Specialists for Elemental Analysis

For more than 30 years ELTRA has been one of the leading manufacturers of elemental analyzers. Starting with combustion analyzers for carbon and sulfur determination ELTRA has extended its product range over the years with analyzers for oxygen, nitrogen and hydrogen as well as thermogravimetric analyzers. ELTRA instruments are used in industries such as steel, mining, automotive and aviation, construction materials and in universities for Research & Development.

ELTRA is synonymous with high quality, customer-oriented solutions and efficient products. Thousands of satisfied customers worldwide are proof of the reliability of ELTRA analyzers.
Thermogravimetric 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 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.

Thermogravimetric Analyzers

For organic and inorganic samples

<table>
<thead>
<tr>
<th>TGA Thermostep</th>
<th>04</th>
</tr>
</thead>
<tbody>
<tr>
<td>In the TGA Thermostep up to 19 samples with a maximum weight of 5 g each can be analyzed fully automatically in one analysis cycle for their thermogravimetric parameters. The analyzer is ideally suited to characterize organic (such as coal), synthetic (such as plastics) and inorganic samples (such as cement).</td>
<td></td>
</tr>
</tbody>
</table>

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ELTRA also provides analyzers for:

<table>
<thead>
<tr>
<th>CS in inorganic samples</th>
<th>CHS in organic samples</th>
<th>ONH in inorganic samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>The <strong>CS-800</strong> is ideal for the quick simultaneous determination of carbon and sulfur in steel, cast iron, nonferrous metals, carbides, ceramics, glass, cement and other inorganic samples.</td>
<td>The <strong>CHS-580</strong> is used for the quick simultaneous determination of carbon, hydrogen and sulfur in samples such as coal, coke, ores, minerals, slag, and many more.</td>
<td>The <strong>ELEMENTRAC ONH-p</strong> is ideally suited for the quick simultaneous determination of oxygen, nitrogen and hydrogen in steel, cast iron, molybdenum, nickel, copper, zirconium, titanium, ceramics and other inorganic samples.</td>
</tr>
</tbody>
</table>
TGA Thermostep

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

**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 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
Simple operation yields quick results: TGA Thermostep

Operation of the TGA Thermostep 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. It is also possible to position a second carousel with crucible lids above the crucibles. Once the analysis is finished, a new cycle can be started after a short cool-down period.

The Standard Operating Procedure

Determination of thermogravimetric parameters with the TGA Thermostep requires the definition of a standard operating procedure (SOP). The conditions for each analysis step are defined in the software. A full range coal analysis, for example, consists of the determination of moisture, volatiles and ash content. An analysis step includes start and end temperature, purging gas, heating rate and end condition. A condition for ending the analysis step can be a defined period of time as well as the obtained mass stability. Moreover, the placement of the crucible lids can be defined for each analysis step.

The graphic shows a typical temperature profile of coal measured with the TGA Thermostep. The flexibility of the Thermostep Software allows for a variety of modifications. Once a standard operating procedure has been defined it can be selected in the software for further analyses.
The TGA Thermostep 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.

NEW: Encapsulated weighing cell

The latest TGA Thermostep 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.

Benefits
- Precise measurements
- Long-term stability
- Low maintenance
- Long operating life
High-capacity heating elements

The latest TGA Thermostep 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.

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.

Purging gas

The TGA Thermostep 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, gently oxidizing the samples.

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.
Intelligent Crucible Management

Benefits

- A maximum of 19 samples plus 1 reference crucible
- Automatic, integrated weighing
- Separate carousel for crucible lids

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 measurement. 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. 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, 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 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.

ASTM-compliant working with TGA Thermostep

ASTM compliance for the measurement of ash, moisture and volatiles

<table>
<thead>
<tr>
<th>Standard</th>
<th>Material to be analyzed</th>
<th>Standard title</th>
</tr>
</thead>
<tbody>
<tr>
<td>D7582 - 12</td>
<td>Coal and Coke</td>
<td>Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis</td>
</tr>
<tr>
<td>D7348 - 08e1</td>
<td>Solid Combustion Residues</td>
<td>Standard Test Methods for Loss on Ignition (LOI) of Solid Combustion Residues</td>
</tr>
</tbody>
</table>
PC control with Windows®-based software

ELTRA’s instrument software ensures convenient control and operation of the analyzers. It is multilingual, easy to understand and provides the following features:

- Custom layouts: user-defined display of windows and storage of different layouts
- User profiles with multi-level access: creation of different hierarchy levels with different authorizations
- Storage of analysis results in database:
- The data of each analysis is stored and can be called up later
- Graphic display of temperature profile and mass loss
- Individual, customer-specific calculations based on the raw data
- Retrieval of sample-related information from any given time during analysis
- LIMS communication and data export
- Applications memory and display of maintenance intervals: individual configuration of maintenance intervals
- Extensive diagnostics function

Customized visualization of measurement results

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

Clearly structured and convenient

![Simultaneous display of temperature (red) and loss in weight (green)](image1)
![Display of analysis results in groups](image2)
![Simultaneous display of result, graph and current measurement position](image3)
The TGA Thermostep applies temperatures of up to 1,000°C under inert or oxidative atmosphere. It allows for flexible and cost-effective thermogravimetric analysis of a great variety of sample materials.

**Typical sample materials**

*Coal, coke, secondary fuels, paper, flour, plastics, ceramics*

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**Example:**

**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. The optional automated crucible lid management of the Thermostep ensures reliable determination of volatile components. In contrast to analyzers of other manufacturers, it is not necessary to open the Thermostep or run a second analysis cycle to determine the volatiles.

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

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**ELTRA coal calibration standard**

19 samples / average weight: 1.1 g coal / analysis time: 5 hours

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Mean value</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>0.32 %</td>
<td>0.08</td>
</tr>
<tr>
<td>Ash</td>
<td>6.6 %</td>
<td>0.05</td>
</tr>
<tr>
<td>Volatiles</td>
<td>9.1 %</td>
<td>0.3</td>
</tr>
</tbody>
</table>
Example:

**Analysis of residual moisture and LOI in 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 °C was added to the LOI test at 1,000 °C. The total analysis time for both parameters in a 1 g sample was 70 minutes.

### Typical results cement

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Cement 1</th>
<th>Cement 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture (105 °C)</td>
<td>0.07 ±0.01 %</td>
<td>3.0 ±0.02 %</td>
</tr>
<tr>
<td>LOI (1,000 °C)</td>
<td>0.08 ±0.01 %</td>
<td>1.9 ±0.01 %</td>
</tr>
</tbody>
</table>

Example:

**Analysis of chemicals**

The TGA Thermostep 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 °C, the mass loss at 200 °C, 450 °C and 850 °C.

### Typical results calcium oxalate

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Mean mass loss</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>105 °C (moisture)</td>
<td>0.2 %</td>
<td>0.01</td>
</tr>
<tr>
<td>200 °C</td>
<td>12.2 %</td>
<td>0.02</td>
</tr>
<tr>
<td>450 °C</td>
<td>18.9 %</td>
<td>0.05</td>
</tr>
<tr>
<td>850 °C</td>
<td>29.8 %</td>
<td>0.03</td>
</tr>
</tbody>
</table>
## Technical Data

### Thermogravimetric Analyzers

**TGA Thermostep**

#### General data

- **Sample weight**: up to 5 g
- **Number of samples**: 19 (+ 1 reference sample)
- **Number of sample carousels**: 2 (crucibles and lids)
- **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 hour / 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)