Figure 1 Thermal Gravimeter Analyzer

DESCRIPTION

Simultaneous thermal analysis refers to the simultaneous application of two or more thermoanalytical methods to the same sample in one instrument. This term, however, is in most cases used for simultaneous measurement of the mass changes and caloric effects on a sample under thermal treatment.

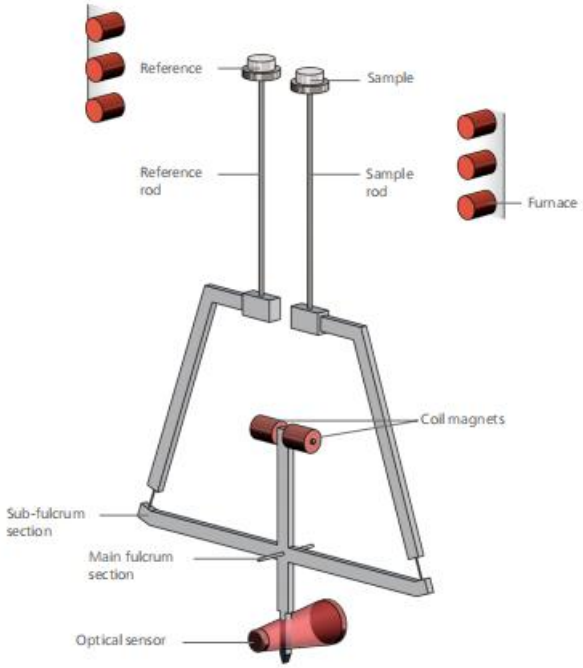
The benefits of such a system are obvious:

- Often, the material available for testing is expensive or difficult to produce. The transformation energetics and mass change are obtained on one sample in one run; the time necessary for the tests is cut in half.
- Influences of the measurement conditions and/or sample preparation can be eliminated. Comparability of characteristic temperatures of the mass changes and caloric effects is ensured.
- Problems resulting from differences in the sample composition for the two simultaneous measurements are removed for inhomogeneous materials.

Standard Solution with Trendsetting Technology

The Machine is equipped with a tailor-made ultra-micro balance with a resolution of 0.03 µg. The self-compensating differential balance operates with two symmetric balance arms, connected to the reference and sample rod in the furnace.

The compensation system (coil magnets) is controlled by a highresolution optical sensor. This avoids rod movements resulting in a stable, high-resolution balance with a weighing range of ± 250 mg. The differential balance set-up compensates for buoyancy effects, even during changes in gas flow.



Gypsum Waste Analysis

Gypsum boards are widely used in applications requiring high fire resistance. The material contains water within its structure, which vaporizes in the presence of fire, enhancing fire protection. Gypsum is fully recyclable, making it one of the few construction materials suitable for closed-loop recycling. To study its thermal behavior under fire exposure and assess its recycling potential, gypsum waste powder was analyzed from room temperature (RT) to 700°C in an air atmosphere. The results showed an endothermic dehydration of gypsum up to 250°C, with a mass loss of approximately 18%. This process was followed by the exothermic burn-up of organic components, which may overlap with the phase transition from Anhydrite III to II. Above 600°C, the mass remained nearly constant, indicating the stability of the material.

Superior Reproducibility

The carbon black content in rubber materials is critical for quality control. In this study, three different rubber mixtures from the same batch were analyzed for their carbon black content. The decomposition behavior was first monitored under an inert atmosphere, after which the atmosphere was switched to air. The measured mass change was **0.282 ±0.006%**, demonstrating the **exceptional reliability and reproducibility** of the **Thermal Gravimetric Analyzer** in precise material analysis.

Phase Transitions of Iron

The Thermal Gravimetric Analyzer provides high DTA sensitivity, allowing the detection of even weak phase transitions. Additionally, its AutoVac function ensures a pure atmosphere by automatically evacuating and refilling the system. The STA measurement of an iron sample from RT to 1600°C revealed a caloric effect at 744°C, indicating a change in the material's magnetic properties. Further crystal structure transformations were observed at 908°C and 1389°C, while an endothermic peak at 1533°C (extrapolated onset) corresponded to the material's melting point. No mass change was detected in the TGA signal, confirming the vacuum-tightness of the system and the high purity of the sample.

Sublimation of an Organic EL (OEL) Layer

Multilayer organic structures exhibit unique optical and physical properties, making them valuable for optoelectronic applications, such as organic light-emitting diodes (OLEDs). One such organic material, α-NPD, is commonly used as a hole transport layer. The vacuum-tight Thermal Gravimetric Analyzer allows for precise sublimation measurements under reduced pressure. Under standard atmospheric pressure, α-NPD sublimates at 380°C (blue curve), while under reduced pressure, the sublimation temperature decreases to 240°C (red curve), demonstrating how pressure affects material phase transitions.

Flavor Capsule in Tobacco Filters

Modern cigarette filters feature embedded flavor capsules containing a liquid solution, designed to enhance or modify the taste of tobacco while maintaining moisture. The TGA-MS measurement of a water-immersed flavor capsule from RT to 500°C revealed multiple mass-loss steps (black TGA curve and dotted red DTGA curve), confirming the sequential release of water. The MS signal (mass number 18) further validated the evaporation of free and bound water. The Thermal Gravimetric Analyzer ensures fast balance stabilization before the test, allowing for the early detection of water evaporation before the release of bound moisture.

Testing and Measuring Capabilities

Caloric Effects

- Phase transition temperatures
- Melting/crystallization behavior
- Phase diagrams
- Reactivity
- Modification changes

Thermogravimetric Effects

- Mass changes
- Temperature stability
- Oxidation/reduction behavior
- Decomposition
- Corrosion studies
- Compositional analysis
- Thermokinetics

Coupling to Evolved Gas Analysis (EGA)

By integrating the Thermal Gravimetric Analyzer with a gas analysis system such as an FT-IR (Fourier Transform Infrared) spectrometer, QMS (Quadrupole Mass Spectrometer), or GC-MS (Gas Chromatograph – Mass Spectrometer), precise identification of evolved gases can be achieved as a function of time or temperature. This enables detailed material characterization and provides a unique fingerprint of the analyzed sample.

- **FT-IR Connection:** Via transfer line for infrared gas analysis.
- **QMS Connection:** Using a capillary for high-precision mass spectrometry.
- **GC-MS Coupling:** Through a transfer line for advanced gas chromatography-mass spectrometry integration.

THERMAL GRAVIMETRIC ANALYZER
S1-HT-102

FEATURES

- **Top-Loading & Differential Balance System:** Ensures automatic protection against condensation and contamination while canceling buoyancy and convection effects, eliminating the need for baseline runs.
- **Cost-Effective & High Performance:** Offers a reliable, fully equipped instrument package for simultaneous measurements up to 1600°C at an attractive price.
- **Versatile Atmosphere Control:** Supports inert, oxidizing, and vacuum environments with software-controlled mass flow controllers (MFCs) for dynamic or static gas changes during tests.
- **Automatic Sample Changer (ASC):** Optional ASC accommodates up to 20 samples, enabling unattended operation, pre-programmed testing, and enhanced throughput.
- **Seamless Evolved Gas Analysis (EGA):** Easily integrates with FT-IR, MS, or GC-MS systems to analyze gases released during thermal treatment.

SPECIFICATION

DESIGN & BALANCE SYSTEM

- **Type:** Top-loading, vertical design with differential balance system
- **Balance System:** High-precision differential balance for enhanced accuracy

Furnace Specifications

- **Temperature Range:** Room Temperature (RT) to 1100°C / 1600°C (with two easily exchangeable furnaces)
- **Heating Rate:** 0.001 to 100 K/min (varies by furnace type)
- **Temperature Precision (Repeatability):** ±0.3 K

- **Furnace Hoist:** Motorized single hoist for easy operation

Balance & Sensor Data

- **Weighing Range:** ±250 mg, with a maximum sample load of 1 g
- **Thermogravimetric Resolution:** 0.03 µg
- **Temperature Sensor:** Type S thermocouple

Atmosphere Control

- **Vacuum Compatibility:** Yes (dependent on the evacuation system)
- **Supported Gas Atmospheres:** Inert, oxidizing, and vacuum environments
- **Gas Flow Control:** Built-in mass flow controllers for precise regulation

Automation & Sample Handling

- **Automatic Sample Changer (ASC):** Optional ASC available for handling up to 20 crucibles, ensuring high-throughput analysis.

APPLICATIONS

- **Material Science & Engineering Studies**: Enables students to validate theoretical principles through practical application.
- **Research & Development**: Suitable for laboratories conducting basic material strength analysis.
- **Mechanical & Civil Engineering Education**: A vital tool for testing the mechanical properties of materials used in various industries.

SOFTWARE

Regulus Software Overview

The **Thermal Gravimetric Analyzer** operates on a **Windows** platform and includes all necessary tools for measurement execution and data evaluation. Its **user-friendly interface** and **automated routines** within the **software** ensure ease of use while offering advanced analytical capabilities.

Key Software Features:

- Developed for **Windows** operating systems.
- **Simultaneous measurement and evaluation** for efficient data processing.
- Supports **operation of multiple instruments** with a single computer.
- **Combined analysis**: Enables comparison and evaluation of **STA, DSC, TGA, DIL, TMA,** and **DMA** measurements within a unified presentation.
- **Customizable analysis**: Users can input and freely position text elements.
- **Derivative calculations**: Supports **1st and 2nd derivative** computations for precise data interpretation.
- **Flexible data visualization**: Adjustable colors and line types for improved readability.
- **Data management**: Storage and retrieval of analyses, with results available via **email**.
- **Context-sensitive help system** for on-demand user support.
- **Automatic instrument detection**, including furnace and sensor recognition.
- **CSV data export** for seamless integration into external applications.
- **Calibration and correction routines** for **temperature, sensitivity, and baseline adjustments**.

- **Picture-in-picture (PIP/FLIP) display** for real-time evaluation of ongoing measurements.

STA-SPECIFIC FEATURES:

TGA (Thermogravimetric Analysis):

- Displays mass change as absolute (mg) or relative (%) values.
- Automatic evaluation of mass change steps and characteristic temperatures.
- Extrapolated onset and endset determination for phase transitions.
- Peak temperature identification with 1st and 2nd derivative values.
- Stability check for early termination of measurements.
- Rate-controlled mass loss for precise thermal analysis.

DTA (Differential Thermal Analysis):

- Displays DTA curves in absolute (µV) or relative (µV/mg) units.
- Automatic determination of onset, peak, inflection, and end temperatures.
- Automatic peak search for efficient data extraction.
- Exothermal representation in compliance with DIN or ASTM standards (user-selectable).
- Conversion of DTA data into DSC signal calculations.

ADVANCED SOFTWARE FEATURES (OPTIONAL):

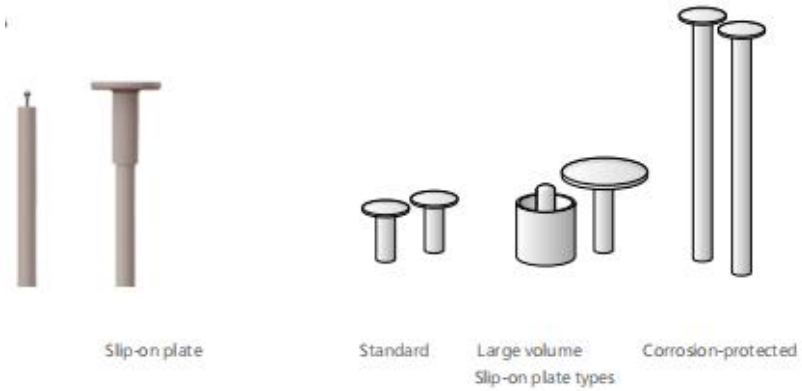
- **Peak Separation Module**: Allows precise differentiation and evaluation of **overlapping transitions**.
- **Thermokinetics Module**: Provides **advanced reaction characterization** and **kinetic parameter evaluation**, enabling predictive analysis of thermal processes.

The Thermal Gravimetric Analyzer software ensures a comprehensive, accurate, and efficient analysis experience, making it an essential tool for high-precision thermal analysis applications.

ACCESSORIES

Sample Pans, Crucibles and Plates

A variety of crucibles made of alumina, platinum, aluminum and quartz are available in different shapes and sizes. The Thermal Gravimetric Analyzer is delivered with slip-on plates which allow for nearly all kinds of sample pans (see table below). The large-volume crucible requires the large slip-on plate.



Crucible Types for Various Applications

Material (Purity)	Diameter / Height (mm)	Volume (µl)	Maximum Temperature (°C)
Al (99.5)	Ø 5.2 / 2.6	45	600
Al (99.5)	Ø 5.2 / 5.1	95	600
Al_2O_3 (99.7)	Ø 5.2 / 2.6	40	1700
Al_2O_3 (99.7)	Ø 5.2 / 5.1	80	1700
Pt/Rh (90/10)	Ø 5.2 / 2.6	45	1600
Pt/Rh (90/10)	Ø 5.2 / 5.1	95	1600
Fused Silica	Ø 5.0 / 2.6	25	1000
Fused Silica	Ø 5.0 / 5.1	55	1000
Large Volume Al_2O_3 (99.7)	Ø 8.0 / 10.0	400	1700

Dimensions & Weights

L x W xH: (0.7 x 0.7 x 0.9) m