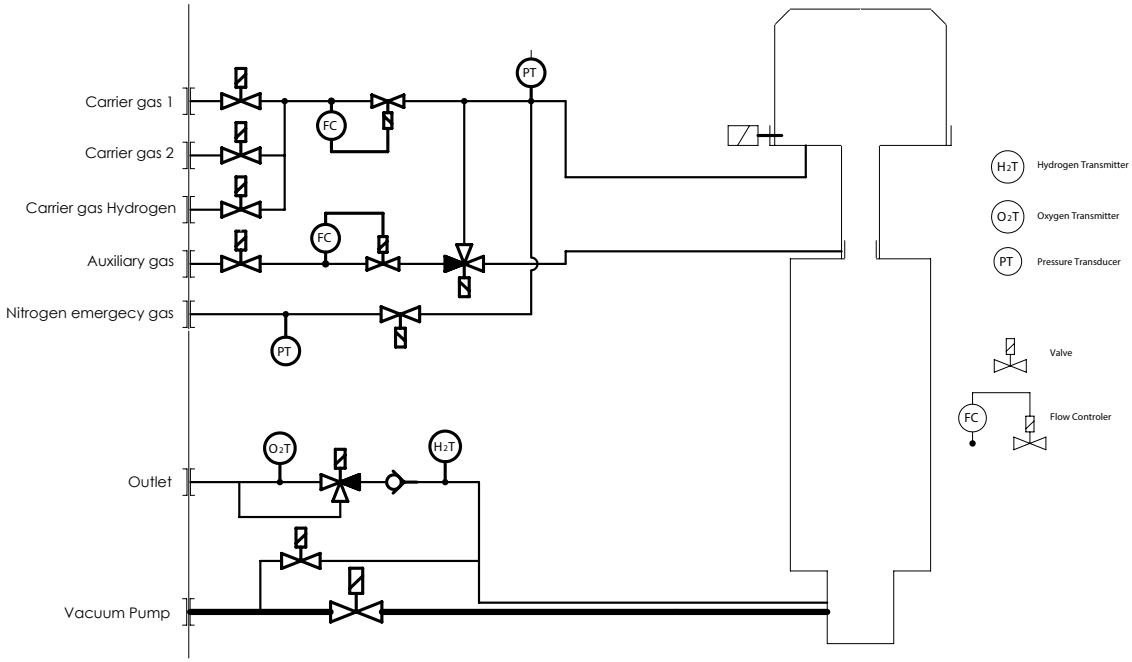
Specific accessories are designed for samples under really harsh conditions while protecting the thermal analyzer. Sleeved thermocouples (alumina or silica) and protected DTA rods are available. Custom designed solutions can be developed for specific testing situations. An accessory is also available to carry out TGA measurements in atmospheres containing 100% corrosive gas. In such a configuration, the standard furnace tube is changed for a tube made of silica.
The temperature control thermocouple is sleeved with silica, the suspensions and crucibles are made of silica. In that configuration, the corrosive gas flows bottom-up, and the balance is protected by a top-down flow of an inert gas.



HYDROGEN ATMOSPHERE

THEMYS can be provided in a specific configuration for operations under H_2 (up to 100%):

- It includes hydrogen and oxygen probes to protect the system and user from blending these gases in the THEMYS furnace and from opening the furnace when THEMYS operates under H_2.
- This configuration is designed to preserve the oxygen probe during standard tests that do not require hydrogen.
- It is also equipped with an inert gas emergency line that injects N_2 in the system in case of hazardous situations.



MOISTURE, WET ATMOSPHERE

THEMYS can be coupled with WETSYS, our wet atmosphere generator. It generates a humid carrier gas up to 90% relative humidity at 70 °C. Possible carrier gases include Air, He, N_2, CO_2, etc. Complex humidity profiles including steps can be programmed. THEMYS can be used up to 1 750 °C with a wet gas.

TGA ACCESSORIES

The large loading capacity of the balances and the large homogeneous temperature zone of the furnace allows the user to choose the right crucible for the right experiment:

- Choose standard cylindrical crucibles or more dedicated ones, like meshed or three-tray crucibles for better gas sample interactions
- Choose the volume related to your sample size, ranging from 130 µl to 2.5 ml
- Choose the most inert crucible material for your sample, atmosphere and temperature range: finest quality platinum, tungsten, quartz, alumina, or graphite

Note that bulk materials can also be held directly on a suspension hook without a crucible, for the best gas sample interactions.

Quartz V: 55 D: 8	**Quartz** V: 350 D: 10	**Quartz** V: 1 570 D: 12	**Alumina** V: 170 D: 7	**Platinum** V: 130 D: 8	**Platinum** V: 1 500 D: 12	**Alumina** V: 1 300 D: 12 or V: 2 500 D: 14	**Meshed platinum** V: 1 800 D: 13	**Tungsten** V: 560 D: 10	**Graphite** V: 550 D: 10	**3-plate Platinum** V: 800 D: 13	**Sample hung without crucible**

MOST FREQUENTLY USED TGA CRUCIBLES – DIAMETERS (D) IN mm, VOLUMES (V) IN µL DTA,

DTA, DSC, STA (TG-DTA, TG-DSC) ACCESSORIES

TRICOUPLE DTA

Protected tricouple DTA

DSC

DTA

OUR RANGE OF DTA AND DSC SENSORS FEATURE:

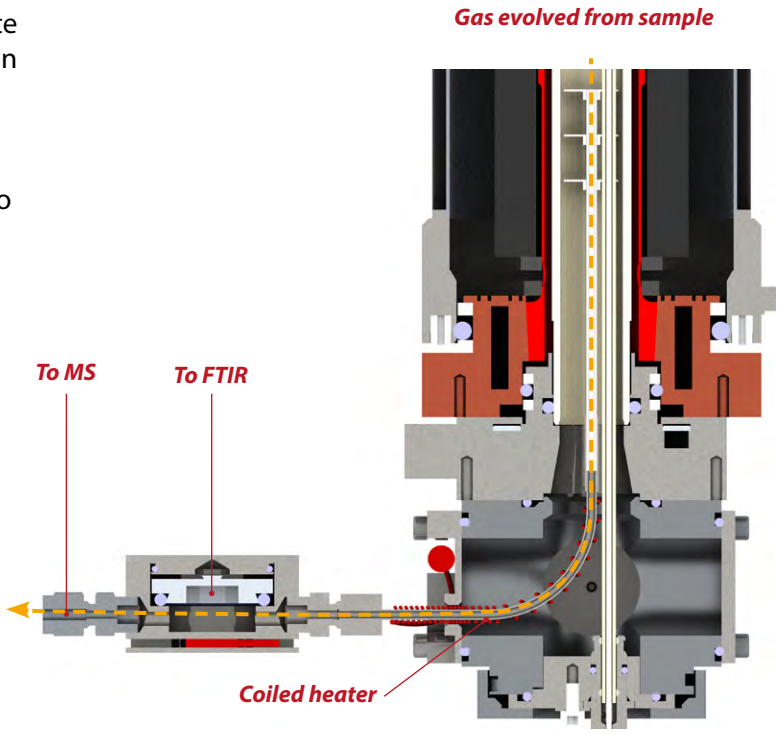
- Temperature ranges up to 800, 1 000, 1 500, 1 600, 1 750, 2400 °C
- The most sensitive thermocouple wires available within each temperature range, including the **type R** thermocouples
- Tricouple DTA systems for enhanced sensitivity even at the highest temperatures
- Protected DTA rods for prolonged thermocouple life even when corrosive species are evolved from the samples
- **Easy Fit** connectors for rod exchange in seconds
- The finest quality metal (aluminum, platinum, tungsten) or ceramic (alumina, zirconia, yttria, graphite) crucibles, with volumes from 20 to 300 µl

EVOLVED GAS ANALYSIS

Evolved Gas Analysis (EGA) enhances the thermogravimetric analyzers capacity to investigate the sample's composition or thermal decomposition chemistry, thanks to the identification of the evolved species by a gas analyzer.

The THEMYS gas sampling system was designed to feature:

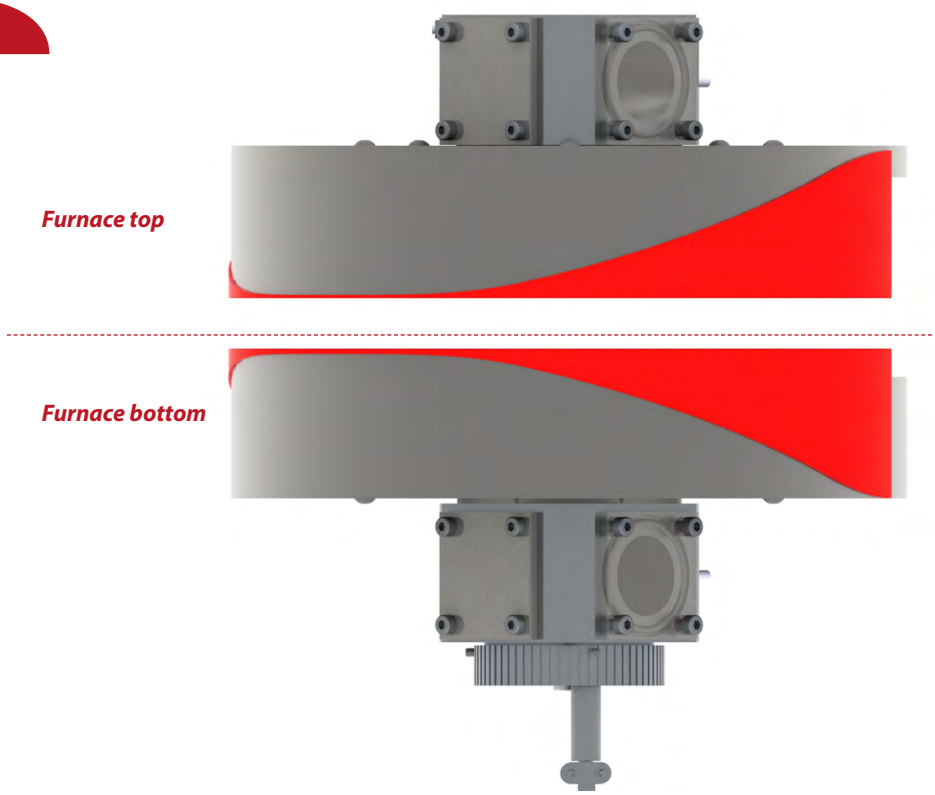
• Coupling to the main gas analysis techniques: Mass spectrometry (MS), FTIR spectrometry, gas chromatography (GC) and combinations of these : TG-MS, TG-FTIR, TG-MS-FTIR, TG-GC/MS
• Transfer lines and parts made to ensure accurate temperature control up to 300 °C, avoiding cold condensation points
• Advanced sampling/gas injection strategies for GC/MS analyses with the unique Auto-Injector system
• Quantitative EGA after calibration using the titration TGA option
• Gas Analyzers data integration in the Calisto thermal analysis software



HEATED COUPLING PART FOR EVOLVED GAS ANALYSIS

EXTRA COUPLING

The THEMYS furnace allows for in-situ analyses with two 4-port parts placed at its top and bottom. Up to 8 extra sensors can be connected (some of the ports may already be occupied with options like vacuum, gas flow or standard EGA coupling).
Extra humidity, oxygen or other measurements can be achieved. Discuss your specific needs with our engineers.



TOP AND BOTTOM FLANGES WITH AVAILABLE PORTS

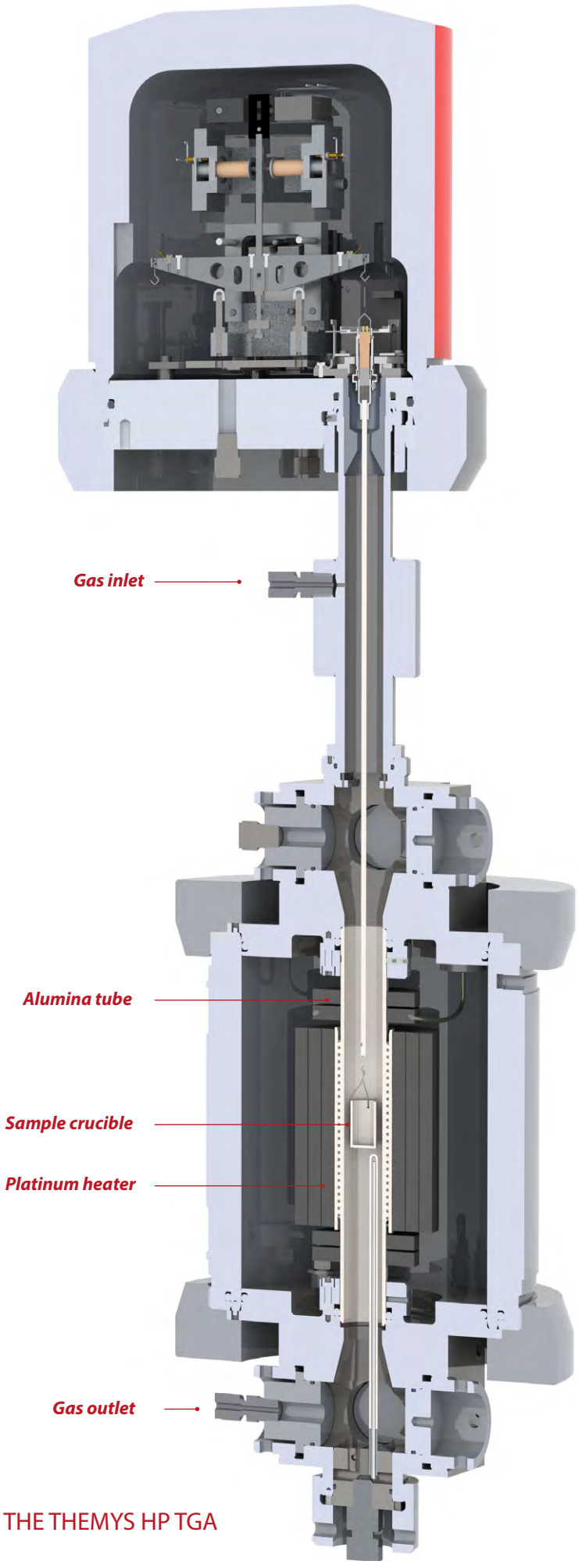
HIGH PRESSURE TGA

THEMYS HP is a robust, flexible high pressure thermogravimetric analyzer featuring:

- **HIGH PRESSURE** model, a specific pressure-rated balance. It is designed based on the vertical hangdown principle for the best mass signal accuracy and stability.

- An alumina protected furnace with a platinum based heating element for isothermal or temperature scanning operations at heating rates up to 100 °C/min.
  The same furnace and balance cover a temperature and pressure range **up to 1 200 °C and 150 bar** with the best TGA detection limit. It avoids multiple furnace changes for different samples or test conditions.

THEMYS HP is typically applied to the study of pyrolysis, combustion, gasification conditions met in many coal, biomass or waste to energy processes. It is also designed for high temperature and pressure corrosion studies, and more particularly with alloys used in the above mentioned processes.
THEMYS HP is the ideal tool for sorption and desorption analysis in the fields of gas capture and storage, gas sensing materials characterization, or more generally sorbent materials characterization.



SCHEMATICS OF THE THEMYS HP TGA

THERMOMECHANICAL ANALYSIS (TMA)

THEMYS platform modularity is perfectly illustrated by the TMA module, which can be easily and quickly exchanged with a balance by a trained user, without the need to involve SETARAM experts.
THEMYS TMA is built on a vertical design. The TMA probe is held by an electromagnetic suspension system so that virtually no load is applied on the sample.

Many benefits arise from this vertical design:

- Soft materials can be accurately tested, e.g. when sintering of powders in a soft binder need to be evaluated
- For volumetric measurements on powders, they can be placed in a cylindrical container
- Traction measurements are also possible on films and fibers

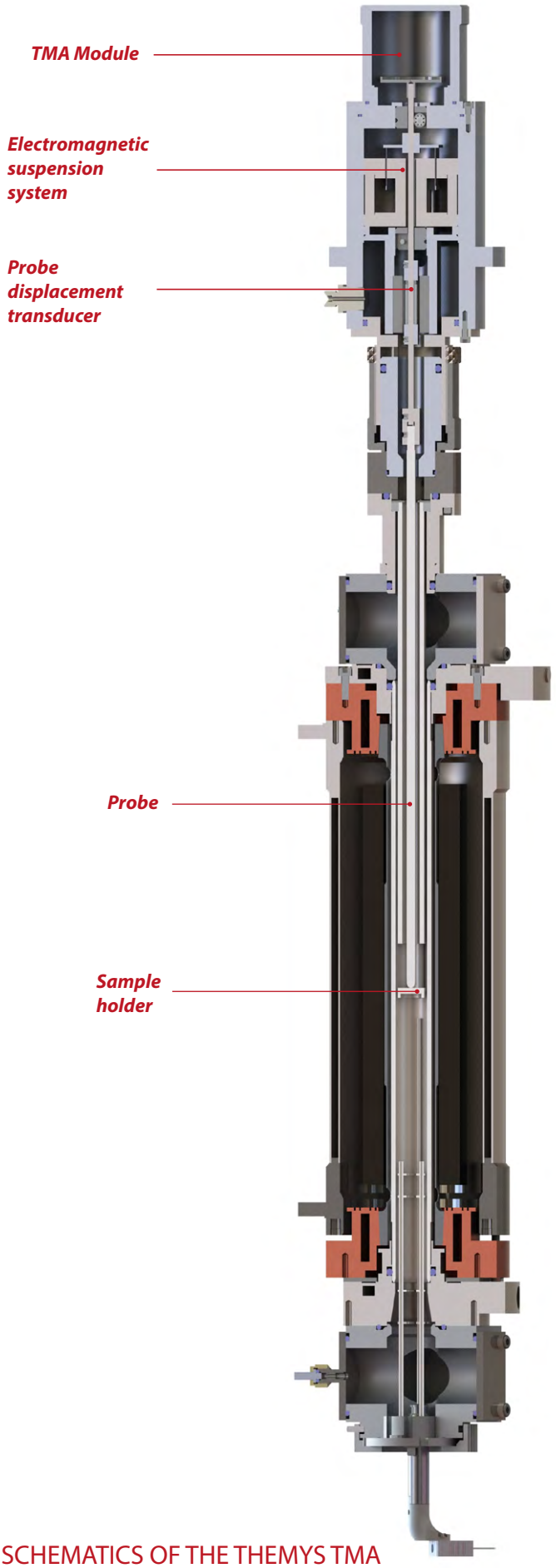
With its sensitivity, THEMYS TMA can detect **very small dimensional changes** and there is no need for large samples for accurate measurements. Both transducer calibration and load are computer controlled for ease of operation.
Various types of probes are available, depending on the sample type, probe material and measurement to be carried out:

- Compression
- Penetration
- Flexure
- Traction
- Volume expansion

The probes are made of silica (for use up to 1000°C), alumina (for use up to 1750 °C), or graphite (for use up to 2400 °C).

CONTROLLED SINTERING RATE

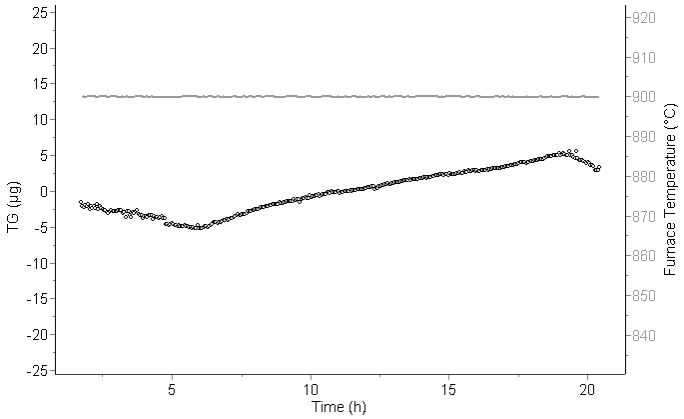
Properties of a sintered material are improved by maintaining a constant and slow sintering rate during densification. Key parameters are maximum heating rate and sintering rate. With THEMYS TMA, the heating rate can be **automatically adjusted to follow a preset sintering rate**. This mode is used to **generate an optimized temperature profile** and to simulate the sintering process.



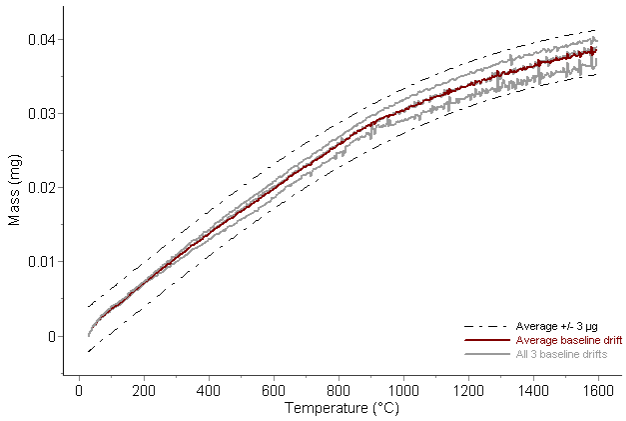
SCHEMATICS OF THE THEMYS TMA

ISOTHERMAL STABILITY

A stable signal under isothermal conditions is required for many gas-solid experiments like oxidation, reduction, adsorption etc.
The High Sensitivity balance exhibits ground breaking stability as shown with this experiment at 900°C, leading to +/- 5 µg variation over a period of 20 hours, i.e. a 0.5 µg/h average and a maximum value less than 2 µg/h.



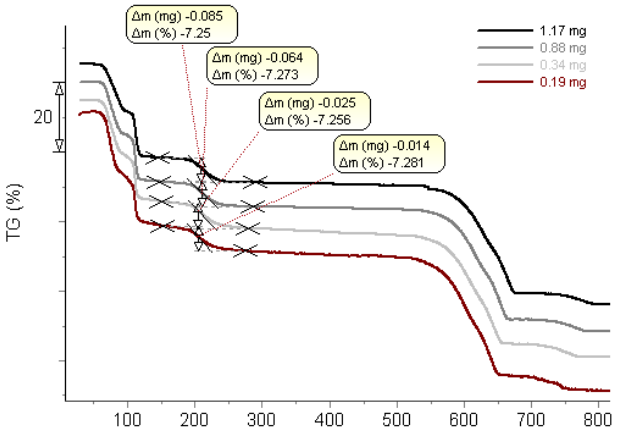
BASELINE DRIFT AND REPEATABILITY



A low TGA baseline drift under temperature scanning conditions is highly desirable for most experiments, leading to large sample mass variations that do not require baseline subtraction.
High baseline drift precision is required for the small sample mass variation experiments when baseline subtraction is necessary.
The opposite chart proves the excellence of THEMYS High Versatility balance in that respect. Data is based on six repeated experiments from 50 °C up to 1 700 °C, at a rate of 10 °C/min. Helium flow of 20 mL/min.

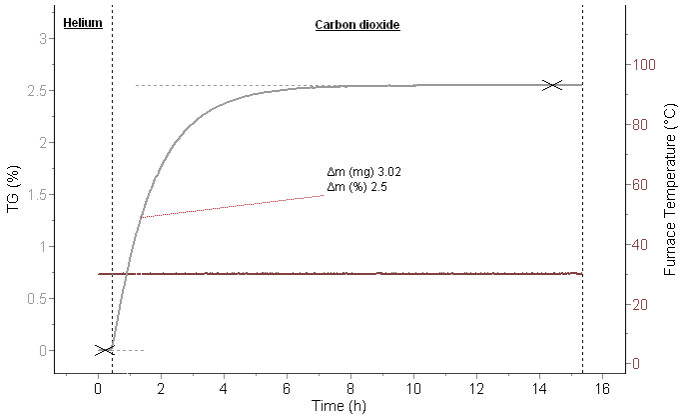
SMALL MASS LOSSES

This test series, with decreasing initial sample masses, shows the strong performance of the High Sensitivity balance, with an accurate quantification of a 0.014 mg mass loss corresponding to one step in the dehydration of $CuSO_4 \cdot 5H_2O$.

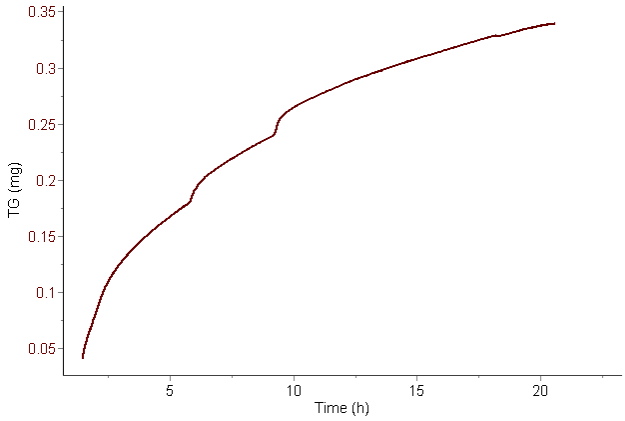


GAS SORPTION

Testing gas sorption properties is essential for porous materials characterization. The vacuum and gas flow management options of THEMYS were designed for that purpose. This example shows the adsorption of carbon dioxide on a ZIF-8 sample at 30 °C. It required a series of heating under vacuum, cooling and gas change at a test temperature that was all programmed and then operated automatically. Sorption capacity and kinetics data can be derived from such a chart.



HIGH TEMPERATURE CORROSION



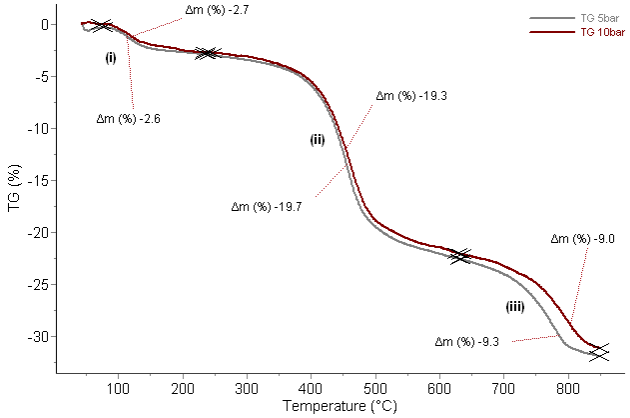
High temperature, corrosion resistant superalloys like the tested INCONEL 600 sample need to be characterized in terms of their oxidation kinetics.

These characterizations typically require the TGA instrument to provide good gas-solid interface, high mass signal stability over long periods of time, and efficient control of the sample atmosphere. At 900 °C the total mass gain of the sample was no more than 298.5 µg, i.e. 0.395mg/cm2. This demonstrates the ability of the TGA instrument to measure small effects over time, thanks to the combination of its high sensitivity and stability.

The bumps on the mass uptake curve at about 6 and 10 hours are linked with the formation of cracks in the oxide layer.

COAL PYROLYSIS

TGA in that field is ideal for characterizing coal feedstocks and provides coal pyrolysis data valuable for the industrial process, like pyrolysis conversion and rate. The chart compares the TGA profile of the same coal from Longkou (Shandong, China) with a drying step and two devolatilization steps (lighter and heavier hydrocarbons).



APPLICATIONS

Depending on its configuration (TGA, STA, TMA) and mode of operation (isothermal, temperature scanning), THEMYS can be applied to the characterization of organic or inorganic materials ranging from polymers, composites, oils, coal, to ceramics, cements, metals and nanomaterials. It is the perfect tool to understand the thermal behavior of materials that are exposed to temperature increase during their production, service life, or recycling. The objectives of TGA or STA experiments are the characterization of material composition, of their thermal resistance, of their corrosion resistance properties, of the efficiency of their synthesis route, of their regeneration / recycling conditions and the detection of their phase transitions. THEMYS TMA provides material thermal expansion data or sintering properties.
High pressure processes like pyrolysis, combustion and gasification are best studied by THEMYS HP, whether it concerns the fuels (biomass, coal…) or the structure of materials (alloys, ceramics…).
THEMYS and THEMYS HP are also meant to characterize the sorption properties of catalysts or porous materials under low, atmospheric or high pressure.

View the application notes in your field, available for download, by visiting www.setaram.com. A huge database is in the application library area of our website. We have also included a powerful search engine that will enable you to find the most applicable data.

SPECIFICATIONS

GENERAL		TGA			HP TGA	STA		TMA
						DTA, TG-DTA	DSC, TG-DSC	
Temperature range		RT to 2 400 °C			RT to 1 200 °C	RT to 2 400 °C	RT to 1 600°C	RT to 2 400°C
Programmable heating rate		0.01 to 100 °C/min			0.01to100 °C/min [a]	0.01 to 100 °C/min		
Crucible volumes or maximum sample size		55 to 2 500 µl or Height: 20 Diam: 14 mm without crucible			1 300 µl	30 to 300 µl	80 to 100 µl	Height : 20 Diam : 10 mm
Vacuum		Primary (<1mbar), forced primary (<5.10^{-2} mbar), secondary vacuum options						
BALANCE		**HIGH SENSITIVITY**	**HIGH VERSATILITY**	**HIGH CAPACITY**	**HIGH PRESSURE**			
Measuring range (mg)	Small	+/- 5	+/- 200	+/- 300	+/- 200			
	Large	+/- 50	+/- 2 000, AUTO-TARE	+/- 3 000	+/- 2 000			
Maximum loading capacity		35 g	35 g	100 g	35 g			
TGA baseline drift (temperature scanning) [b, c]		30µgupto1000°C 40µgupto1600°C	35µgupto1000°C 50µgupto1700°C	<100µgupto1700°C	- [d]			
TGA baseline drift precision [c]		+/- 3 µg	+/- 10 µg	-	+/- 200 µg			
Balance resolution (small range)		0.00059 µg	0.023 µg	0.03 µg	0.023 µg			
DTA/DSC						**DTA, TG-DTA**	**DSC, TG-DSC**	
Calorimetric precision [c, e]						+/- 2 %[f]	+/- 1 %	
Temperature precision [c, e]						+/- 0.8 °C	+/- 0.4 °C	
Temperature accuracy [c, e]						+/- 0.4 °C	+/- 0.25 °C	
TMA								
Resolution								0.2 nm
Measuring range								+/- 2 mm

a. Value at P_{atm} may vary according to pressure / b. Under helium flow / c. Typical data / d. Pressure dependent / e. Based on metal standard melting / f. If calibrated / RT = Room Temperature
Specifications are subject to change

Option : AKTS Thermokinetics software for comprehensive investigation of reaction or decomposition

Switzerland – France – China – United States – India – Hong Kong
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