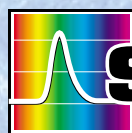
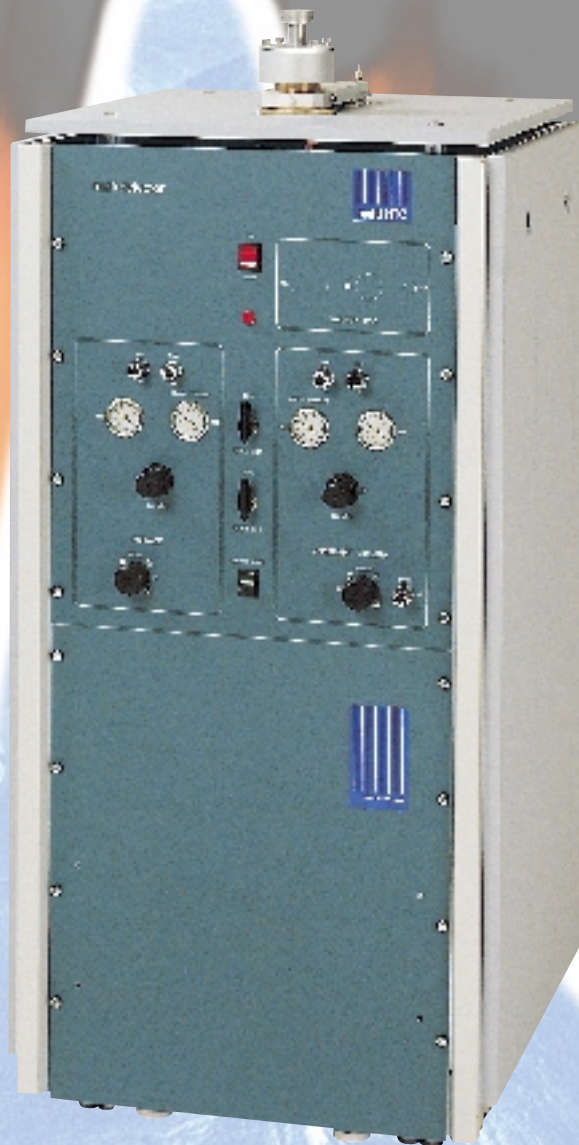


96 Line

A modular line of high temperature -
high volume thermal analyzers



SETARAM

KEP
TECHNOLOGIES

Scientific & Industrial Equipment

96 Line

A modular line of high temperature - high volume thermal analyzers

96 Line thermal analyzers can be distinguished by the following characteristics:

• Multi-modularity

All the analyzers of 96 Line, DTA, DSC, MHTC (calorimetry and DSC), TGA and TMA, are designed to adapt to the same structure (furnace, electronics, gas circuits, atmosphere control). This configuration enables economic testing of samples with different thermal analysis techniques.

• High temperature

The furnace is based on graphite resistor technology, which provides safety during operations at high temperatures. It covers a range from ambient temperature to 2,100°C, with regulation adapted to different temperature ranges. The furnace has a large zone of homogeneous temperature.

Temperature range (°C)	Amb/1600	Amb/1750	Amb/2100
Heating element	Graphite	Graphite	Graphite
Temperature control thermocouple	Type S	Type B	Type W5

• Varied and controlled atmospheres

The atmosphere control device is designed to permit scanning different gases (inert and reactive) during analysis with precision control of their flows in particular so as not to disturb thermogravimetric measurements. The chamber is built to withstand high vacuums before and during analysis. The different operations of introducing gases, creating a vacuum and switching gases during analysis can be fully automated and controlled from a computer.

• Large sample volume

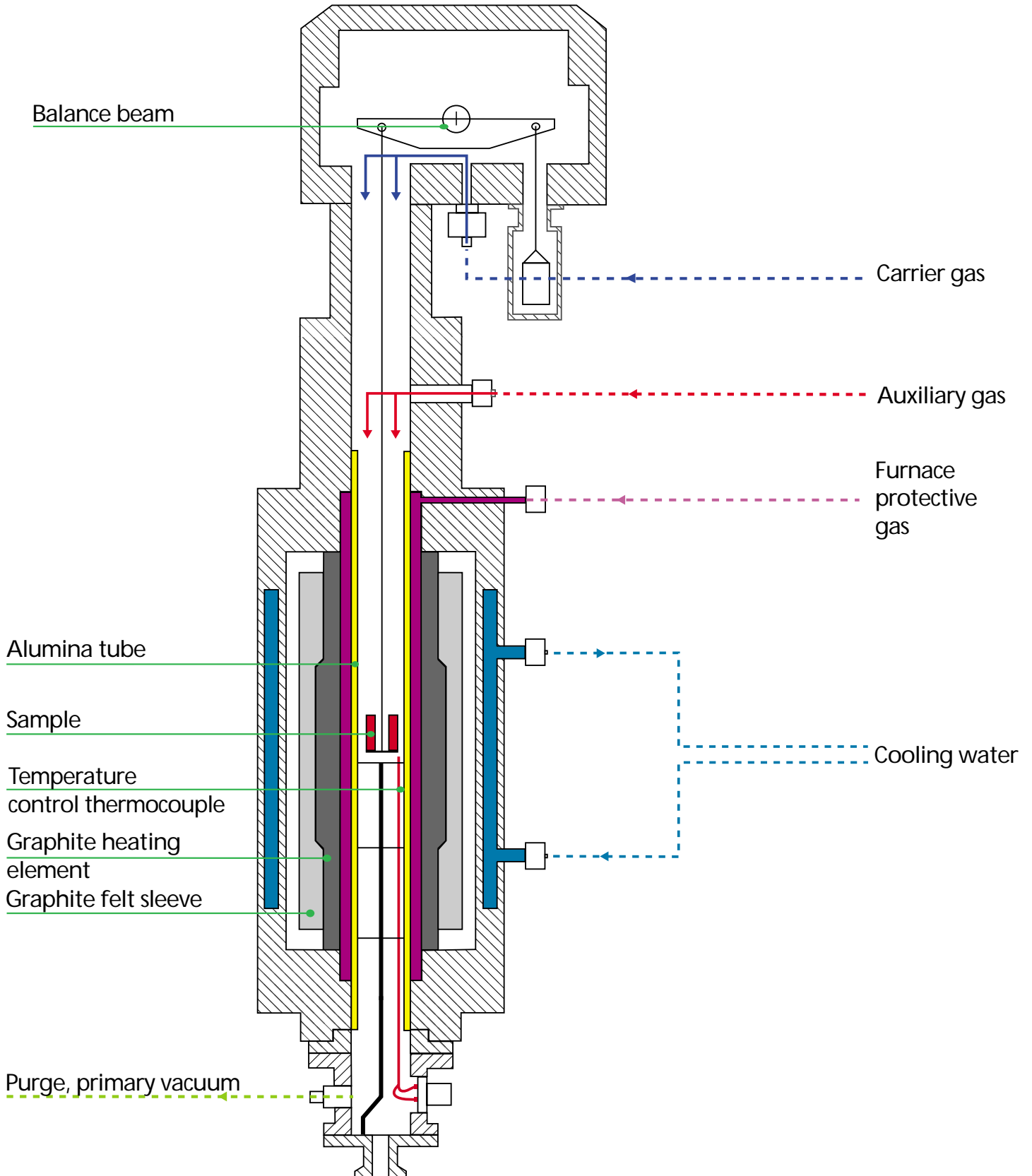
Thanks to the furnace's large diameter, the sensors developed for DTA, DSC, MHTC, TGA and TMA measurements can accommodate large samples. For all thermal analysis techniques, the fact of being able to use a large sample volume has many advantages and provides possibilities such as:

- Analysing bulky, massive and non-dissociable samples (e.g. corrosion studies of mechanical parts, etc.)

Module used	Max. sample volume		
	V (µl)	Ø (mm)	H (mm)
DTA	500	6,5	16
DSC	420	7	11
MHTC hf-dsc	450	6	16
MHTC drop	5700	14,5	35
TGA	18100	21,5	50
TMA		18 (15 recommended)	50

- Working on mixed materials (concrete core sample, etc.)
- Detecting very weak variations of mass in TGA (corrosion studies on sheet steel, etc.)

Principle diagram (TGA 96 model)



MHTC 96

For calorimetric measurements and DSC

Experimental constraints from one experiment sometimes contradict each other, i.e. low sample mass for good transition detection, high sample mass for accurate determination of heat capacity, analysis at constant and variable temperatures, use of the crucible open or closed. Two types of sensor have been developed to meet these different needs.

MHTC 96 hf-dsc

Two ceramic chambers (one measurement and one reference), arranged vertically, side by side, are linked together by two conducting plates with 20 thermocouples. The differential detector thus formed completely envelopes the experiment chamber (the sensor uses the "quasi-Calvet" principle), thereby providing global and precise measurements.

Two sensors are available according to the temperature range used:

- MHTC 96 hf-dsc ambient / 1400°C: S type thermocouple
- MHTC 96 hf-dsc ambient / 1600°C: B type thermocouple

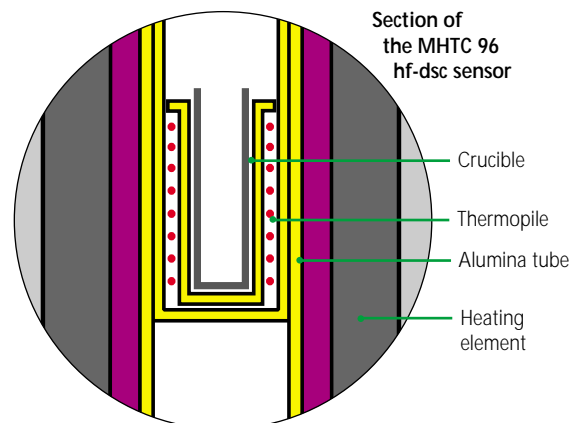
Applications

- Study of structural transformations in metals and alloys, minerals, oxides;
- Formulation of equilibrium diagrams,
- Study of material reactivity at high temperatures,
- Determination of heat capacity C_p according to the continuous or incremental method.



MHTC 96 hf-dsc sensor

MHTC 96	Type of crucible	Max. sample size		
		V(μl)	∅ (mm)	H (mm)
hf-dsc	Alumina	450	6	16
Amb / 1400° C	PtRh10%	450	6	16
Amb / 1600° C	(+alumina lining)	300	5	15,5
drop Amb / 1300° C	PtRh10%	5000	13,5	35
Amb / 1500° C	Alumina	5700	14,5	35



Section of the MHTC 96 hf-dsc sensor

MHTC 96 drop

The calorimetric sensor is symmetrical and designed to contain 2 crucibles arranged one below the other. The lower crucible is used for the reference and holds an inert material to offset the heat capacity of the sample contained in the upper crucible.

A thermopile composed of a chain of 28+28 thermocouples connected in series covers the surface of the measurement and reference crucibles.

Two sensors are available depending on the temperature range used:

- MHTC 96 drop ambient / 1300°C: S type thermocouple
- MHTC 96 drop ambient / 1500°C: B type thermocouple

Applications:

- In isotherm mode, it is especially used for the drop calorimetric method to determine the specific heat of very different materials, the heat of solution of metals in metal baths (alloy formation), oxides in oxide baths (complex oxide formation), calculation of thermal balances, etc.
- In programmed mode, it is DSC mode for large samples. The applications are analogous with those of the DSC sensor.



MHTC 96 drop sensor

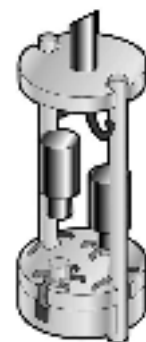
DTA 96, DSC 96

Tri-couple DTA sensors

The excellent symmetry of the detectors, each composed of three thermocouples, guarantees very good base line stability and a very high detection threshold for thermal measurement. The alumina crown surrounding the detector guarantees accurate and reproducible positioning of the crucible.

Two types of DTA rod are available for the 96 Line:

- Ambient to 1600°C: S type thermocouple
- Ambient to 1750°C: B type thermocouple



DTA rod

High temperature DTA sensor

High temperature measurements require the use of tungsten-rhenium thermocouples. This high temperature DTA is composed of a tungsten base plate with two machined housings for the measurement and reference crucibles and tungsten-rhenium thermocouples that also ensure good rod rigidity.

- Ambient to 2100°C: W5 type thermocouple



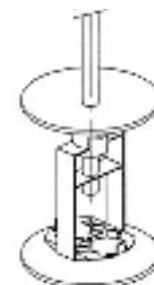
High temperature DTA rod

DSC sensor

The characteristics of plate type DSC sensors are good quantitative measurement of thermal effects and low variation of sensitivity vis-à-vis the measurement temperature.

Composed of a metal support with two housings for the crucibles, the sensor provides good thermal contact between the crucible and the detector. Two plate DSC rods are available for 96 Line:

- Ambient to 1500°C: S type thermocouple
- Ambient to 1600°C: B type thermocouple



DSC rod

Applications :

- Detection of phase transformations, heat capacity measurement
- Study of small samples
- Utilization of faster heating rate (up to 20°C.min⁻¹)

96 Line sensor	Type of crucible	Max. sample volume		
		V (µl)	∅ (mm)	H (mm)
DTA Amb / 1600° C Amb / 1750° C Amb / 2100° C	PtRh 10 %	500	6,5	16
	Alumina	250	3,8	20
	Carbon, tungsten	220	5,5	9,5
DSC plate Amb / 1500° C Amb / 1600° C	PtRh 10 %	420	7	11
	Alumina	360	6,5	11

TGA 96

This is an extremely high performance weighing module used with the TGA 96. This balance can weigh samples up to 100 g and are well adapted to measuring both micro-quantities of samples (a few milligrams) and large and dense samples, while maintaining measurement resolution to within a microgram whatever the mass analyzed.

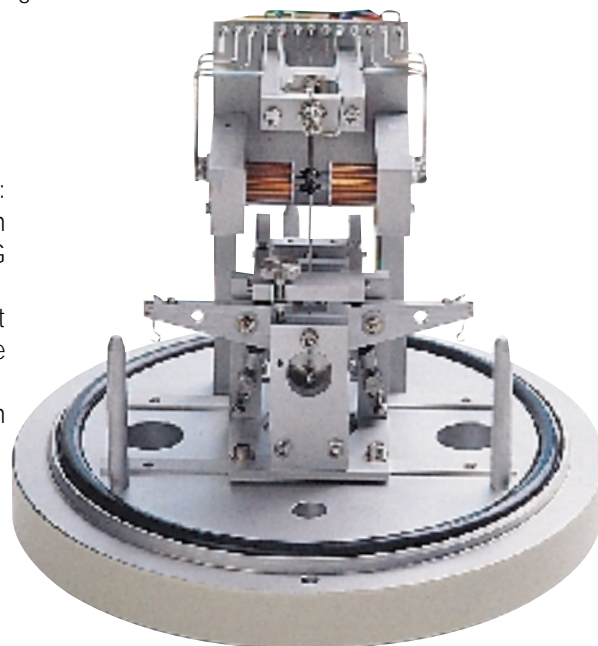
Three temperature versions of the TGA 96 are available according to the regulation used:

- Ambient to 1600°C: S type regulation thermocouple
- Ambient to 1750°C: B type regulation thermocouple
- Ambient to 2100°C: W5 type regulation thermocouple

Crucibles adapted to different applications

Several configurations are possible to analyze samples with the TGA 96:

- The sample is placed in a crucible suspended from the balance by an adapted suspension system. Several types of crucible are available for TG measurements.
- The sample can also be suspended directly from the balance without being placed in a crucible beforehand. Maximum dimensions of the crucible are length: 80mm, height: 20 mm.
- The sample can be placed in a TGA rod with a built-in thermocouple in order to measure its temperature.



	Type of crucible	Max. sample volume		
		V(μl)	∅ (mm)	H (mm)
TGA 96 Amb / 1600° C	Without crucible		20	80
	PtRh10%	18100	21,5	50
Amb / 1750° C	PtRh10% grid	15700	20	50
Amb / 2100° C	Alumina	17300	21	50
	Graphite	14900	19,5	50
	Alumina crucible with TGA rod	2000	9,8	26

Simultaneous TGA-DTA/DSC measurements

The DTA and DSC rods presented on the previous page can be suspended from the balance. Thus, TG signal measurement (gain or loss of sample mass) is obtained at the same time as the DTA or DSC signal measurement (according to the rod used), providing qualitative and quantitative data on the thermal exchange of the sample.

Simultaneous TGA-DTA

- Ambient to 1600°C
- Ambient to 1750°C
- Ambient to 2100°C

Simultaneous TGA-DSC

- Ambient to 1500°C
- Ambient to 1600°C

DTA and DSC rods designed for measuring small sample quantities (typically 100 μl) can also be used with the TGA 96.

A wide range of measurement possibilities:

In addition to the TGA 96, SETARAM proposes different accessories and software that permit in particular:

- TGA measurements in corrosive atmospheres,
- Controlled rate TGA measurement (CRTA: controlled rate thermal analysis)
- Coupling with a gas analyzer either FTIR or mass spectrometry type. A special coupling part is installed at the furnace outlet and serves as interface between the TGA 96 and the gas analyzer.

TMA 96

Vertical dilatometry

The TMA 96 has a vertical configuration that permits working under very low loads (from 2 g) without subjecting the sample to effort. The displacement sensor of the TMA 96 is both robust and highly accurate. It detects variations of displacement as low as 0.01 micron. The sensor of the TMA 96 uses an electromagnetic system allowing automatic control of force on the sample up to 1.5 N. This force can be increased by adding weight (up to 200 grams) on the upper plate. The computer manages calibration adjustment and force control automatically.

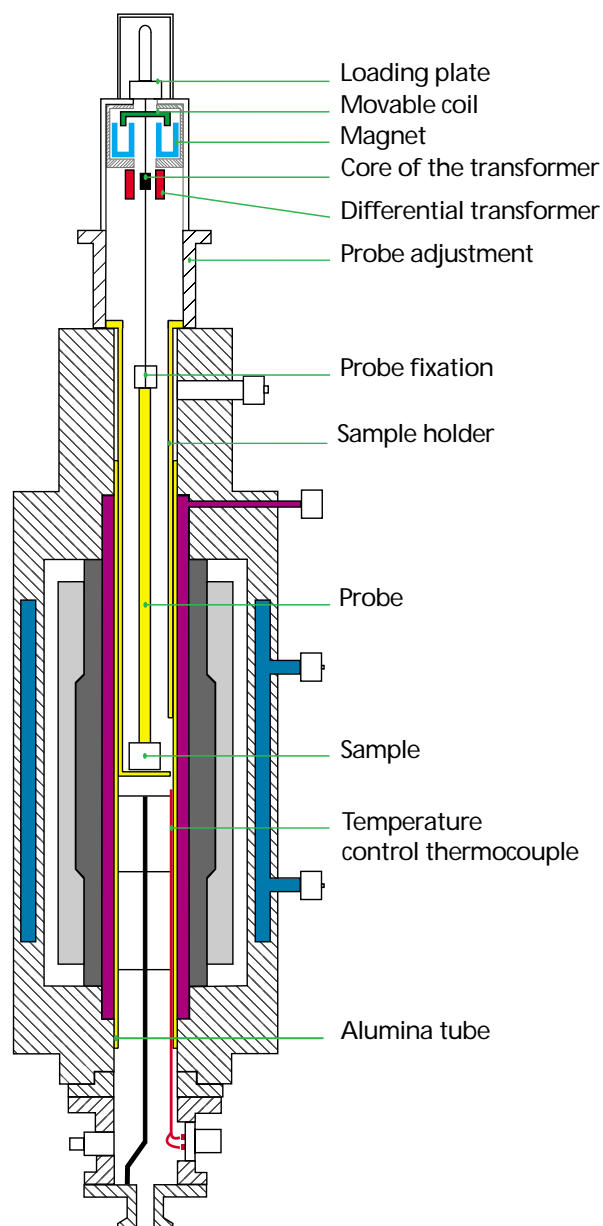
Three temperature versions are available according to the regulation used:

- Ambient to 1600°C: S type regulation thermocouple
- Ambient to 1750°C: B type regulation thermocouple
- Ambient to 2100°C: W5 type regulation thermocouple

Adapted probes

The TMA 96 is used with "spherical end compression" type probes.

The probes are made of alumina for ambient temperature to 1750 °C, whereas graphite probes are used above 1750°C.



TMA	Type of probe	Max. sample volume	
		Ø (mm)	H (mm)
Amb / 1600° C Amb / 1750° C	Alumina	18 (15 recommended)	50
Amb / 2100° C	Graphite	18 (15 recommended)	50

96 Line

Technical characteristics

LIGNE 96	DTA 96	DSC 96	MHTC 96 hf-DSC	MHTC 96 drop	TGA	TMA
Temperature range of measurement module (°C)	amb / 1600 amb / 1750 amb / 2100	amb / 1500 amb / 1600	amb / 1400 amb / 1600	amb / 1300 amb / 1500	amb / 1600 amb / 1750 amb / 2100	amb / 1600 amb / 1750 amb / 2100
Volume of crucible (μ l)	220 / 550	360 / 420	300 / 450	5000 / 5700	18000	/
Max. sample size (mm)	/	/	/	/	/	L=50 D:18 (15 recommended)
Resolution	/	5 μ W	7 μ W	8 μ W	0,3 μ g	1,6 nm
RMS noise	/	66 μ W	83 μ W	330 μ W	0,3 μ g	10 nm
Specific RMS noise	/	0.16 μ W/ μ l	0.18 μ W/ μ l	0.05 μ W/ μ l	0.016 μ g/ml	0.2 . 10 ⁻⁶
Measurement range	/	/	/	/	+/- 2000 mg	+/- 6 mm

The 96 Line is equipped with SETSOFT 2000, the thermal analysis software from SETARAM