

SETSYS Evolution

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

The top of the range in
thermal analyzers



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SETSYS Evolution

The new benchmark!

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

In order to develop evermore possibilities of analysis, measurement safety and ease of use from its devices, SETARAM has launched the SETSYS Evolution line. Its analyzers provide all the strong points of SETSYS Evolution analyzers:

- **The widest temperature range**

With a range of measurement from -150 to 2400°C , SETSYS Evolution offers the widest temperature range on the market.

- **Modularity**

The different measurement modules (DTA, DSC, TGA, TMA adapt and can be interchanged) are built 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.

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

A digital display on the front casing 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.

Thus SETSYS Evolution continues to represent the top of the range in 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 for 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: the DSC permits determining the temperature of this transformation and quantifying the heat.

TGA :

Thermogravimetry is a technique that measures the variation of mass of a sample when the latter 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 :

Thermomechanic 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, traction and flexion.

SETSYS Evolution

A common structure

-150°C  +2400°C

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.

• Five versions of temperature

Five different versions of structure are available according to the nature of the furnace and temperature regulation.

	SETSYS Evolution Cryo	SETSYS Evolution 12	SETSYS Evolution 16	SETSYS Evolution 18	SETSYS Evolution 24
Temperature range (°C)	- 150 to 400	Ambient to 1200	Ambient to 1600	Ambient to 1750	Ambient to 2400
Furnace	Cryostat	Metal	Graphite	Graphite	Graphite
Thermocouple régulation température	Type P platinel	Type S Pt/Pt-Rh 10%	Type S Pt/Pt-Rh 10%	Type B Pt-Rh 6%/Pt-Rh 30%	Type W5 WRe 5%/WRe 26%

• Varied and controlled atmospheres

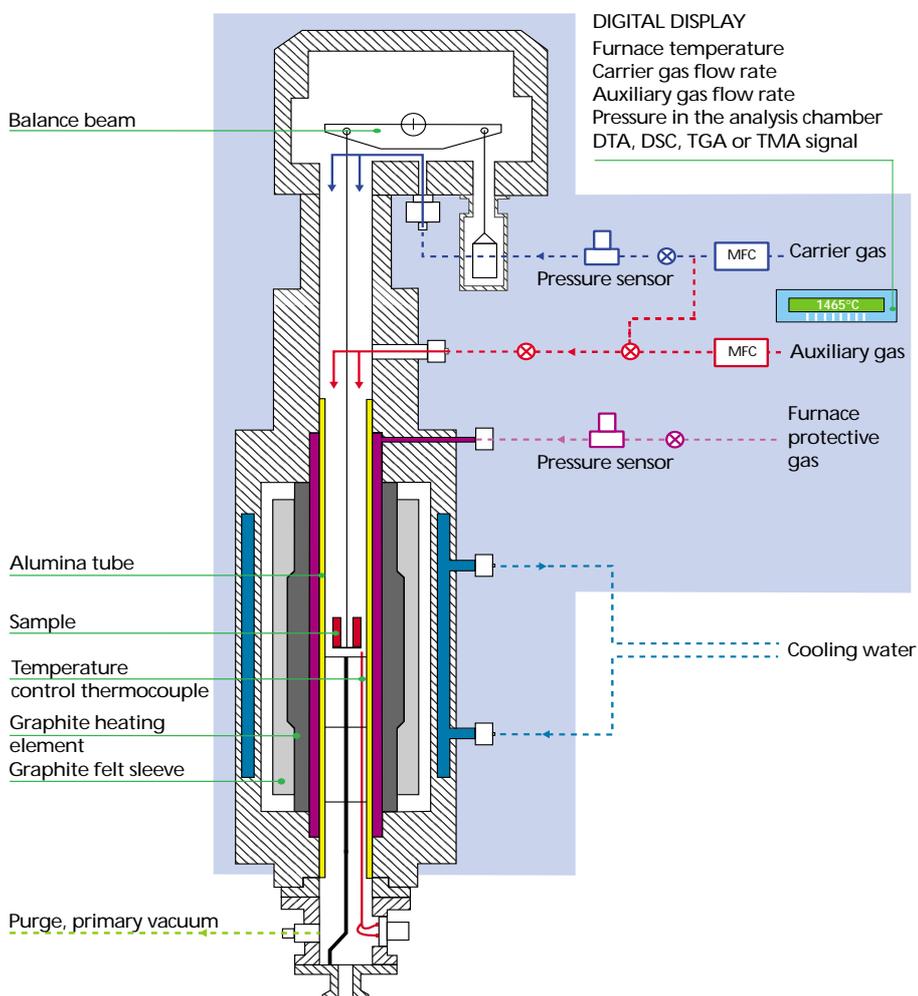
The basic version of the SETSYS Evolution is equipped with a carrier gas circuit whose flow can be adjusted and controlled by an MFC* (range from 0.24 to 12 l/h, precision $\pm 0.2\%$ full scale and $\pm 0.8\%$ of the measurement). A pressure transducer (10 mbar / 1.6 bar) permits measuring the pressure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

A new "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 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 the cooling water and protection gas is automatic.



SETSYS Evolution DTA

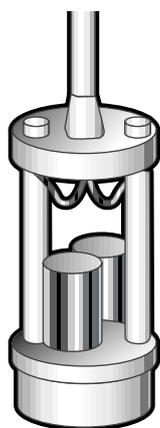
Measurement up to 2400°C

-150°C  +2400°C

- **SETSYS Evolution DTA: five models from - 150°C to 2400°C...**

Five different differential thermal analyzers models have been developed, under the brand-name SETSYS Evolution DTA, covering a wide temperature range from - 150 °C to 2400°C.

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



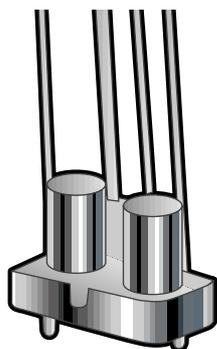
- **A high performance tricouple transducer**

The tricouple transducer, due to its high qualitative and quantitative performances, is the most commonly used on the models in the SETSYS Evolution DTA range. The excellent symmetry in the detectors, each made up of three thermocouples, guarantees very good baseline stability and a very high detection limit for thermal measurements.

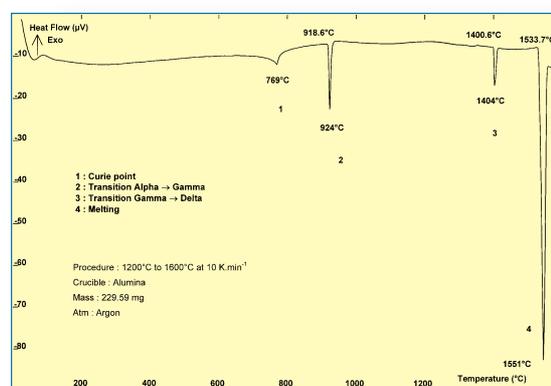
The alumina crown enclosing the detector ensures that the crucible's positioning is accurate and reproducible.

SETSYS Evolution Instrument	temperature range of the furnace (°C)
DTA cryo	-150 to 400
DTA 12	20 to 1200
DTA 16	20 to 1600
DTA 18	20 to 1750
DTA 24	20 to 2400
DSC cryo	-150 to 400
DSC 15	20 to 1600
DSC 16	20 to 1750

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

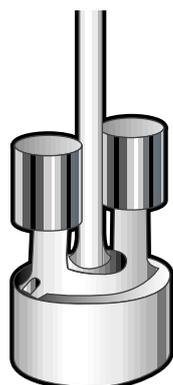


High-temperature, thermal measurements require the use of tungsten-rhenium thermocouples. To produce such a transducer with the SETSYS Evolution DTA 24 analyzer a tungsten bed-plate is machined with two housings for the crucibles. Measurements for the thermal signal and the temperature are provided by the tungsten-rhenium thermocouples which ensure rigidity in the DTA rod. The detector is essentially dedicated to very-high-temperature measurements.

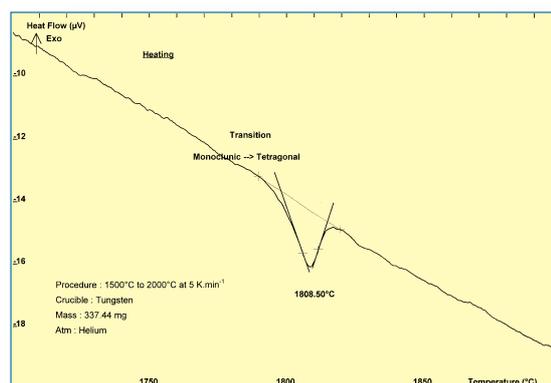


Phase transitions and melting of iron

- **A protected transducer**



In the protected DTA rod the measurement and reference thermocouples are covered by the crucibles. They are more protected in case aggressive gas emanates from the sample.



Phase transitions of HfO₂

SETSYS Evolution DSC

Measurement up to 1600°C

-50°C  +1600°C

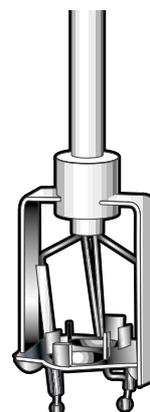
- **SETSYS Evolution DSC: three models from - 50°C up to 1600°C**

Three temperature versions of SETSYS Evolution DSC are available. Depending on the requested temperature range the nature of the constituting elements of the DSC plate rod changes.

type of thermocouples	maximum use range of the rods (°C)
P	-150 to 1000
P	20 to 1000
S	20 to 1600
B	20 to 1750
W5	20 to 2400
E	-50 to 800
S	20 to 1500
B	20 to 1600

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

The DSC plate rod is composed of a machined metallic plate with two housing for the measurement and the reference crucibles. Flat bottom crucibles are used to optimize the thermal contact. Small pins provide a good positioning of the crucible on the transducer.



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

Up to 1750°C, platinum, alumina and zirconia crucibles are offered for tri-couple 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.

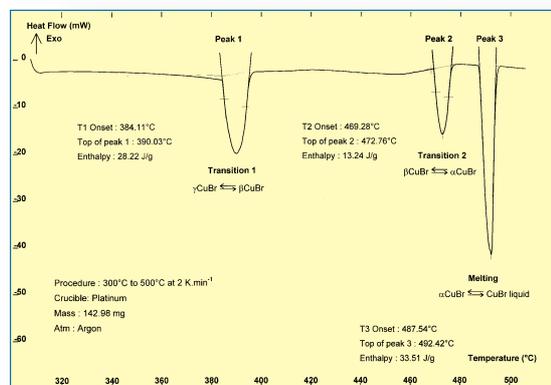
For limited-temperature use (up to 500°C) aluminium crucibles, fitted with lids, are appropriate for studying controlled dehydration.

The DSC plate rod requires the use of alumina or platinum crucibles with a flat bottom.

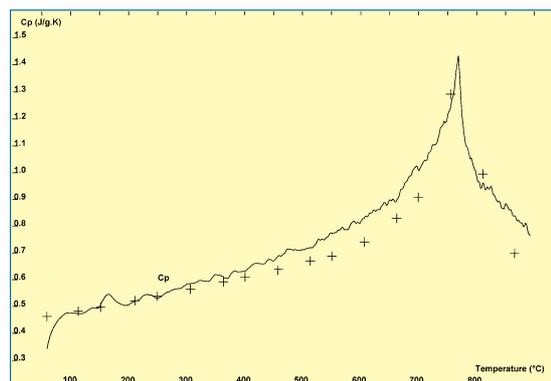
For high temperature or special measurements tungsten or graphite crucibles with covers are offered.



Applications for the SETSYS Evolution DTA or DSC models are very numerous : change of state (fusion, solidification), phase transitions, dehydration, decomposition, oxidation and reduction are equal interest for organic and inorganic products, metals and alloys, types of ceramics, glass...



Phase transitions and melting of CuBr



Cp measurement of iron

SETSYS Evolution TGA

The thermogravimetry from -150 up to 2400°C

-150°C +2400°C

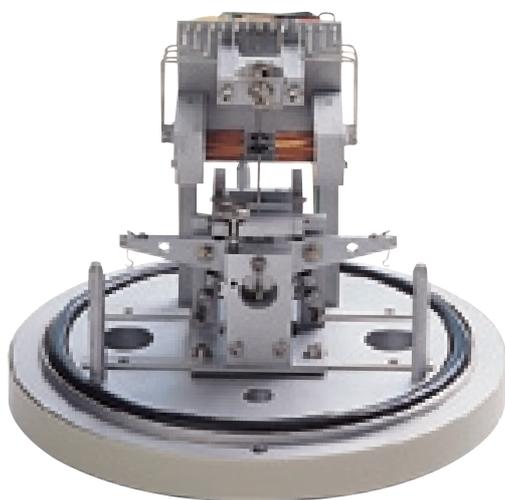
- **SETSYS Evolution TGA : a balance of very high stability and fidelity...**

For the SETSYS Evolution TGA, SETARAM has benefitted from its long experience in the field of balances to design a weighing module with exceptional performances.

With a maximum load of 35 grammes the balance is well adapted for the analysis of microquantities of sample (a few milligrammes) up to bulky and dense samples, while maintaining a measuring resolution equivalent to a microgramme whatever the mass analyzed. Fitted with a beam articulated on a torsion ribbon, the SETSYS Evolution TGA balance possesses at the same time 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 in a vacuum or under gas sweeping.

Another version of balance is available offering a maximum capacity of 100 g.



For studying the interaction between the sample and a gas latticed platinum crucibles ensure better contact. In certain cases, especially for metals, the sample can be hung directly from the balance without any crucible.

- **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 set 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



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

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

- **Control the rate of reaction**

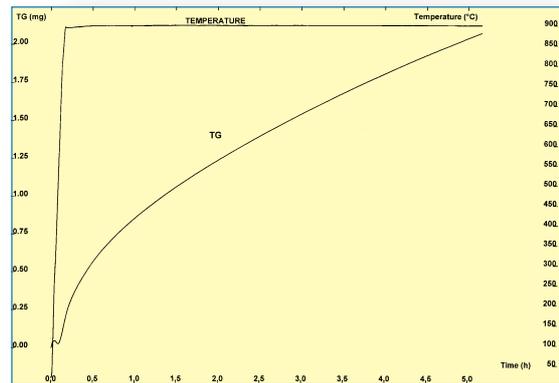
The various SETSYS Evolution TGA models are controlled by the computer as a function of the temperature cycle where the temperature-scanning rate is linear.

For certain applications (decomposition, sintering), the rate of variation in mass must be controlled. A "controlled-rate thermogravimetry" software provides for such handling operations.

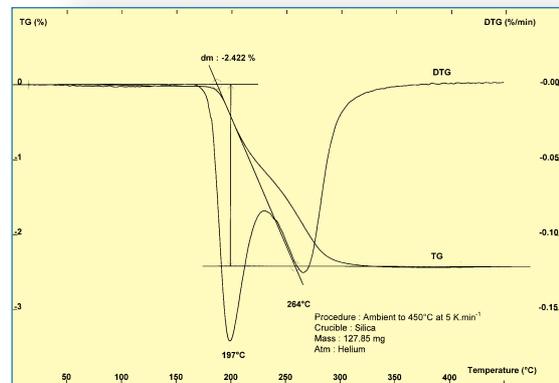
- **Applications based on the furnace and atmosphere**

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.

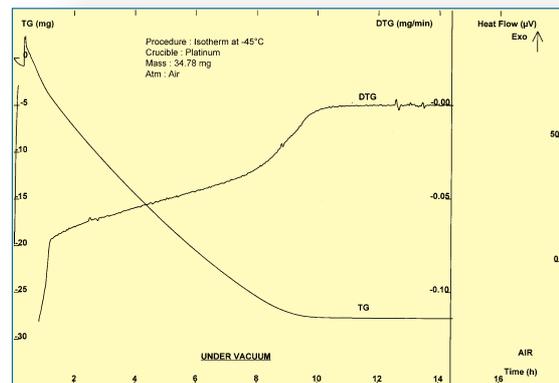
The choice of atmosphere and the facility for varying them during the analysis are very attractive for studying the thermal behaviour of materials in an inert or reactive atmosphere as well as for analyzing composition.



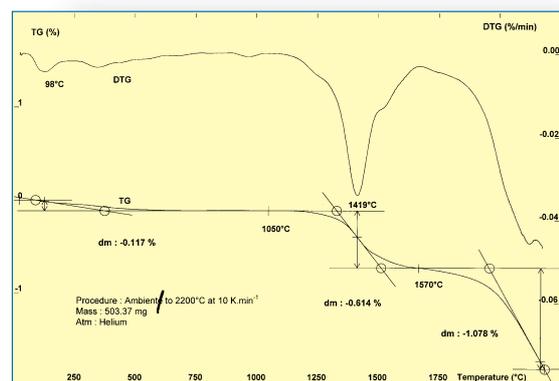
Oxidation of a metallic plaque



Desorption of deuterium in $YFe_2D_{2,54}$



Freeze drying of a wood sample



Heating of SiC

SETSYS Evolution TGA-DTA/DSC

The simultaneous TGA and DTA/DSC measurements

-150°C  +2400°C

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

The thermogravimetric method only supplies variation in mass on the sample. By coupling DTA to thermogravimetric measurement the corresponding thermal effect is combined and measured. On the other hand DTA detects transformations in the sample (fusion, crystallization, transition) not combined with variations in mass.

To optimise 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 produce simultaneous TGA and DTA 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). Passing from one mode to the other is rapid 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 is issued from developments produced by SETARAM on the SETSYS series with the graphite-resistor furnace.

At present, a table thermobalance provides for simultaneous TGA-DTA measurements in inert gas (argon) up to 2400°C without the need for excessive care.

• 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/Pt-Rh 10 % (1600°C), Pt-Rh 6 % /Pt-Rh 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

The couplig to gas analyzers

With the coupling of a gas analyzer to the thermoanalyzer, it is easier to identify the emitted vapours and to understand the mechanisms in transformation.

There are essentially two types of coupling for gas analyzers to TGA-DTA thermoanalyzer :

- mass-spectrometry coupling
- fourier-transform, infra-red coupling.

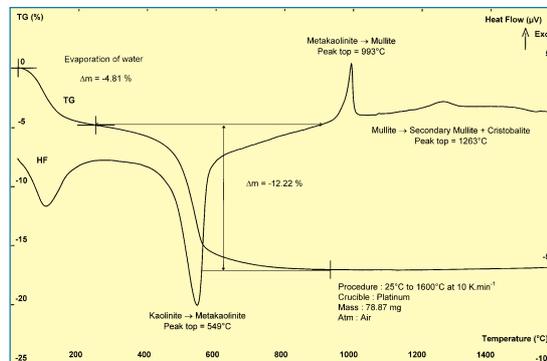
• Mass spectrometry coupling (MS)

SETARAM offers two types of TGA-MS coupling systems:

- by SuperSonic System (supersonic release gas sampling system). This system offers unmatched measurement capabilities: remarkable resolution and high sensitivity, even for the heaviest molecules, and no condensation phenomenon. It permits studying any type of material, including inorganic materials (glasses and ceramics) at high temperatures for masses up to 1024 amu.
- By optimized sampling system and by heated capillary for non-condensable products for masses up to 300 amu.

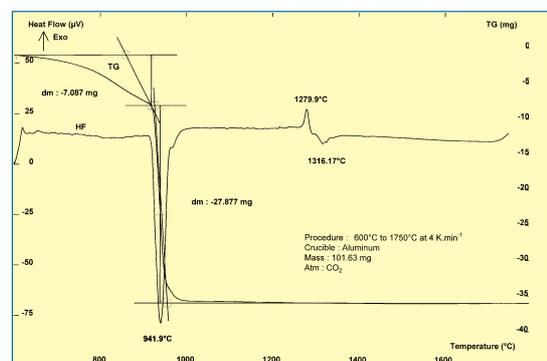
• Fourier-transform, infra-red coupling (FTIR)

The TGA-FTIR coupling system uses an interface composed of a heated transfer line, an isolating valve and a transmission cell adapted to gases. The FTIR is particularly well suited to studying organic products.



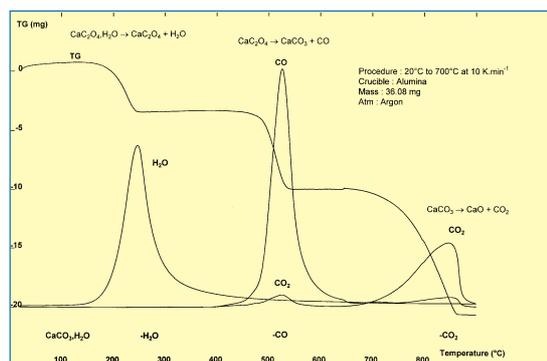
Analysis of kaolinite

TGA-DTA



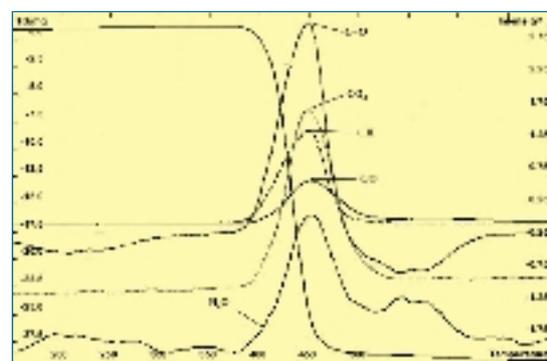
Thermal behaviour of a raw cement

TGA-DTA



Decomposition of calcium oxalate

TGA-MS



Thermal decomposition of PET

TGA-IRTF

SETSYS Evolution TMA

The dilatometric measurement from -150 up to 2400°C



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

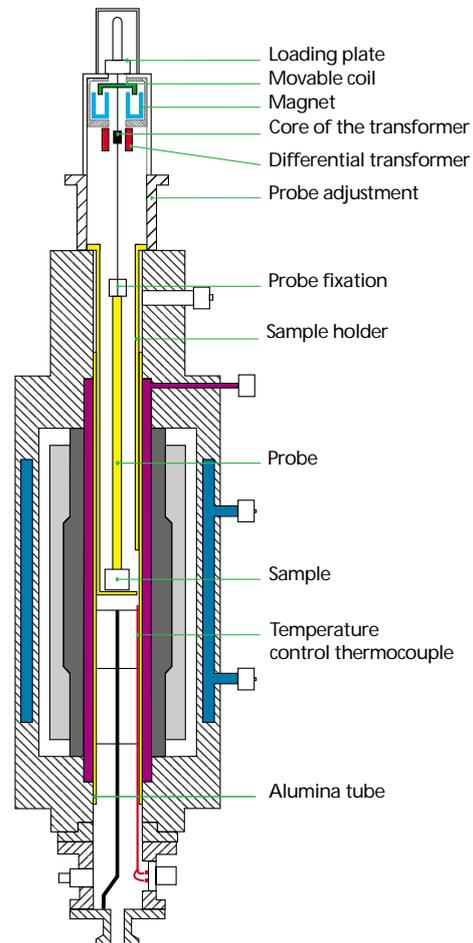
The displacement transducer on the SETSYS Evolution TMA model is especially distinguished by its robustness and its great accuracy variations in movement as small as 0.01 micron can be detected with such a transducer.

Developed by SETARAM, the transducer on the SETSYS Evolution TMA uses an electromagnetic fitting allowing automatic piloting of the force on the sample up to 1.5 N. This force can be increased by adding weights (up to 200 grammes) on a top plate. Calibrating the transducer and controlling the force are handled automatically by the computer.

- **Controlled-rate dilatometry and TMA**

As for the other models the computer automatically operates 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) adapted 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 especially appropriate for measuring controlled-rate sintering.



SETSYS Evolution TMA	cryo	12	16	18	24	
Temperature range (°C)	-150 to 400	20 to 1000	20 to 1200	20 to 1600	20 to 1750	20 to 2400
Type of probe	silica		alumina		graphite	

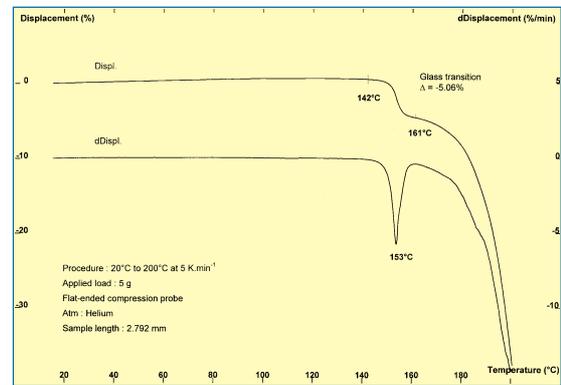
• Choose your probe

Based on the application to be carried out various types of probes can be fitted to the TMA transducer :

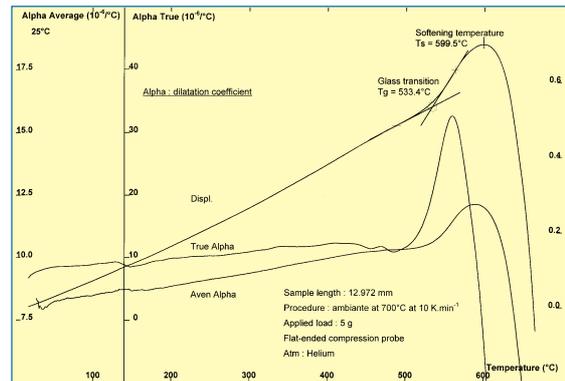
- the "compression" ① probe with a flat or spherical end for studying compression. Used without force on the sample, it is part of measuring coefficients of expansion
- the "penetration" ② probe with a fine-cross end section. With this probe high pressures can be produced on the sample
- the "three-point,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

• Varied applications depending on the probes and the temperature

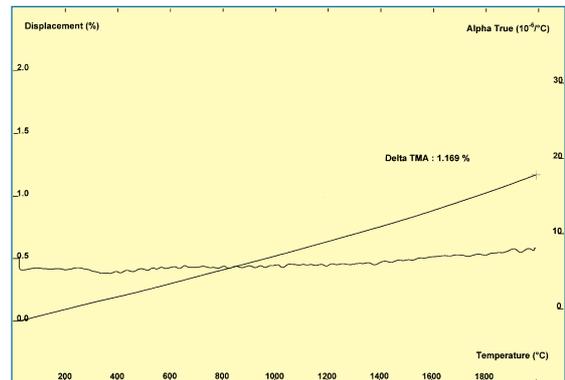
Especially appropriate for measuring the variations in size of solids or powders, the SETSYS Evolution TMA is used for characterizing polymers and compounds (softening, glass transition, degradation, reticulation) under varied stresses, for studying films and fibres, for sintering ceramics, for studying the thermal behaviour of alloys and, in a general way, for measuring coefficients of expansion.



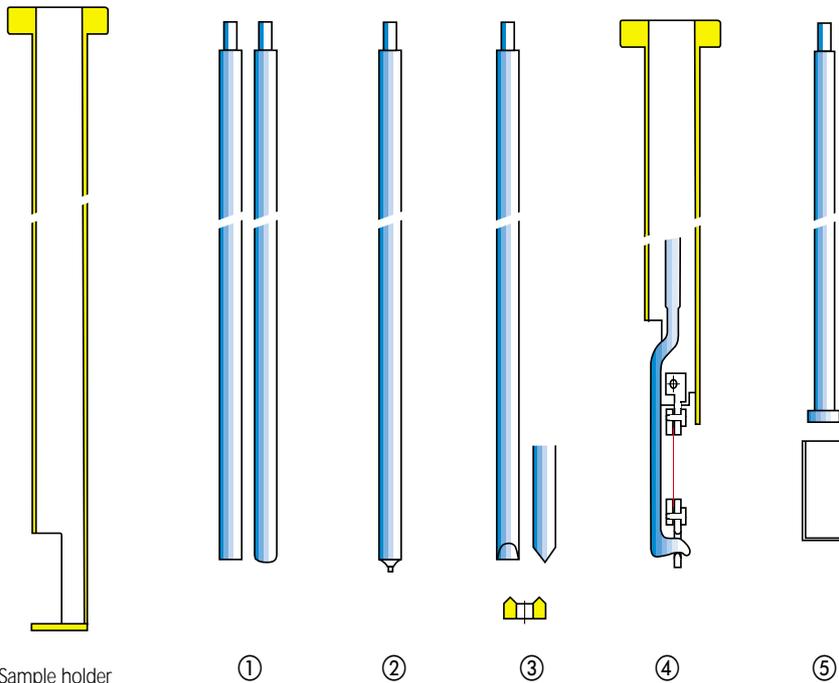
Dilatometric analysis of a polycarbonate



Glass transition, softening temperature and dilatation coefficient of a glass



Dilatation of a graphite sample



SETSYS Evolution

Some technical features

Setsys Evolution	DTA	DSC	TGA		TMA
			balance 35g	balance 100g	
Temperature range of the module (C°)	-150/400	-50/400	-150/400		-150/400
	Amb/1000	Amb/800	Amb/1000		Amb/1000
	Amb/1600	Amb/1500	Amb/1600		Amb/1600
	Amb/1750	Amb/1600	Amb/1750		Amb/1750
	Amb/2400		Amb/2400		Amb/2400
Crucible volume (µl)	30/100	80/100	50/1500		/
Max. size of the sample (mm)	/	/	/		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.



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Excellence in thermal analysis and calorimetry.

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