Diamond Thermogravimetric/Differential Thermal Analyzer

high sensitivity thermal analysis

PerkinElmer™
precisely.

two techniques,

one solution

QUICK GLANCE

- Patented dual balance beam design for high sensitivity
- Controlled Rate Thermal Analysis (CRTA)
- Easy to connect Evolved Gas Analysis (EGA)
- Autosampler for increased throughput

The Diamond Thermogravimetric/Differential Thermal Analyzer (TG/DTA) combines the high flexibility of the differential temperature analysis (DTA or DSC) feature with the proven capabilities of the thermogravimetry (TG) measurement technology.

The ability to perform these complementary measurements simultaneously aids in the characterization of materials and provides better verification and interpretation of results. The combination allows you to determine whether an endothermic or exothermic transition is associated with a weight loss (e.g. loss of volatile) in contrast to a melting or crystallization process.

The design of the Diamond TG/DTA ensures that the sample is exposed to the identical thermal treatment and environment when comparing the two signals. The result is highly reliable characterization information. The horizontal design makes it possible to use high flow rates and is easily coupled with IR, MS or GC/MS techniques for hyphenated applications.

a broad range of applications

What does TG/DTA measure?

The TG determines the weight change of a sample whereas the DTA measures the change in temperature between a sample and the reference as a function of temperature and/or time.

Key TG/DTA applications

- Compositional analysis
- Decomposition temperatures
- Engine oil volatility measurements (TGA Noack test)
- Filler content
- Flammability studies
- Heat of transition
- Lifetime predictions (via TGA kinetics software)
- Measurement of volatiles (e.g., water, oil)
- Oxidative stabilities
- Thermal stabilities
- Transition temperatures
- Catalyst and coking studies

The Diamond TG/DTA principle

When a weight change occurs on the sample side, the beam is displaced. This movement is detected optically and the drive coil current is changed to return the displacement to zero. The detected drive coil current change is proportional to the sample weight change and is output as the TG signal. The DTA detects the temperature difference between the sample holder and the reference holder using the electromotive force of thermocouples, which are attached to the holders. This differential is output as the DTA signal.

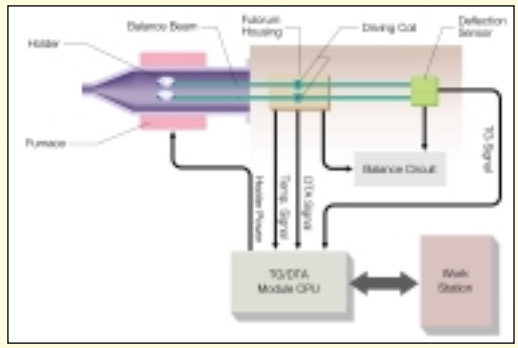


Figure 1. Schematic of the Diamond TG/DTA.

flexibility coupled with high performance

Outstanding Stability and Sensitivity

The Diamond TG/DTA features a proven dual balance beam design which provides drift-free baselines. The dual balance design minimizes the effects of changing purge gases and purge gas flow rates and reduces chimney and convection effects. The system is very resistant to external disturbances such as room temperature fluctuations and/or vibrations. The Diamond TG/DTA provides high quality results for very low mass weight loss events over an entire temperature range.

Easier Data Interpretation

The system automatically converts DTA signals to DSC (Differential Scanning Calorimeter) units providing easier data interpretation. The simultaneous DTA signal featured with the Diamond TG/DTA can be automatically converted from DTA units (mV) to more user-friendly DSC units (mW) based on the heats of melting of high purity metal standards.

True Horizontal Purge Gas Flow

The Diamond TG/DTA features a horizontal balance design, so the gas flow is perpendicular to the weight direction. This means that there is very little effect caused by purge gas flows, even up to rapid purge rates of 1000 mL/min. The Diamond TG/DTA horizontal purging is ideal for hyphenated techniques such as TG-IR or TG-MS. High purge gas flow rates allow measurements of highly condensable or oily volatiles since they are purged from the TG/DTA instrument before significant condensation occurs. This is valuable for difficult tests such as the TGA Noack test for assessing engine oil volatiles.

Accurate Temperature Data Over Wide Temperature Range

The DTA signal featured with the Diamond TG/DTA provides direct temperature calibration using high purity metal standards. The thermocouple is in direct contact with the sample platform ensuring very accurate temperature data. With a 20-point temperature calibration, the Diamond TG/DTA provides the most accurate temperature data over the widest temperature range. In addition, the software makes calibration very easy to perform.

Reactive Atmosphere Handling

A separate purge port is provided for reactive or corrosive purge gases. The separate port permits introduction of the corrosive or special purge gases directly into the furnace tube (bypassing the electronics), minimizing the occurrence of damage to the balance assembly or electronics.

it's never been easier!

High Measurement Efficiency

The Diamond TG/DTA offers automated forced air cooling of the furnace at the conclusion of an experiment to facilitate sample turnaround time.

Vacuum Operation

The design of the Diamond TG/DTA allows for the running of experiments under vacuum conditions (down to 10-2 torr).

User Maintenance

The step beam replacement capability simplifies the beam exchange and allows the user to perform the change easily. Light weight user replaceable parts help minimize service costs and time.

Figure 2. User maintenance.

Hyphenated Techniques

The Diamond TG/DTA is compatible with most FT-IR systems (including the PerkinElmer Spectrum One), Mass Spectrometer and GC-MS systems. The connection is accomplished very easily with the optional gas transfer system. TG/DTA delivers quantitative results whereas the coupling with IR or MS can identify the evolved gases.

Key applications for hyphenated techniques

- Molecular structure analysis
- Identification of decomposition products for safety applications

State-of-the-art Autosampler

The Diamond TG/DTA can couple with a rugged autosampler accessory for fully automated, unattended operation of up to 30 samples at a time, offering unsurpassed reliability.

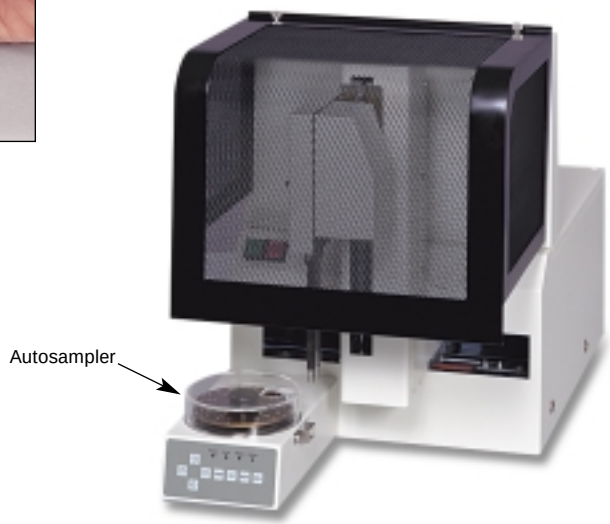


Figure 3. Autosampler of Diamond TG/DTA (protection cover is optional).

software simplifies your analysis

Unsurpassed Resolution and Improved Compositional Analysis

The Diamond TG/DTA permits samples to be analyzed using both the Auto Stepwise Isothermal and Controlled Rate Thermal Analysis (CRTA) techniques. This provides the highest possible resolution of overlapping weight loss events using an automated adjustment of the applied heating rate based on the sample's rate of weight loss. The application of these controlled rate techniques is ideal for compositional analysis or for the unambiguous determination of two or more overlapping weight loss transitions.

Patented Highway Kinetics Software

One of the unique offerings of the Diamond TG/DTA is the special Highway TGA Kinetics software package. This software improves quantitative compositional analysis through sophisticated mathematical algorithms (peak/curve separation and Arrhenius first order kinetics) to better separate overlapping weight loss events. Samples can be analyzed at normal TGA heating rates (e.g. 20˚C/min) and, with the use of the Highway software, the TGA data can be transposed to very slow (0.01˚C/min) heating rates. This provides greatly improved separation of overlapping weight loss events. TGA results can also be transposed to very fast heating rates (>500˚C/min) for estimation purposes. This feature helps to reduce the measurement time by 50% to 90%.

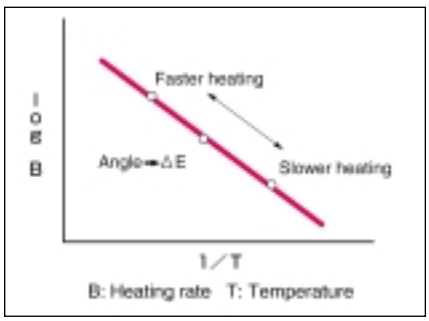


Figure 4. Conversion principle of Highway TA.

Principle of Controlled Rate Thermal Analysis (CRTA)

CRTA is the measurement method which automatically controls the heating rate dependent on the physical property change of a sample. The application mode is set depending on the setting conditions.

Auto Stepwise for Better Step Separation

Upper setting limit > lower limit

Once the reaction rate of the sample reaches the upper limit set by the user, the temperature is held isothermally. Scanning continues when the reaction reaches the lower limit set by the user.

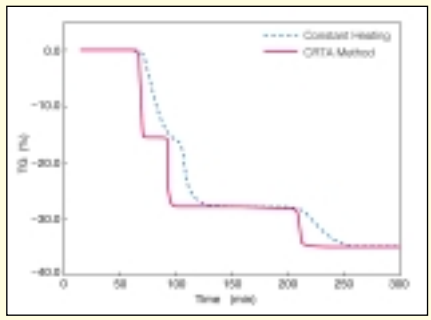


Figure 5. Auto stepwise mode (TG).

Uniform Velocity Control Mode

Upper setting limit = lower limit

Under this condition the heating rate is controlled so that a uniform weight loss is maintained as specified by the user.

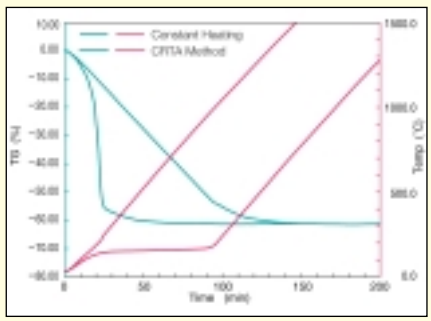


Figure 6. Uniform velocity weight loss control mode (TG).

improve sampling productivity

Gypsum

Gypsum, one of the most widely used minerals in the world, literally surrounds us. Gypsum is used worldwide in concrete for highways, bridges, buildings and many other structures that are part of our everyday life. Most gypsum in the United States is used to make wallboard for homes, offices and commercial buildings. A typical new American home contains more than seven metric tons of gypsum.

The analysis of the material is important for the long-term properties of the product. The TG/DTA data generated with an actual heating rate of 10˚C/min was used to predict a heating rate of 1˚C/min and 0.1˚C/min. By simulating the data with a reduced heating rate, the steps of weight reduction due to dehydration were clearly separated. The Highway Kinetics software dramatically accelerates the analysis and increases laboratory throughput.

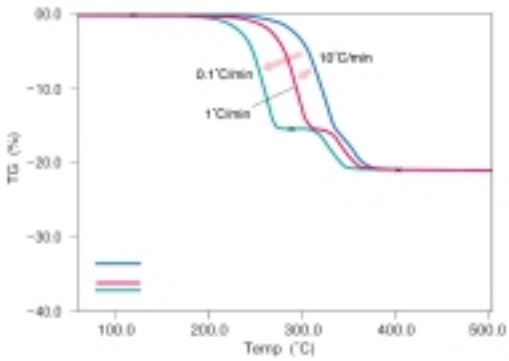


Figure 7. Gypsum run with 10˚C/min (blue) and simulated for 1˚C/min (red) and 0.1˚C/min (green).

Chloroprene Rubber Decomposition Measurements and Highway TA

Data with an actual heating rate of 100˚C/min was used to predict a heating rate of 1˚C/min. By simulating the data with a reduced heating rate, the weight loss steps were clearly separated. The advantage of this technique is a dramatic reduction in experimental time.

Calcium Oxalate

In an inert atmosphere, calcium oxalate shows three weight losses in the TG signal. All three are endothermic reactions as shown in the DTA signal. The first step represents the loss of water followed by CO and finally CO_2.

The data was collected with an actual heating rate of 100˚C/min. The result was used to predict a heating rate of 10˚C/min. When comparing to a scan with 10˚C/min, the measured data is remarkably consistent with the predicted one. With a measurement time of only 9 minutes, results are similar to those that would be obtained by a 90-minute measurement.

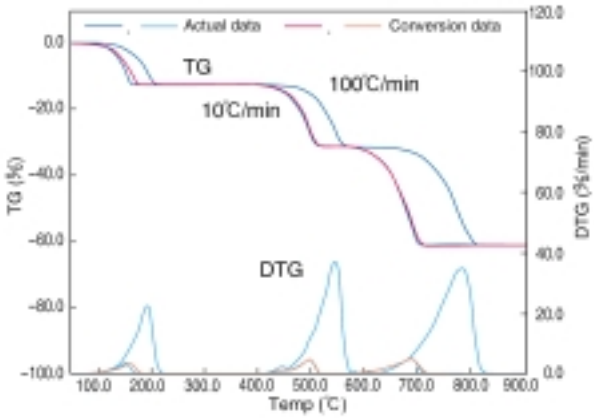


Figure 8. TGA Measurements of Calcium Oxalate.

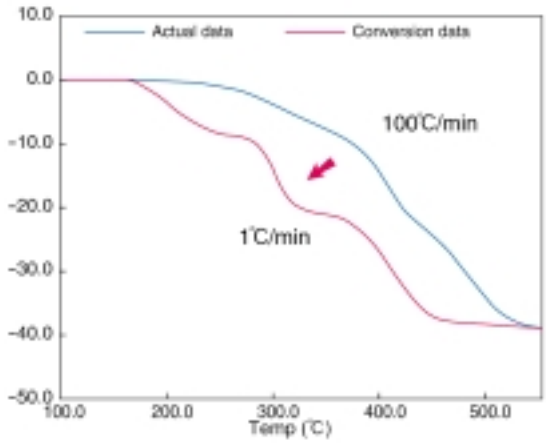


Figure 9. Chloroprene rubber decomposition measurements and Highway TA.

Kaolinite

A clay mineral formed by the weathering of feldspar is one of the most common minerals on earth and is used in the ceramics industry for the production of clay products. Investigations by the Diamond TG/DTA exhibit the following characteristics:

The first small wide DTA peak above ambient temperature is the loss of free water followed by a large endothermal effect between 400˚C and 800˚C which corresponds to a weight loss of water due to a dehydroxylation of the crystal lattice.

The sharp exothermic effect at 1000˚C is due to a structural organization which has been described as the crystallization of the dehydrated matrix into a spinel phase.

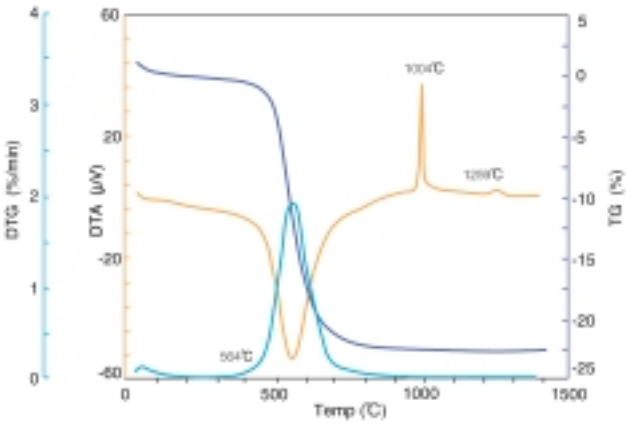


Figure 10. TG/DTA Measurement of Kaolinite with 20°C/min.

Dolomite $Ca\,Mg(CO_3)_2$

Dolomite is a common sedimentary rock-forming mineral that can be found in massive beds that are several hundreds of feet, found all over the world and are quite common in sedimentary rock sequences.

The decomposition of a dolomite sample follows two steps:

$$CaMg(CO_3)_2 \rightarrow CaCO_3 + MgO + CO_2$$
$$CaCO_3 \rightarrow CaO + CO_2$$

Two weight losses are expected in a TG/DTA measurement. When analyzing the sample under nitrogen purge, the two steps are not completely separated. The atmosphere above the sample could have a great influence on the separation of the de-carbonation phenomena. Using CO_2 as a purge gas allows the clear separation of the two expected steps. The first weight loss of approximately 800°C is attributed to the CO_2 evolved in the first step and is equal to 23.9% of the initial mass of the dolomite. The second weight loss of approximately 950°C is attributed to the CO_2 in the second step.

In a dolomite/calcite mixture (50/50), the Diamond TG/DTA is perfectly suited to separate the CO_2 of the second step of the dolomite and the CO_2 of the calcite step.

The first weight loss with 12% proves that the dolomite content is 50.2% (12/23.9) in this mix. With the CO_2 purge gas it is very easy to separate weight losses in dolomite and to quantitatively determine dolomite in calcite.

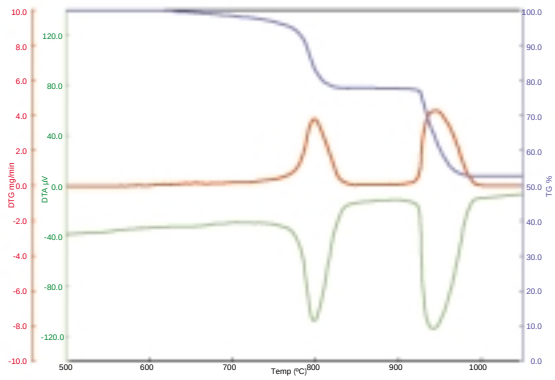


Figure 11. TG/DTA Measurement of Dolomite.

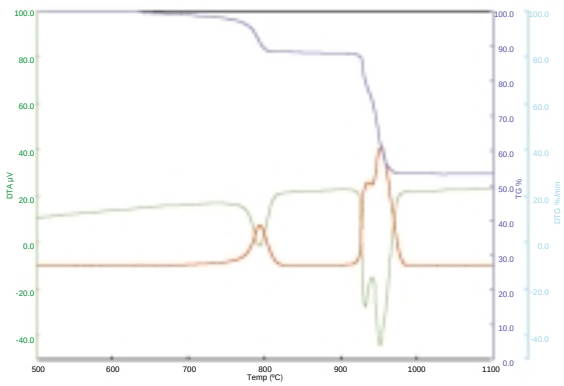


Figure 12. TG/DTA Measurement of Dolomite/Calcite mix.

solutions for material property analysis

DSC

The Differential Scanning Calorimeter (DSC) measures the amount of energy absorbed or released by a sample as it is heated, cooled or held at a constant temperature. This technique is used for polymer and pharmaceutical applications. PerkinElmer offers the best of both worlds—high performance power-compensation technology and value heat-flux DSC technology.

Diamond DSC

Pyris™ 6 DSC

DMA

The Diamond Dynamic Mechanical Analyzer (DMA) measures changes in mechanical behavior, such as modulus and dampening, as a function of temperature, time, frequency, stress or a combination of these parameters. It analyzes a wide variety of polymers, polymer blends, elastomers and composites. DMA is also used in foods, pharmaceuticals, electronics, coatings, high performance aerospace materials and various inorganic materials.

Diamond DMA

TMA

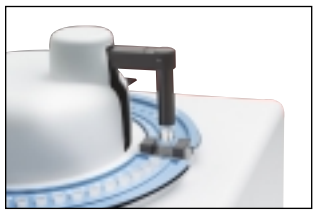
The Diamond Thermomechanical Analyzer (TMA) determines dimensional changes in materials as a function of temperature or time. It is used in research and quality control to measure changes in length, width, thickness and linear expansion of materials.

Diamond TMA

TGA

The Thermogravimetric Analyzer (TGA) measures the change in weight of a sample as it is heated, cooled or held at a constant temperature. Our instruments provide robustness and reliability for quality control and the answers researchers need to solve even the toughest problems.

Pyris 1 TGA



Pyris 6 TGA Autosampler

Diamond TG/DTA Specifications	
Temperature Range	Ambient – 1500°C
Automatic Cooling Unit	Forced Air Cooling
Cooling time	1000°C - 50°C within 15 min
Balance Type	Horizontal Differential Type
TG measurement Range (Sensitivity)*	200mg (0.2µg)*
DTA Measurement Range (Sensitivity)*	+/- 1000µV (0.06µV)*
Programmable Rate	0.01 – 100°C/min
Sample Pan Material	Platinum , Aluminum, Alumina
Sample Pan Volume	45 µl or 90µl optional
Atmosphere	Air, Inert Gas, Vacuum (10-2 Torr)
Purge Gas flow Rate	0-1000ml/min

* The value used when the heater is working is based on the measurement condition of SII standard

Ordering Information

N535-0010	Diamond TG/DTA Lab System 115V Transformer required for 230V use
N535-0012	Diamond TG/DTA Lab System - Vacuum 115V Transformer required for 230V use
N535-0100	EXSTAR Thermal Analysis Software and Muse

Optional Accessories

N535-0202	Diamond TG/DTA Air Cooling Unit with gas switching feature
N535-0200	Diamond TG/DTA Autosampler with 30 positions
N535-2028	Protection Cover for Autosampler Unit
N535-0201	Diamond TG/DTA Gas Transfer System Standard Unit for hyphenated applications, requires N535-0203 for FTIR and N535-0204 for MS
N535-0203	Diamond TG/DTA Gas Transfer Accessory for FTIR
N535-0204	Diamond TG/DTA Gas Transfer Accessory for MS

Contact your local sales representative for further information or a quotation.

PerkinElmer, Inc.

Expect MORE from the LEADER in high sensitivity thermal analysis instruments

With over 40 years experience and a complete product offering that includes Differential Scanning Calorimeters (DSC), Thermogravimetric Analyzers (TGA), simultaneous Thermogravimetric/Differential Thermal Analyzers (TG/DTA) and Mechanical Analyzers (DMA and TMA), PerkinElmer is the leader in high sensitivity thermal analysis instrumentation. In combination with the largest technical service and support staff in the industry and its solid reputation for high-sensitivity products, PerkinElmer thermal analysis equipment continues to deliver fast, accurate, reproducible results.

Whatever you're looking for, we've got it

PerkinElmer Life and Analytical Sciences is a world leader in chemical analysis. Our Analytical Instrument technologies serve the fast-evolving pharmaceutical, chemical, environmental and semiconductor industries, providing integrated solutions—from sample handling to interpretation and communication of test results.

As one of the best-known brands in research, analysis and testing, ours was probably the first analytical instrument you ever used. In addition to our Thermal Analysis systems, we offer a broad range of solutions in Luminescence, UV-Vis, NIR, GC, GC/MS, MALDI-TOF MS, HPLC, AA, ICP, ICP-MS, Elemental Analysis, FTIR and LIMS. There are over 60 years of experience built into every product we make. So, for leading edge R&D and demanding QA/QC, you get the speed, accuracy and reliability you seek, for the productivity you need.

Our service and support people are located in 125 countries throughout the world and are factory trained. Compliance doesn't get any easier than with our Enhanced Security software, including 21 CFR Part 11 technical compliance on many products. And online consumables and accessories ordering lets you get your hands on what you need fast.

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