



instruments





THERMOGRAVIMETRIC ANALYZER



YEARS

# THERMO-GRAVIMETRIC ANALYSIS

Thermogravimetric analysis is used to determine the mass loss of a sample as a function of the temperature. Suitable instruments include standard laboratory ovens and muffle furnaces with a fixed temperature and subsequent weighing, as well as TGA analyzers with integrated balance and a variable temperature range.

ELTRA's TGA Thermostep ML combines the drying and ashing process with integrated weighing. For the determination of various thermogravimetric parameters in one analysis cycle, the software allows to define different temperatures and gases (e.g. oxygen or nitrogen) for each analysis step.

# **RELIABLE AND FLEXIBLE**

ELTRA TGA analyzers are an ideal alternative to standard laboratory ovens and muffle furnaces for thermogravimetric analysis. Thanks to a programmable furnace that is connected to an integrated balance, heating and weighing are combined in one instrument. This saves time-consuming manual work and allows for high sample throughput. In addition, typical parameters such as moisture, ash and volatiles can be determined in one analysis run.

The TGA Thermostep ML processes up to 19 different samples, typically weighing between 500 mg and 5 g, in one analysis cycle. The surrounding atmosphere and temperature of up to 1,000 °C within

the heating chamber can be freely defined by the user during analysis to create a standard operating procedure. The crucible lids, covering of the samples, can be raised or lowered at each stage of the analysis, thus allowing for safe and ASTM-compliant determination of volatiles in coal samples.

#### TYPICAL SAMPLE MATERIALS

Coal, coke, secondary fuels, gypsum, flour, plastics, ceramics and many more.



ELTRA THERMOSTEP ML

#### **BENEFITS**

- Measurement of up to 19 samples in one analysis
- I Sample weights of up to 5 g
- Fast heating rates, accurate temperature control
- I High-performance, precise weighing cell
- Robust design allows for use in laboratories and production

#### **PRECISE RESULTS**

### HIGH-PERFORMANCE ANALYSIS TECHNOLOGY

The TGA Thermostep ML is a powerful thermogravimetric analyzer characterized by robust design, high precision and flexibility. It is possible to apply different atmospheres and to use sample weights of up to 5 g. The Thermostep reliably and efficiently measures parameters such as moisture, ash and volatiles according to a user-defined SOP.

#### **PURGING GAS**

The TGA Thermostep ML is very flexible with regards to the purging gas used. At each stage of the analysis either nitrogen, oxygen or surrounding atmosphere can be selected. In the latter, the surrounding atmosphere penetrates into the TGA Thermostep ML, gently oxidizing the samples.

#### **TEMPERATURE CONTROL**

The furnace temperature is monitored by two thermocouples which are not encapsulated. One thermocouple monitors the temperature inside the furnace, the other monitors the temperature within the heating element. Due to the absence of the encapsulation the heating can be controlled quickly and precisely.

#### **NEW: ENCAPSULATED WEIGHING CELL**

The latest TGA Thermostep ML generation features an encapsulated weighing cell with 0.1 mg resolution providing highly precise measurements. The encapsulation isolates the weighing cell from the ambient atmosphere and is extremely stable. The weighing cell is connected to the furnace by a ceramic spindle with pedestal on which the crucibles are placed.





#### HIGH-CAPACITY HEATING ELEMENTS

The latest TGA Thermostep ML generation utilizes three heating elements with an improved capacity of 1800 W each (5400 W total power). The result is a faster heating rate and improved stability, especially at high temperatures. The heating elements, located in the upper and lower furnace, provide homogeneous temperature distribution.



#### COOLING

At the end of each analysis cycle, the cool down process starts. It is possible to program the automatic opening of the TGA furnace lid as a function of the temperature to support the cool down process. For example, the Thermostep can be programmed to open the furnace lid at 650 °C halfway and at 500 °C completely. In addition, at 300 °C an integrated fan is automatically started.

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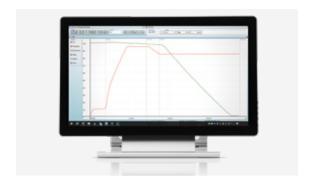
# SIMPLE OPERATION YIELDS QUICK RESULTS

Operation of the TGA Thermostep ML is simple, convenient and safe. After selecting the Standard Operating Procedure (SOP) in the PC, the sample ID's can be entered into the software. The samples are then weighed in the crucible at the position assigned to the sample ID in the carousel. After one sample has been weighed, the carousel automatically rotates to the next position and the next registered sample can then be weighed in the crucible.

Alternatively, a carousel filled with samples which has been weighed externally, can be placed into the analyzer. Once the analysis is finished, a new cycle can be started after a short cool-down period.



Weighing the sample



Display of analysis results

# WORKING IN COMPLIANCE WITH STANDARDS

The ELTRA TGA Thermostep ML complies with the following international standards, among others:



Norm	Material Name	
D7582 - 12	Coal, coke	Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis
D7348 - 08e1	Combustion residues	Standard Test Methods for Loss on Ignition (LOI) of Solid Combustion Residues

#### THE TGA APPLICATION INSTRUCTIONS

In order to determine thermogravimetric parameters with the TGA Thermostep ML, an application instruction must be created once. For this purpose, the general conditions for the individual analysis steps are defined once in the Thermostep software. An application for complete coal analysis consists, for example, of the determination of moisture, volatile components and ash. An analysis step includes the specification of start and end temperature, the purging gas to be used, the heating rate and the end criterion.

Both time and mass stability can be selected as criteria for the end of an analysis step. In addition, it can be specified in each analysis step whether the crucible lids are to be put on.



#### THE ELTRA APPLICATION LABORATORY

For many applications (e.g. TGA analysis of plastics) there are no standards for automated thermogravimetric analysis. However, in order to guarantee a safe and reliable measurement, the ELTRA laboratory in Haan is available for application advice and free trial measurement using the complete analyzer range (TGA, as well as C/S and O/N/H analysis).

Our participation in round robin tests (e.g. ASTM Powder Metallurgy) and in the certification of reference materials (e.g. ECRM 268-1; ECRM 049-1) ensure a consistently high analysis quality.

# **EASY SAMPLE OPERATION**

#### SAMPLE CAROUSEL AND REFERENCE CRUCIBLE

The metal sample carousel accepts up to 19 ceramic crucibles. Position no. 20 is reserved for the reference crucible which is part of every measure- ment. It is used to compensate for weight loss in the crucible, a physical effect which could lead to measurement errors at high temperatures.

#### **SAMPLE WEIGHING**

The samples are weighed automatically in the TGA Thermostep ML. The analyzer allocates the positions of the crucibles in accordance with the number of samples to be measured to ensure the best possible stability during weighing. The software then connects to every occupied position and weighs one sample after the other.

Optionally, an external weighing station is available. Thus it is possible, for example during the final stages of cooling down of the TGA Thermostep ML, to weigh in a new sample carousel and introduce it to the analyzer with one single movement. This procedure helps to reduce waiting times between two analysis cycles.

#### **CRUCIBLE LIDS**

For applications such as the precise and ASTM-compliant analysis of volatiles in coal or of very reactive sample materials, it is essential to cover the crucibles. The TGA Thermostep ML is equipped not only with a sample carousel but with a second carousel for the crucible lids.

A software-controlled mechanism integrated in the carousel holder lifts and lowers the lids without interrupting the analysis by opening the TGA.

#### **BENEFITS**

- I A maximum of 19 samples plus 1 reference crucible
- I Automatic, integrated weighing



#### **CLEARLY STRUCTURED AND CONVENIENT**

# PC CONTROL WITH WINDOWS®-BASED SOFTWARE

 ${\tt ELTRA's\ instrument\ software\ ensures\ convenient\ control\ and\ operation\ of\ the\ analyzers.}$ 

It is multilingual, easy to understand and provides the following features:

- I Custom layouts: user-defined display of windows and storage of different layouts
- I User profiles with multi-level access: creation of different hierarchy levels with different authorizations
- I Storage of analysis results in data base:
- I The data of each analysis is stored and can be called up later
- I Graphic display of temperature profile and mass loss

- I Individual, customer-specific calculations based on the raw data
- Retrieval of sample-related information from any given time during analysis
- I LIMS communication and data export
- Applications memory and display of maintenance intervals: individual configuration of maintenance intervals
- I Extensive diagnostics function

#### **CUSTOMIZED VISUALIZATION OF MEASUREMENT RESULTS**

- I Display of measurement results after each analysis step
- I Individual calculations possible
- I Ash content can refer to dry or moist samples
- I Export and printing of measurement results

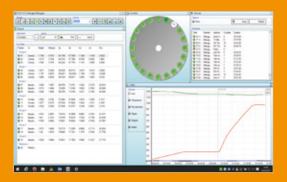




Simultaneous display of temperature (red) and loss in weight (green)



Display of analysis results in groups



Simultaneous display of result, graph and currel measurement position

#### **APPLICATIONS**

# **ANALYSIS OF COAL**

The determination of moisture, ash and volatiles in coal is a routine application in coal-fired power plants. This can be done manually with various muffle furnaces or in a TGA Thermostep ML.

The TGA Thermostep ML meets the requirements of, for example, ASTM Norm D7582.



# COAL CALIBRATION STANDARD

**Number of samples** 19 samples

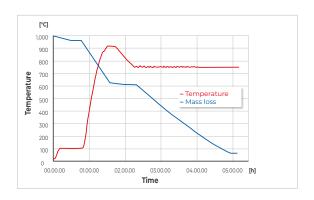
Average weight

1.1 g coal

Analysis time

5 hours

Parameters	Mean value	Standard deviation
Moisture	0.32 %	0.08
Ash	6.6 %	0.05
Volatiles	9.1 %	0.3



# **ANALYSIS OF CHEMICALS**

The TGA Thermostep ML is ideally suited to determine the various degrees of decomposition of chemicals at different temperatures.

The example shows calcium oxalate; the moisture content was analyzed at  $105\,^{\circ}$ C, the mass loss at  $200\,^{\circ}$ C,  $450\,^{\circ}$ C and  $850\,^{\circ}$ C.



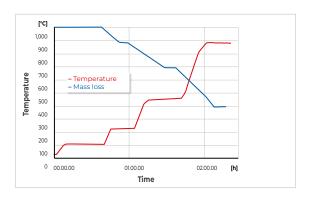
# TYPICAL RESULTS CALCIUM OXALATE

Number of samples 10 samples

**Average weight** 500 g

**Analysis time** 2.5 hours

Temperature	Mean mass loss	Standard deviation
105 °C (moisture)	0.2 %	0.01
200 °C	12.2 %	0.02
450 °C	18.9 %	0.05
850 °C	29.8 %	0.03



# **ANALYSIS OF CEMENT**

The **LOI test (loss on ignition)** is particularly important for inorganic materials. For this test the sample is quickly heated to a defined high temperature. This method is used to rapidly determine the volatile components without modifying the sample characteristics too much.

To determine residual moisture in cement an intermediate step at  $105^{\circ}$ C was added to the LOI test at  $1,000^{\circ}$ C. The total analysis time for both parameters in a 1g sample was 70 minutes.



# TYPICAL RESULTS CEMENT

Number of samples

10 samples

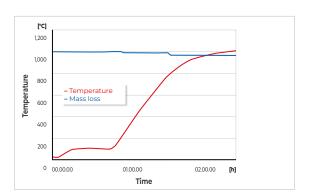
Average weight

1a

Analysis time

70 minutes

Parameters	Cement 1	Cement 2
Moisture (105 °C)	0.07 ±0.01 %	3.0 ±0.02 %
LOI (1,000 °C)	0.08 ±0.01 %	1.9 ±0.01 %



#### **TECHNICAL DATA**

Sample weight	up to 5 g
Number of samples	19 (+ 1 reference sample)
Number of sample carousels	1 (crucibles)
Precision	0.02 %
Resolution of balance	0.1 mg
Furnace temperature	From room temperature to 1,000 °C
Temperature control	Precision: 2 % or ±2°C / stability: 2 % or ±2°C
Gas flow rate	Adjustable from 1 to 10 l/min
Gas pressure	Air 5 – 6 bar (75 – 90 psi) / nitrogen 2 – 4 bar (30 – 60 psi) / oxygen 2 – 4 bar (30 – 60 psi)
Gas purity	Compressed air 99.5 % (oil and fat free) / nitrogen (99.9 %); oxygen (99.9 %)
Operating temperature / humidity	10 – 35 °C / 20 – 80 % humidity (not condensating)
Exhaust air	Connection to required / fan included in delivery scope / 4 m³ per minute / diameter of: 100 mm
Power supply	230 V (±10 %) / one phase / 50/60 Hz / 32 A (analyzer) 230 V (±10 %) / one phase / 50/60 Hz / 2 A (PC, fan)
Weight	65 kg
Dimensions (B x H x T)	55 x 52 x 62 cm
Interfaces	serial and USB
Accessories	Computer, monitor, printer (exact specifications on request)





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SCIENCE FOR SOLIDS

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