

SETSIS Evolution

**High modularity
Thermal Analyzers
DTA, DSC, TGA,
TGA-DTA/DSC,
TGA-EGA, TMA**

State of the Art

- CALISTO software



SETSYS Evolution

The best in class

The SETSYS Evolution line of analyzers is now the reference in their area of measurement.

SETSYS Evolution line has been designed in order to develop ever-increasing possibilities of analysis, measurement safety and ease of use from its devices.

- **The widest temperature range**

With a measurement range from ambient temperature to 2400°C, SETSYS Evolution offers the widest temperature range on the market

- **Modularity**

The different measurement modules (DTA, DSC, TGA, TMA) can be adapted interchangeably around the same structure comprising the furnace, electronics, gas circuits, atmosphere control, etc.

- **Measurement performance**

All the SETSYS Evolution measurement modules satisfy unique resolution, precision and sensitivity criteria.

In addition, the SETSYS Evolution line benefits above all from major technological advances for the management of gas circuits and safety features (see page 3).

A digital display on the front panel provides fast access to data such as furnace temperature, carrier gas and auxiliary gas flows, pressure in the furnace and in the furnace protection chamber, etc.

SETSYS Evolution is today the state-of-the-art thermal analyzers.



DTA:

Differential thermal analysis is a technique that measures the difference in temperature between a sample and a reference (a thermally inert material) as a function of time and temperature, when they are subjected to a temperature program in a controlled atmosphere. The DTA method makes it possible to detect any change in all categories of material.

DSC:

Differential scanning calorimetry is a technique used to determine the variation of thermal flows emitted or received by a sample when subjected to a temperature program in a controlled atmosphere. When heating or cooling, any change occurring in the material is accompanied by an exchange of heat: DSC enables the temperature of this transformation and the quantity of the heat produced to be determined.

TGA:

Thermogravimetry is a technique that measures the variation of mass of a sample when it is subjected to a temperature program in a controlled atmosphere. This variation of mass can be a loss (vapor emission) or a gain (fixing of gases).

TMA:

Thermomechanical analysis is a technique that measures the deformation of a sample under non-oscillating stress when subjected to a temperature program in a controlled atmosphere. The stresses applied can be compression, tensile or bending stresses.

A common structure

The DTA, DSC, TGA and TMA analyzers of the SETSYS Evolution line adapt to a common structure housing the furnace, electronics, gas circuits, atmosphere control, etc.

• Four temperature versions

Four different versions of structure are available depending on the nature of the furnace and temperature regulation.

| | SETSYS Evolution 12 | SETSYS Evolution 16 | SETSYS Evolution 18 | SETSYS Evolution 24 |
|---------------------------------------|-----------------------|-----------------------|----------------------------|---------------------------|
| Temperature range (°C) | Ambient to 1200 | Ambient to 1600 | Ambient to 1750 | Ambient to 2400 |
| Furnace | Metal | Graphite | Graphite | Graphite |
| Thermocouple (temperature regulation) | Type S Pt/PtRh 10% | Type S Pt/PtRh 10% | Type B PtRh 6%/PtRh 30% | Type W5 WRe 5%/WRe 26% |
| Scanning rates (°C/min) | 0.01 to 50 | 0.01 to 100 | 0.01 to 100 | 0.01 to 100 |

• Varied and controlled atmospheres

The basic version of the SETSYS Evolution is equipped with a carrier gas circuit whose flow can be adjusted and controlled by an MFC* (range from 0.24 to 12 l/h, precision $\pm 0.2\%$ full scale and $\pm 0.8\%$ of the measurement). A pressure transducer (10 mbar /1.6 bar) measures the pressure in the balance and in the furnace.

A "gas mixture" option comprises the auxiliary gas circuit equipped with an MFC* (range 0.02 – 1 l/h, precision $\pm 0.2\%$ full scale, $\pm 0.8\%$ of the measurement). The carrier and auxiliary gas circuits are linked by a three-way control valve that mixes the two gases in proportions ranging from 50/50 to 1/99.

*MFC: Mass Flow Controller

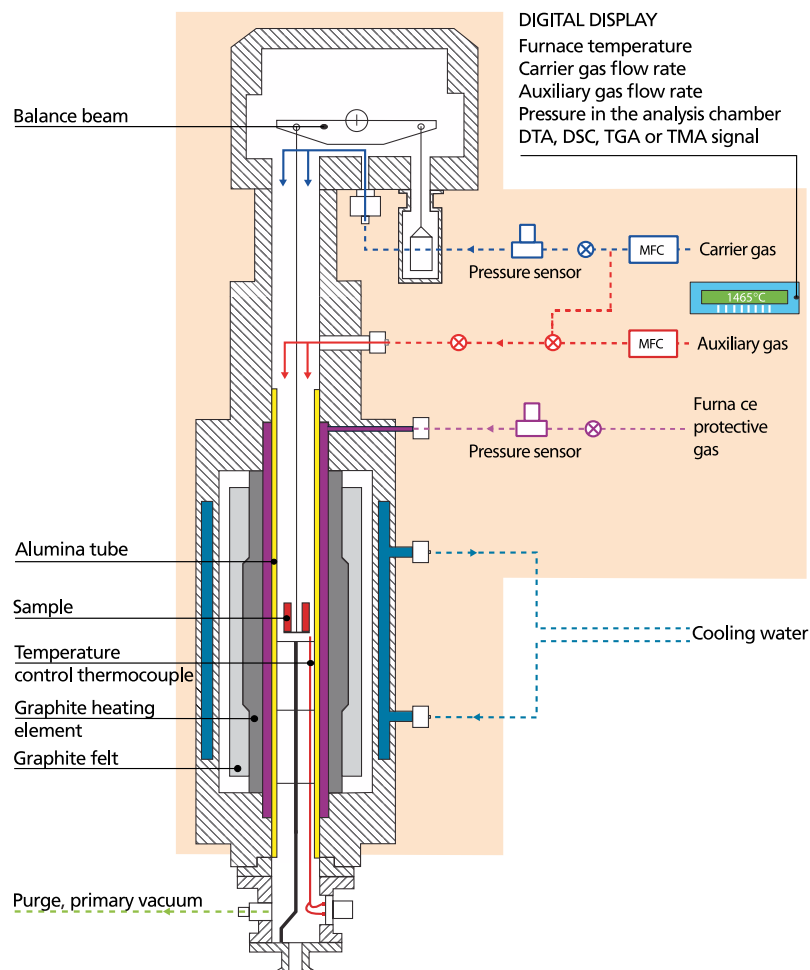
• Safety

As for safety, the furnace cannot be opened if too hot ($T > 70^\circ\text{C}$) or under vacuum. Furthermore, it cuts off in case of runaway.

Starting and stopping of the cooling water and protection gas is automatic.

• Remote electronics

If the SETSYS Evolution has to be installed in a glove box or hot cell, the electronic circuitry can be installed in a separate remote box to prevent it from being exposed to the radiation



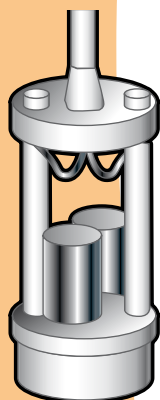
SETSYS Evolution DTA

Measurement up to 2400°C

- **SETSYS Evolution DTA: four models from ambient to 2400°C...**

Four different differential thermal analyzer models have been developed, under the brand-name SETSYS Evolution DTA, covering a wide temperature range from ambient to 2400°C.

Four types of thermocouples are used to produce the various transducers (tricouple, protected, plate) adapted to the different models (see table below).



- **A unique tricouple transducer**

This transducer features three thermocouples for the sample and reference sites resulting in unsurpassed sensitivity and resolution. Furthermore, the symmetry of the detector guarantees excellent base line stability.

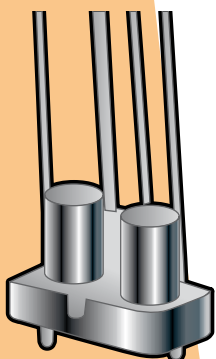
| SETSYS Evolution Instrument | Temperature range of the furnace (°C) | Type of thermocouples | Maximum use range of the rods (°C) |
|-----------------------------|---------------------------------------|-----------------------|------------------------------------|
| DTA 12 | 20 to 1200 | P | 20 to 1000 |
| DTA 16 | 20 to 1600 | S | 20 to 1600 |
| DTA 18 | 20 to 1750 | B | 20 to 1750 |
| DTA 24 | 20 to 2400 | WV5 | 20 to 2400 |

- **A plate transducer for very-high temperature measurements**

High-temperature thermal measurements require the use of tungsten-rhenium thermocouples. To produce such a transducer with the SETSYS Evolution DTA 24 analyzer a tungsten bed-plate is machined with two housings for the crucibles.

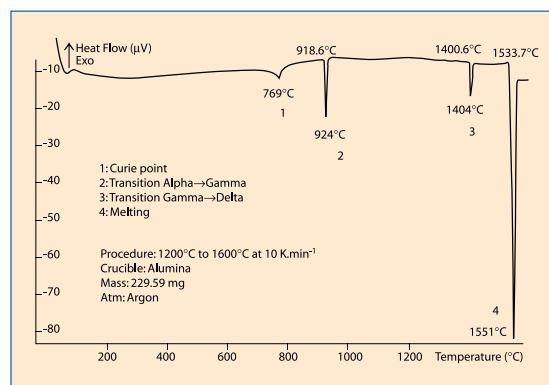
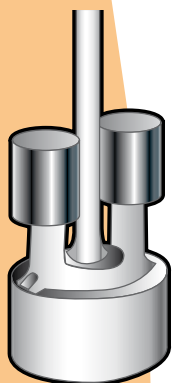
The thermal signal and temperature measurements are provided by the tungsten-rhenium thermocouples which ensure rigidity in the DTA rod.

The detector is essentially dedicated to very-high temperature measurements.

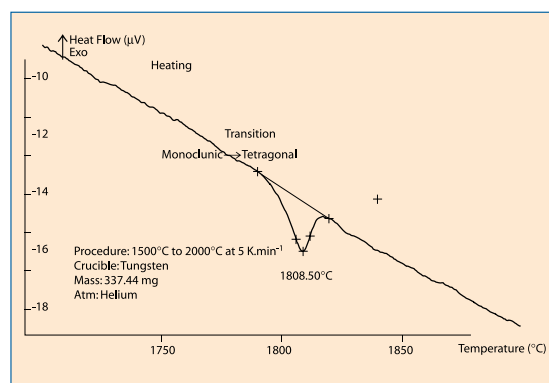


- **A protected transducer**

In the protected DTA rod the measurement and reference thermocouples are covered by the crucibles to protect them if corrosive gas emanates from the sample.



Phase transitions and melting of iron



Phase transitions of HfO₂

SETSYS Evolution DSC

Measurement up to 1600°C

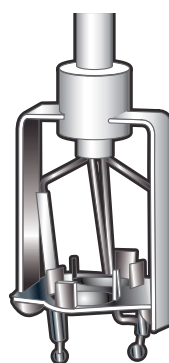
- SETSYS Evolution DSC: two models from ambient to 1600°C**

Two temperature versions of SETSYS Evolution DSC are available. The nature of the DSC plate rod constituents changes according to the desired temperature range.

| SETSYS Evolution Instrument | Temperature range of the furnace (°C) | Type of thermocouples | Maximum use range of the rods (°C) |
|-----------------------------|---------------------------------------|-----------------------|------------------------------------|
| DSC 15 | 20 to 1600 | S | 20 to 1500 |
| DSC 16 | 20 to 1750 | B | 20 to 1600 |

- A DSC-type plate transducer for quantitative measurements**

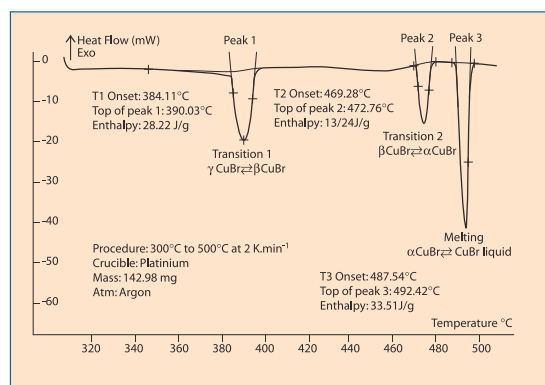
The DSC plate transducer offers an alternative to the use of DTA rods when a quantitative measurement is desired. The DSC plate transducer consists of a machined metallic plate with two housings for the measurement and the reference crucibles. Flat-bottomed crucibles are used to optimize the thermal contact. Small pins ensure correct positioning of the crucible on the transducer.



- SETSYS Evolution DTA/DSC : crucibles adapted to each model of transducer**

Up to 1750°C, platinum, alumina and zirconia crucibles are proposed for tricouple and protected transducers. Three volumes of crucible (20, 100 and 300 mm³) are available according to the mass of sample to be analyzed or the thermal effect to be measured. For limited-temperature use (up to 500°C), aluminum crucibles fitted with lids are appropriate for studying controlled dehydration.

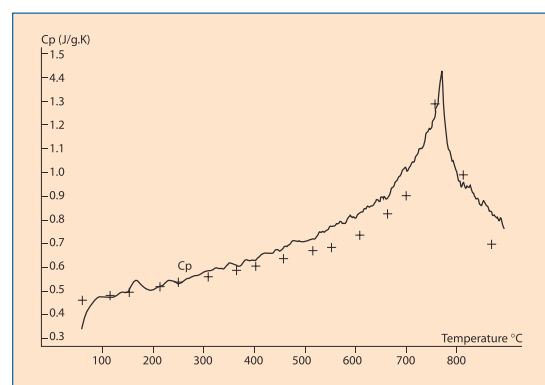
The DSC plate rod requires the use of flat-bottomed alumina or platinum crucibles. For high temperature or special measurements, tungsten or graphite crucibles with covers are available.



Phase transitions and melting of CuBr



The SETSYS Evolution DTA or DSC models have numerous applications: change of state (fusion, solidification), phase transitions, dehydration, decomposition, oxidation and reduction, and can be used equally well for organic and inorganic products, metals and alloys, ceramics, glass, etc...



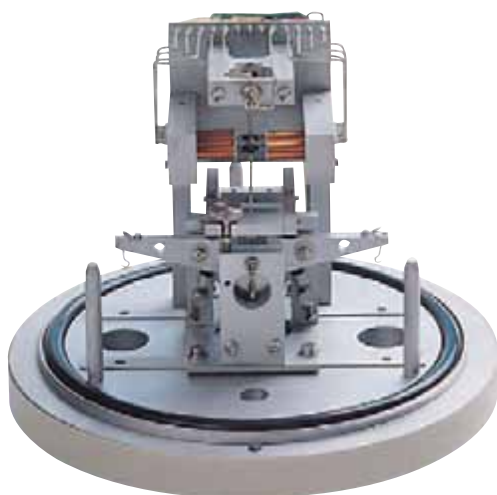
Cp measurement of iron

SETSYS Evolution TGA

Thermogravimetry from ambient to 2400°C

- **SETSYS Evolution TGA: a balance of very high stability and fidelity...**

For the SETSYS Evolution TGA, SETARAM has benefited from its extensive experience in the field of balances to design a weighing module with exceptional performance. With a maximum capacity of 35 grams the balance is well suited for the analysis samples ranging from microquantities (a few milligrams) to bulky and dense materials, while maintaining a measuring resolution equivalent to a microgram whatever the mass analyzed. Fitted with a beam articulated on a torsion ribbon, the SETSYS Evolution TGA balance displays great stability, fidelity and sensitivity due to a high-performance, optical and electronic detection fitting. The weighing module is robust and made fluid-tight for work under vacuum or with gas scavenging. Another version of balance is available offering a maximum capacity of 100 g.



- **Crucibles adapted to different applications**

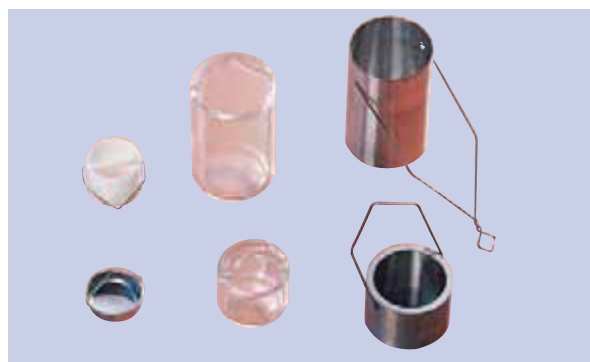
The crucible containing the sample is hung from one end of the beam, with the other end taking the rebalancing tare.

The experimental chamber delimited by the furnace (diameter: 18 mm) is adapted for the use of varied crucibles depending on the temperature range, the mass of sample to be analyzed and the type of reaction to be simulated. These crucibles are of various materials and volumes:

- silica up to 1000°C
- alumina, platinum up to 1750°C
- graphite, tungsten up to 2400°C

Latticed platinum crucibles ensure better contact when studying the interaction between the sample and a gas.

In certain cases, especially for metals, the sample can be hung directly from the balance without using a crucible.



• **A crucible-holding rod for accurate temperature measurement**

To obtain a temperature measurement nearer the sample, a sample-holding rod hung from the balance is used. The measuring thermocouple can be placed on the bottom of the crucible or within the sample (if sample-thermocouple compatibility permits).

• **Control the rate of reaction**

The various SETSYS Evolution TGA models are controlled by the computer as a function of the temperature cycle where the temperature-scanning rate is linear. For certain applications (decomposition, sintering), the rate of variation in mass must be controlled. A "controlled-rate thermogravimetry" software provides for such operations.

• **Applications based on the furnace and atmosphere**

Applications of the SETSYS Evolution TGA models are very varied: dehydration, dehydroxylation, pyrolysis, decomposition of minerals and organic matter, oxidation and combustion of organic products and fuels, degradation of polymers, characterizing of ceramics, oxidation of alloys.

Having the possibility of choosing the atmosphere and varying it during the analysis is a great advantage when studying the thermal behavior of materials in an inert or reactive atmosphere as well as for analyzing composition.

• **Devices for good sample-gas exchange**

To study the oxidation of a metal, it is possible to hang the sample directly from the balance suspension. The sample can be a plate measuring up to 13 mm x 30 mm providing an active surface of 7.8 cm² (figure ①).

When the sample is in powder form, the three-tray vessel is most useful as each tray has a surface area of 1.33 cm², therefore the powder can be spread over a total surface area of 4 cm² (figure ②).

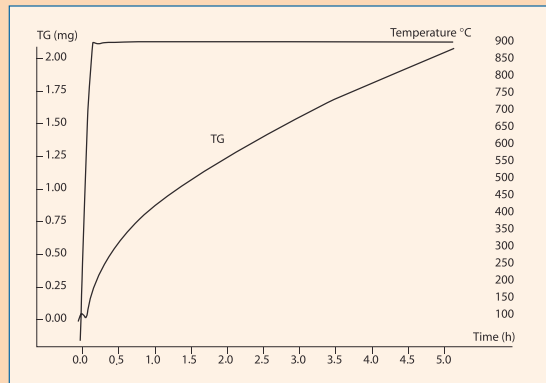
For studies at high temperatures, you can also use a **suspension rod + vessel** made entirely of sapphire. The advantage of sapphire over platinum is that it is inert to oxidation.

Measurement under controlled humidity atmosphere

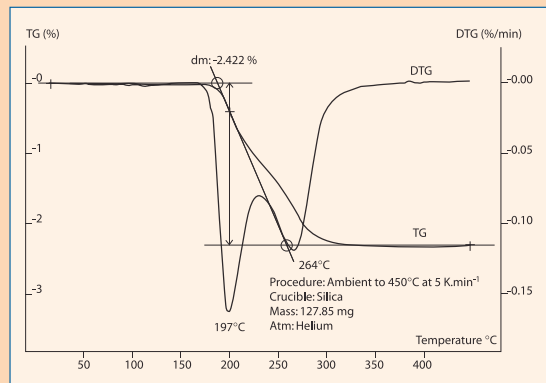
Humidity is known to considerably affect the stability of many products such as pyrotechnical materials, polymers, pharmaceutical products, foodstuffs, plasters, cements, metals and alloys, etc. To evaluate the influence of humidity on the long term stability of these products, it is necessary to specifically study their behavior under relative humidity. Thus SETARAM has developed a controlled humidity generator WETSYS*, designed to offer you the following possibilities:

- Generation of precise and controlled humidity,
- Use with SETARAM thermal analyzers including SETSYS Evolution and calorimeters, as well as with other analysis instruments.

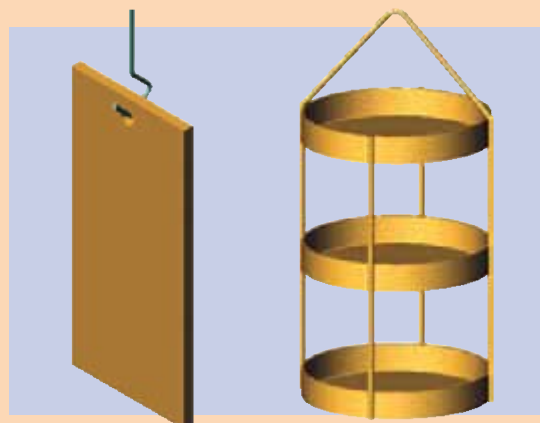
*Ask for the specific WETSYS brochure.



Oxidation of a metallic plaque



Desorption of deuterium in YFe₂D_{2,54}



① **Plate sample** ② **Three-tray vessel**



SETSYS Evolution TGA-DTA/DSC

The simultaneous TGA and DTA/DSC measurements

- **SETSYS Evolution TGA-DTA: couple DTA to your balance...**

The thermogravimetric method only indicates the variation in mass of the sample. By coupling DTA to thermogravimetric measurement, the corresponding thermal effect is combined and measured.

DTA moreover detects transformations in the sample (fusion, crystallization, transition) that are not associated with variations in mass. To optimize its range of SETSYS Evolution TGA thermobalances, SETARAM has developed a fitting for hanging the DTA transducer on to the weighing module so as to take simultaneous TGA and DTA measurements on the same sample.



With a single structure the thermobalance can thus be used in thermogravimetry alone (SETSYS Evolution TGA model) or in simultaneous TGA-DTA mode (SETSYS Evolution TGA-DTA model). Changing from one mode to the other is quick and easy.

- **SETSYS Evolution TGA-DSC : couple DSC to your balance**

In the same way, it is possible to couple a DSC rod to the balance and thus to obtain simultaneously the measurement of the TGA and the DSC signals, which will give quantitative information on the thermal exchanges.

- **SETSYS Evolution TGA-DTA 24... very high temperatures at a reasonable price**

The SETSYS Evolution TGA-DTA 24 results from developments made by SETARAM on the SETSYS series with the graphite-resistor furnace. At present, a compact, sturdy and easy-to-use table thermobalance provides for simultaneous TGA-DTA measurements in inert gas (argon) up to 2400°C.

- **Four transducers from -150°C to 2400°C**

The thermal transducers used on the SETSYS Evolution TGA

DTA models are identical to those on the SETSYS Evolution DTA models:

- the tricouple transducer
- the monocouple, protected transducer
- the DSC-type, plate transducer

These three transducers are made of platinel (1000°C), Pt/PtRh 10 % (1600°C), PtRh 6 % /PtRh 30% (1750°C)

- the very high temperature plate transducer in tungsten-rhenium for simultaneous TGA-DTA measurements up to 2400°C.



SETSYS Evolution TGA-EGA

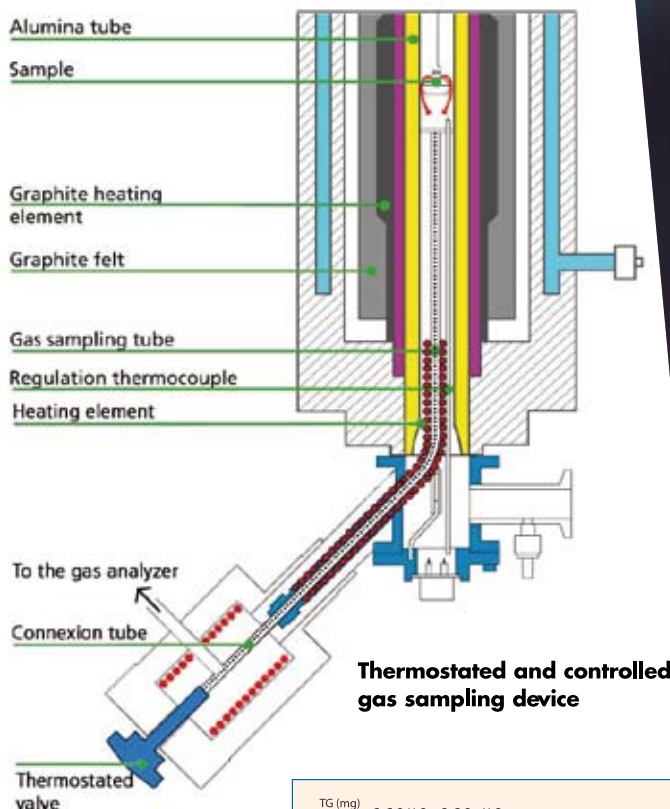
Coupling to gas analyzers

By coupling a gas analyzer to the thermoanalyzer, it is easier to identify the emitted vapors and understand the transformation mechanisms.

SETARAM Instrumentation offers you the suitable solutions:

- **For MS, FTIR, or GC coupling**

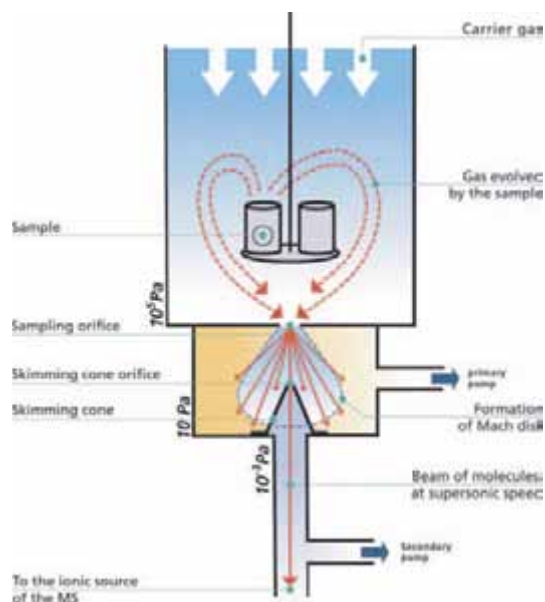
A thermostated and controlled gas sampling device using a coupling system with isolating valve has been designed. This sample system features a thermostated line up to the sample area preventing any condensation of the evolved gas. For MS coupling it allows the measurement of non-condensable products for masses up to 300 amu.



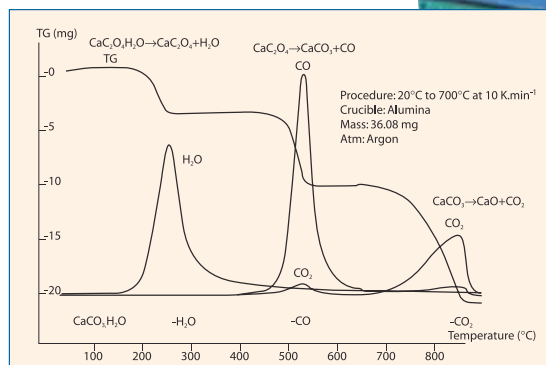
Thermostated and controlled gas sampling device

- **For high-performance TGA-MS coupling**

A unique SuperSonic System (supersonic release gas sampling system) offering unmatched measurement capabilities (remarkable resolution, high sensitivity, no condensation) even for the heaviest molecules. It allows any type of material to be studied, including inorganic material (glasses and ceramics) at high temperatures for masses up to 1024 amu.

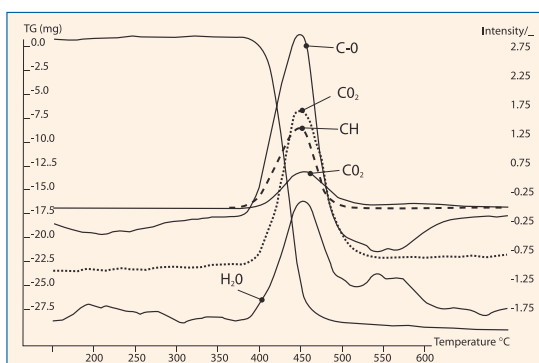


SuperSonic System



Decomposition of calcium oxalate

TGA-MS



Thermal decomposition of PET TGA-IRFT

SETSYS Evolution TMA

Dilatometric measurement from ambient to 2400°C

- **SETSYS Evolution TMA : an accurate and robust displacement transducer...**

The displacement transducer on the SETSYS Evolution TMA models is characterized by its robustness and high accuracy: it can detect dimensions changes as small as 0.01 micron.

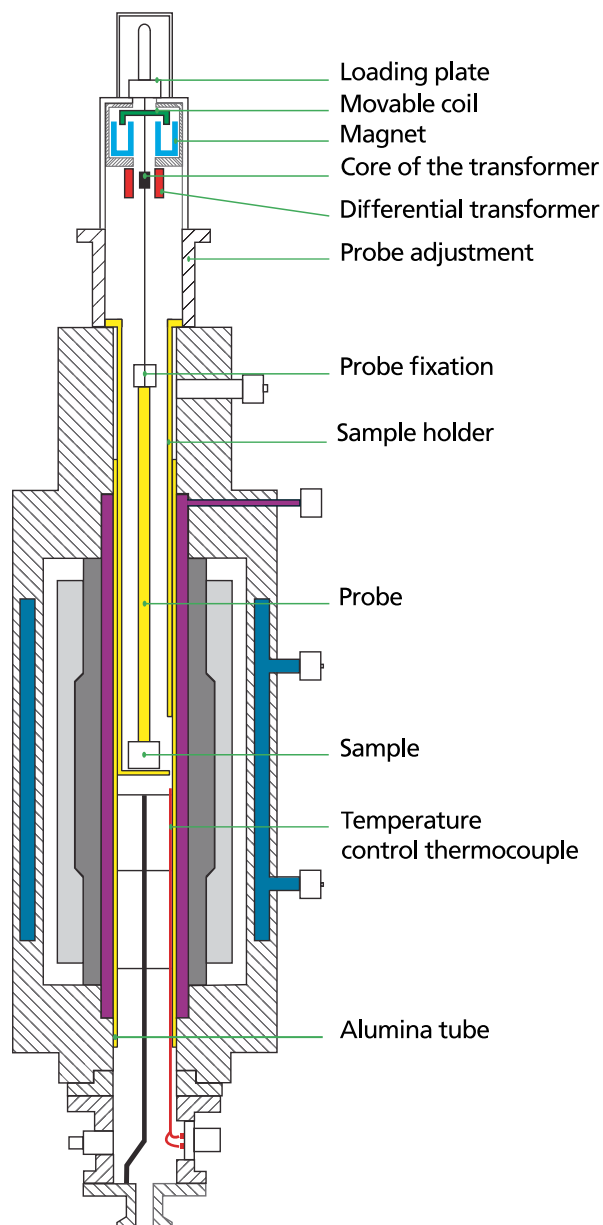
The transducer on the SETSYS Evolution TMA uses an electromagnetic system allowing automatic control of the force on the sample between 0.01 and 1.5 N. This force can be increased by adding weights (up to 200 grams) on a top plate. Transducer calibration and force control are ensured automatically by the computer.

- **Controlled-rate dilatometry and TMA**

As for the other models, the computer automatically manages operation of the SETSYS Evolution TMA, especially the force on the sample.

After acquiring the TMA signal and the temperature (measured by a thermocouple near the sample), dedicated software determines the transition temperatures, plots the derivative curve (DTMA) and corrects the base line.

Specific software provides for accurate measurement of the coefficient of expansion in the materials. A calibration curve in the form of a polynomial introduced into the computer guarantees the accuracy of the coefficient of expansion measured up to 2400°C. With such software, the SETSYS Evolution TMA can be used as a dilatometer. The SETSYS Evolution TMA is also particularly suitable for measuring controlled-rate sintering.



| | | | | |
|-------------------------------|------------|------------|------------|------------|
| SETSYS Evolution TMA | 12 | 16 | 18 | 24 |
| Temperature range (°C) | 20 to 1000 | 20 to 1200 | 20 to 1600 | 20 to 2400 |
| Type of probe | silica | alumina | alumina | graphite |

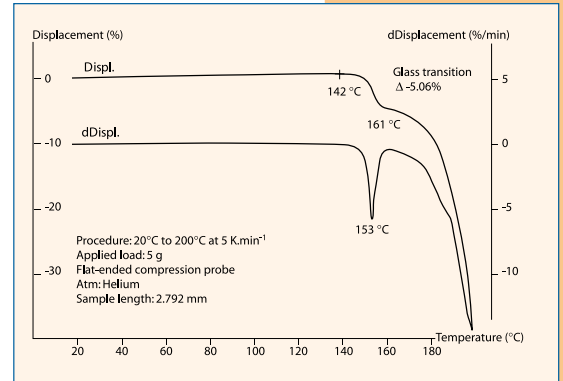
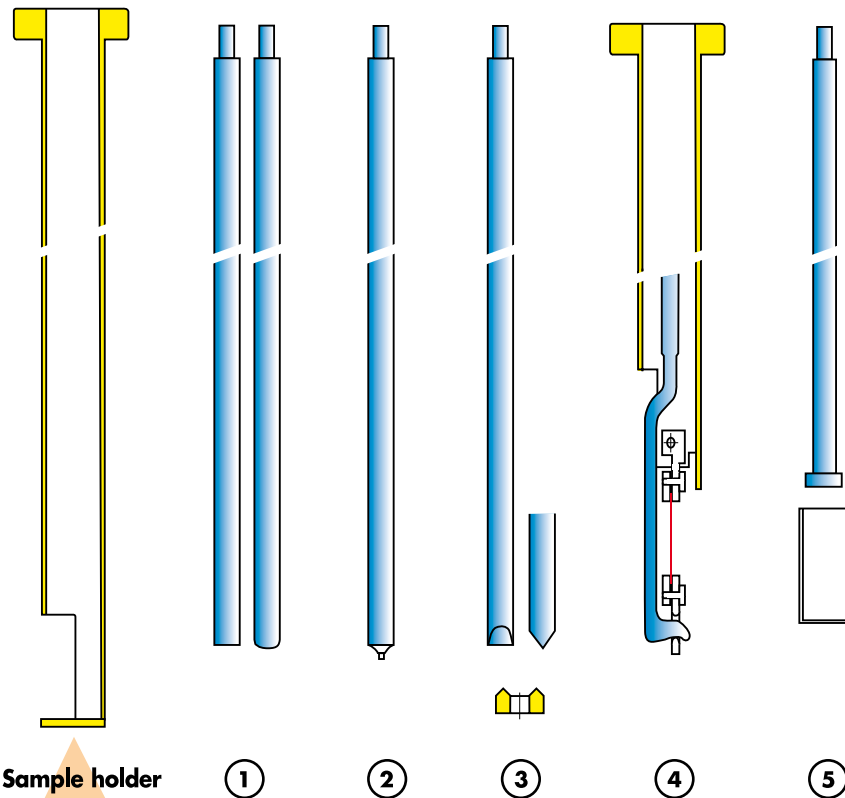
Choose your probe

Various types of probes can be fitted to the TMA transducer, depending on the application to be carried out:

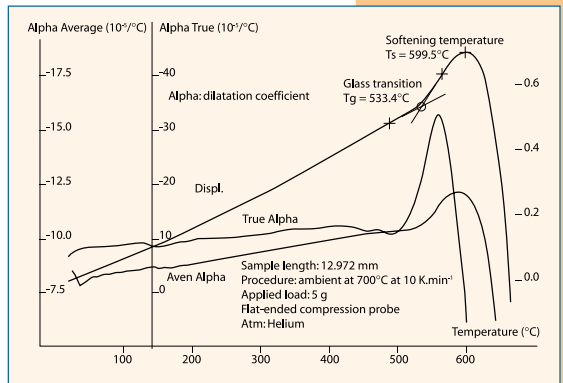
- the "compression" ① probe with a flat or spherical end for studying compression. Applied without force on the sample, it is used for measuring coefficients of expansion
- the "penetration" ② probe with a small cross-section tip. With this probe, high pressures can be produced on the sample
- the "three-point, flexure" ③ probe made up of a base with two knives and a rod with a knife-shaped cross section
- the "tensile" ④ probe comprising a lower clamp fixed to the measuring tube and an upper clamp joined to the probe, for studying fibers and films under tensile force
- the "volume-expansion" ⑤ probe comprising a crucible and a flat-bottomed probe, for studying expansion in powders.

Varied applications depending on the probes and the temperature

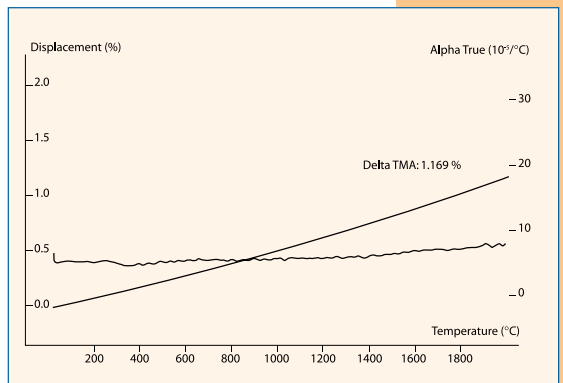
Particularly appropriate for measuring variations in size of solids or powders, the SETSYS Evolution TMA is used for characterizing polymers and compounds (softening, glass transition, degradation, reticulation) under varied stresses, for studying films and fibers, for sintering ceramics, for studying the thermal behavior of alloys and, more generally, for measuring coefficients of expansion.



Dilatometric analysis of a polycarbonate



Glass transition, softening temperature and dilatation coefficient of a glass



Dilatation of a graphite sample

SETSYS Evolution

Specifications

| Technical data | | | | | |
|---|--|---------------------------------|--|--|-----------------------|
| SetSYS Evolution | DTA | DSC | TGA | TMA | |
| | | | balance 35g balance 100g | | |
| Temperature range of the module (C°) | Amb/1000 Amb/1600 Amb/1750 Amb/2400 | Amb/800 Amb/1500 Amb/1600 | Amb/1000 Amb/1600 Amb/1750 Amb/2400 | Amb/1000 Amb/1600 Amb/1750 Amb/2400 | |
| Crucible volume (µl) | 30/100 | 80/100 | 50/3000 | / | |
| Max. size of the sample (mm) | / | / | L : 20 Ø14 | L : 20 Ø :10 | |
| Resolution | / | 0.4 µW | 0.03 µg | 0.3 µg | 0.2 nm |
| Noise RMS | / | 16 µW | 0.03 µg | 0.3 µg | 5 nm |
| Specific noise RMS | / | 0.16 µW/µl | 0.02 µg/ml | 0.2 µg/ml | 0.25.10 ⁻⁶ |
| Measuring range | / | / | ± 200 mg | ± 2 g | ± 2 mm |

The SETSYS Evolution line is equipped with SETSOFT 2000, the thermal analysis software from SETARAM.

Option:

AKTS thermokinetics software to get the most from the thermal analyzer.

