

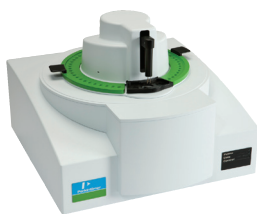
Thermogravimetric Analysis (TGA)

A Beginner's Guide

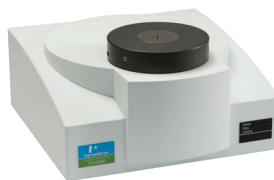
This booklet provides an introduction to the concepts of Thermogravimetric Analysis (TGA). It is written for scientists unfamiliar with TGA.



Pyris 1 TGA



TGA 4000



STA 8000

The Thermogravimetric Instrument Family

The Thermogravimetric Analyzer (TGA) is an essential laboratory tool used for material characterization. TGA is used as a technique to characterize materials used in various environmental, food, pharmaceutical, and petrochemical applications. PerkinElmer is the leader in TGA. We have been manufacturing Thermal Analysis Tools since 1960, and no one understands the applications of TGA like we do. In the following pages, we answer common questions about what a TGA is, how they work, and what they tell you.

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22 Common Questions about TGA

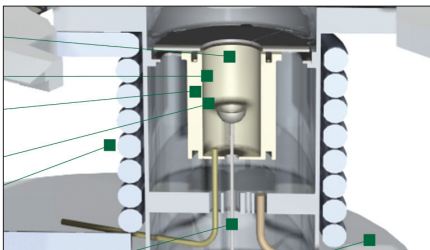
Q What is TGA?

A Definition: Thermogravimetric Analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere.

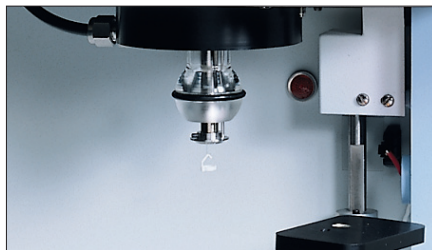
An Alternate Definition: TGA is a technique in which, upon heating a material, its weight increases or decreases. **A Simple TGA Concept to remember:** TGA measures a sample's weight as it is heated or cooled in a furnace.

A TGA consists of a sample pan that is supported by a precision balance. That pan resides in a furnace and is heated or cooled during the experiment. The mass of the sample is monitored during the experiment. A sample purge gas controls the sample environment. This gas may be inert or a reactive gas that flows over the sample and exits through an exhaust.

PerkinElmer offers two types of TGAs, a top-loading TGA 4000 and a bottom-loading or hangdown, Pyris 1 TGA. The TGA 4000 supports the sample pan above the balance via a "stem" support rod. The Pyris 1 TGA supports the sample pan via a "hangdown" below the balance. Both styles take advantage of gravity to obtain very accurate and reproducible measurements.



TGA 4000.



Pyris 1 TGA.

These instruments can quantify loss of water, loss of solvent, loss of plasticizer, decarboxylation, pyrolysis, oxidation, decomposition, weight % filler, amount of metallic catalytic residue remaining on carbon nanotubes, and weight % ash. All these quantifiable applications are usually done upon heating, but there are some experiments where information may be obtained upon cooling. Both the Pyris™ 1 TGA and the TGA 4000 are controlled by PerkinElmer's proprietary thermal software, Pyris Software, and have autosampler accessories for unattended operation.

Both TGAs can be used for Evolved Gas Analysis incorporated into a hyphenated analytical system.

- Pyris 1 TGA – IR, MS or GC/MS
- TGA 4000 – IR or MS

Q What is the difference between the Pyris 1 TGA and TGA 4000?

A

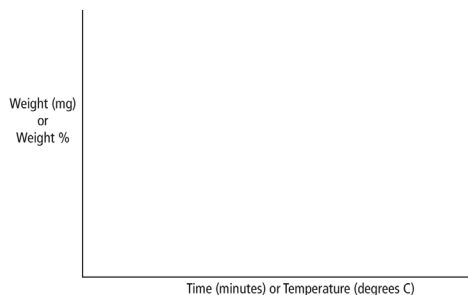
Table 1. TGA Differences.

	Pyris 1 TGA	TGA 4000
Balance Type	Hangdown Pan	Top-Loading Pan
Standard Furnace	Temperature Range: Subambient to 1000 °C	Temperature Range: Ambient to 1000 °C
High Temperature Furnace	Temperature Range: 50 °C to 1500 °C	None
Balance Precision	0.001%	0.01%
Balance Capacity	1300 mg	1500 mg

Q How is a TGA Thermal Curve displayed?

A

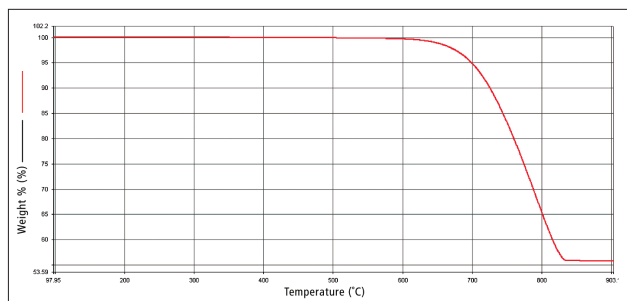
The abscissa (X-axis) can be displayed as time or temperature and the ordinate (Y-axis) can be displayed as weight (mg) or weight percent (%).



Q What does a TGA Thermal Curve look like?

A

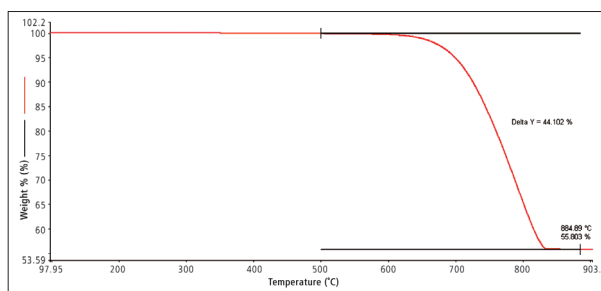
A TGA thermal curve is displayed from left to right. The descending TGA thermal curve indicates a weight loss occurred. A 15.013 mg sample of calcium carbonate was analyzed. Temperature Program = Heat from 100 °C to 900 °C @ 10 °C/minute in Nitrogen atmosphere with a purge rate of 20 mL/minute.



Q What can we learn from this TGA Thermal Curve?

A We must first list all pertinent information we know about the sample before we can analyze the data. In this case of calcium carbonate we know:

- CaCO_3 is an irritant!; so be careful handling the sample, wear eye protection
- It is hygroscopic (it absorbs or attracts moisture from the air).
- Upon heating calcium carbonate it undergoes a reaction where bound CO_2 is released from the material and only calcium oxide remains after the experiment.



Now we can fully investigate this material. The first calculation that could be done, just to ensure the sample is pure, is to calculate the formula weight by substituting the atomic mass in the formula.



Where: Ca = 40.08 atomic mass units (amu)

C = 12.011 amu

O = 15.9994 amu

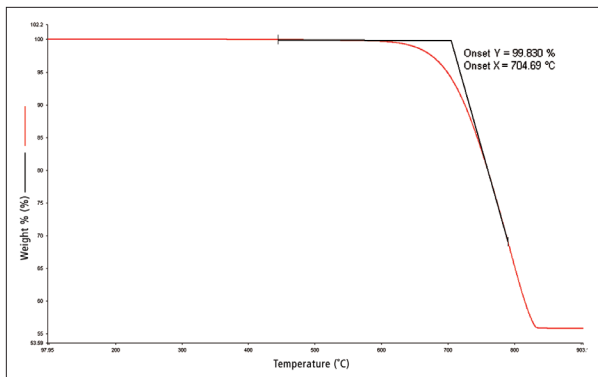
$$\text{Ca} + \text{C} + \text{O}_3 \\ (40.1) + (12.0) + [(16) \times (3)]$$

$$\begin{array}{rcl} \text{CaCO}_3 \rightarrow & \text{CaO} & + \quad \text{CO}_2 \\ 100.1 & = & [(40.1) + (16)] + [(12.0) + (16) \times (2)] \\ & & (56.1) \quad + \quad (44) \end{array}$$

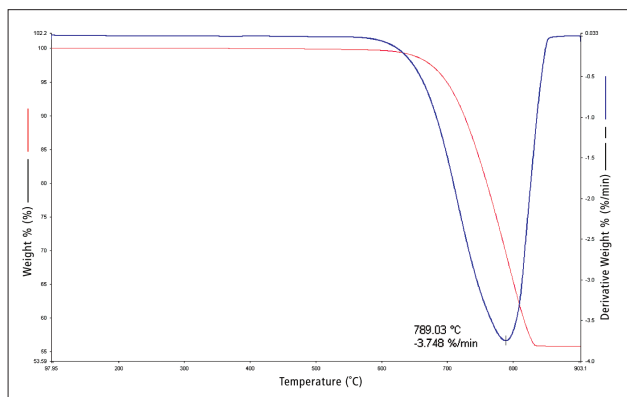
$$(100.1) = (100.1) \text{ amu}$$

The measured values are almost exactly equal to the calculated values. The delta Y software calculation was used to measure the “as-run” sample. So the first characteristic that we learn about this sample is that it is very pure.

Component	Expected/Calculated Values	Measured Value
CaO	56.1 amu	55.803 amu
CO ₂	44 amu	44.102 amu



To further characterize this calcium carbonate sample we can calculate the extrapolated onset temperature that denotes the temperature at which the weight loss begins. We use the extrapolated onset Temperature (T_0) because it is a reproducible temperature calculation and it is specified to be used by ASTM®, <http://www.astm.org/> and ISO, <http://www.iso.org>. The extrapolated onset Temperature is 704.69 °C.



The next calculation that is useful and finalizes the characterization of calcium carbonate is the peak calculation of the 1st derivative of the weight loss curve. The 1st derivative curve is easily displayed by selecting that option under the MATH drop-down list. The 1st derivative peak temperature (T_p) is 789.03 °C. The peak of the first derivative indicates the point of greatest rate of change on the weight loss curve. This is also known as the inflection point.

Q What is reproducibility and why is it important?

A Reproducibility is the ability to create the same experiment over and over. Reproducibility is very important when comparing one set of data to another. If the experiment is reproduced the same each time then any variation within the data is due to a change in the sample; not operator error!

There six (6) TGA operating variables that contribute to reproducibility:

- Calibration
- Furnace Cleanliness
- Sample Preparation
- Temperature Range
- Temperature Scanning Rate
- Sample Atmosphere

Q When should I check the TGA calibration?

A Calibration is easily checked. It is best to check the TGA calibration periodically. How often should this be done? This depends on the nature of the samples you're running. If you are running samples that relatively clean and do not coat the furnace or the furnace tube (Pyrex® glass surrounding the furnace area), then it is recommended to check daily, weekly, or somewhere in between. As a new TGA user, it is best to check calibration first each day before you begin your experiments. Once you gain an understanding of the nature of your calibration and often how it changes, you can then change the checking interval to, for instance, every other day.

If you move your instrument, then check calibration.

If you shut your instrument down for a long period of time, then check calibration.

If you are running samples that create decomposition residue that collects on the furnace and on the furnace tube. Please clean your furnace between runs. Check your calibration after cleaning the furnace and the furnace tube.

Q How do I clean the TGA furnace?

A The furnace is easily cleaned by having the furnace open to air and heating it up to 900 °C and actually "cooking" all organic residue off the furnace. For the Pyris 1 TGA, there is a CLEAN Button on the software Instrument Control Panel. Once depressed, this action lowers the furnace and elevates the temperature for enough time to clean the furnace. All organic condensates will be baked off.

Q When and how do I clean the TGA furnace tube?

A For the Pyris 1 TGA it is recommended to have two (2) furnace tubes if you are running samples that generate heavy black decomposition products that collect on the furnace tube. Having two (2) furnace tubes enables you to run the TGA with a clean furnace tube and while you scrub the dirty furnace tube. You may ask, “When do I know if the furnace tube is affecting my analysis?” You’ll know when you see a Curie point change during calibration check and the Curie point is not within your acceptable temperature limits. Please see the HELP files for the procedure to remove the furnace tube.

To clean the furnace tube, use mild soap and water and a bottle brush to scrub the inside of the furnace tube. If this doesn’t remove the residue, then use the appropriate solvent. When using solvents always read the MSDS and if possible consult with your lab safety person for any precautions. Once clean, rinse the furnace tube with water and let it air dry.

Q What is the best way to prepare a sample?

A This depends on the nature of your sample. Below are six (6) suggestions to consider when preparing a sample.

- The sample size should be between 2 and 50 mg.
- If you have minimum amount of sample, run at least 1 mg.
- If possible, cover the bottom of the pan with the sample material.
- The sample pans, ceramic or platinum, can accommodate liquids, powders, films, solids or crystals.
- Once you have decided on the sample form, then for best results, use approximately the same sample weight during each experiment. This will ensure reproducibility.
- Many small pieces of sample are better than one large chunk. It is better to have a large surface area exposed to the sample purge.

Q What temperature range should I run in?

A You should know your sample. You should know the approximate temperature of the material’s transition of interest. Once you have determined the event temperatures, perform a survey scan. A survey scan is run at 20 °C per minute and it begins and ends 100 °C below and above the transition of interest. Review the survey scan and adjust the beginning and ending temperatures accordingly.

Please be aware that there may be an occasion to study a material at an isothermal temperature. Your TGA is capable of conducting isothermal experiments and also cooling experiments. For cooling experiments, the scanning rates may be limited to slower rates than heating. This all depends on the temperature limits of your experiment.

Q What temperature scanning rate should I use?

A Sometimes you may want to change the temperature scanning rate. If you need better resolution of the transitions you should scan slower, in this case, maybe change from 20 °C to 10 °C per minute.

If you are **not** concerned about the temperature transition and only want to know inert filler content of the material, then you could scan at 50 °C/minute and decompose the polymer quickly and just calculate the remaining filler after all other is fully decomposed. This will save time and be reproducible.

Q What do I need to know about sample atmosphere?

A At the beginning of each day always check the gas supplies to the instrument. Ensure that the purge rates are set correctly. If you are using bottled gas, then check how full the tank is. If it shows less than 300 psi in the tank, consider replacing the tank. It is always best to change your purge gas before the tank runs dry. If you are using house gas, then you might consider installing a filter or a dryer or both in the gas supply line.

Important: Never use pure hydrogen gas. If you are going to use a specialty gas besides air, oxygen, argon, or nitrogen, then please check with your gas supplier or lab safety person to ensure that you are aware of all potential hazards.

Many material scientists conduct oxidative studies with a TGA. This means that either scanning or isothermal, air or oxygen is used as the sample environment. This can be done by employing the oxidative gas directly from the very beginning of the experiment or introducing it during the experiment. Introducing an oxidative gas during an experiment is quite easy. It is done either through a Mass Flow Controller built into the analyzer and controlled by Pyris Software or it is introduced via an external mass flow controller that is controlled by Pyris Software.

Important: For the Pyris 1 TGA it is important to remember that the balance purge gas should always remain nitrogen, even if you are switching sample gases. Also it is important to always have the balance purge 10 mL/minute higher than the sample purge. Some people run the balance purge differential higher than that. This balance gas positive purge differential prevents volatile gases from back streaming into the balance area. The last thing you want to happen is to have volatile residue contaminate your ultra precision balance. Never purge the balance with reactive gases.

Important: Many times oxidative studies create hydrocarbon decomposition residue that may coat your furnace and furnace tube. Some scientists increase the air or oxygen purge to 50 mL/minute to evacuate these containments through the exhaust. Please remember to increase the nitrogen balance purge also.

Q When should I calibrate my TGA?

A The following are reasons to check calibration:

- When you change the temperature range that you've been working in.
- When you change the purge gas rate.
- When you change the purge gas.
- For the Pyris 1 TGA – when you change the hang down wire.
- For the TGA 4000 – when you change the sample pan holder (the stem).
- If the instrument is moved or re-leveled.

If you feel the instrument calibration exceeds your acceptable temperature limits, then recalibrate. Typical calibration accuracy limits are within 1 °C to 5 °C of the expected value. You must choose your limits based on your application.

Q How do I calibrate my instrument?

A Your instrument has a series of calibration routines located in the Pyris Software under Calibrate. Please read the HELP files about calibration before calibrating your TGA.

Important: always restore calibration default values before performing a new calibration. You can tell if the calibration default values have been implemented by the date and time of each calibration routine.

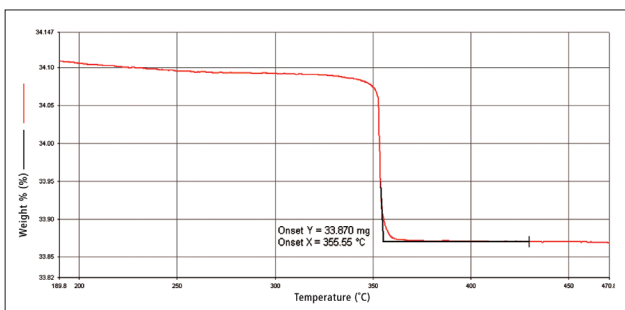
The calibration routines include:

- **Restore Defaults**
- **Balance calibration** – balance calibration is a self contained routine that prompts the user to empty the balance pan, zero the balance, and place a calibration weight that is supplied with the instrument, in the sample pan.
- **Furnace calibration** – furnace calibration is a 9 point calibration that has limits defined by the user. All you have to do is follow the prompts input the high and low range temperature limits and depress the start calibration button. The furnace calibration is a self contained routine that completes in approximately fifty (50) minutes.
- **Temperature calibration** utilizes Curie point Reference Materials. Curie point Reference Materials are materials that upon heating lose their affinity for magnetism. The exact Curie point is the point where the material no longer exhibits magnetic properties. This point is located at the “End set” of the weight loss. Below is an example of a Nickel Curie Point calculation of a Pyris 1 TGA using the ONSET Calculation at the end of the weight loss.

Temperature calibration is a self contained routine that prompts the user to introduce a Curie point standard cut ~3 mm in length, into the sample pan; then zero the pan and Reference Material. The software prompts you to introduce a magnetic force to create a synthetic weight, read that weight, and then the routine conducts a temperature scan.

The magnetic force is applied by placing a permanent magnet below the sample pan, for a Pyris 1 TGA or placing the permanent magnet above the sample pan, for a TGA 4000.

Because the magnet is pulling up on TGA 4000 because the permanent magnet is placed on top of the sample, the thermal curve would be similar to the Pyris 1 TGA weight loss curve above, **except** the curve would be inverted indicating a weight gain.



Nickel Curie Point Calculation – The Curie point is the temperature at which a material does not have an affinity for magnetism.

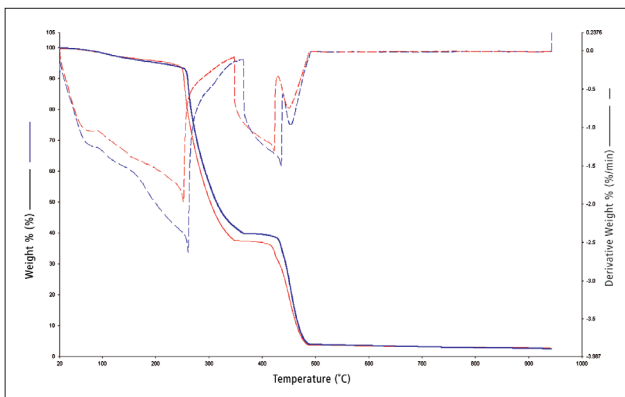
TGA Application curves

Most TGA experiments use an inert sample purge gas. This is done so the sample only reacts to temperature during decomposition. When the sample is heated in an inert atmosphere this is called pyrolysis.

Pyrolysis is the chemical decomposition of organic materials by heating in the absence of oxygen or any other reagents.*

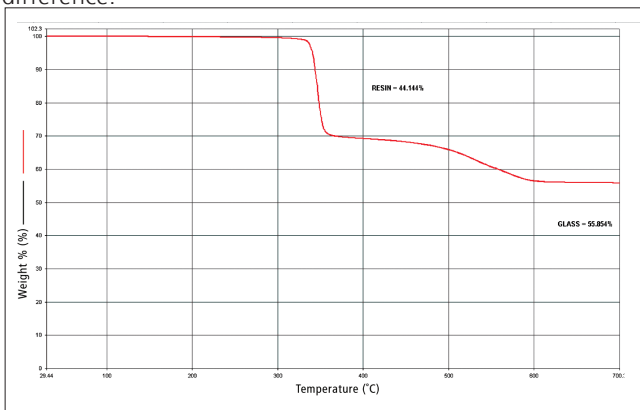
There are times when you may want to use a reactive sample purge gas, such as oxygen. When using oxygen as a purge gas you may want to switch gases from nitrogen to oxygen during the experiment. This is a common technique to identify the percent carbon in a material.

* <http://www.reference.com/browse/pyrolysis>



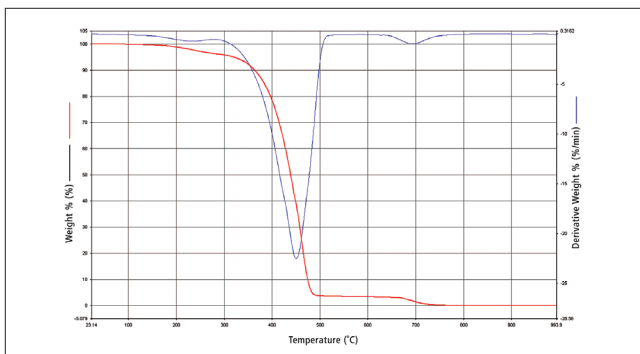
Q How can the TGA be used to compare two (2) similar products?

A Here two (2) types of coffees are compared, Zimbabwe in blue and Columbian in red, using the same method conditions. The solid lines are the weight loss curves and the dashed lines are their respective derivatives. The differences are easily displayed, but it will take a coffee scientist to interpret the significance of the difference.



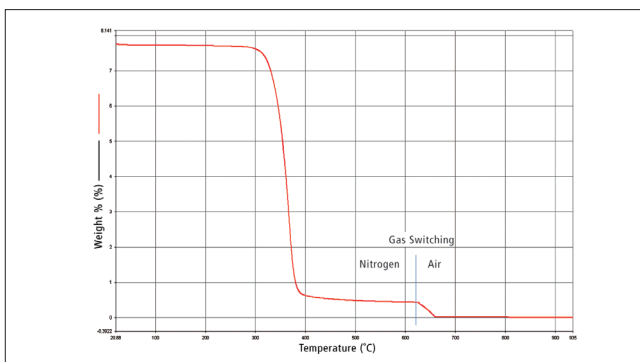
Q How can the TGA be used as a QA/QC tool to ensure products meet their material specifications?

A Above is a fiberglass reinforced printed circuit board. This analysis determines the amount of resin and the amount of fiberglass used in this material. The delta Y calculation was used to determine the component percentages. Resin = 44.144%; Glass = 56.854%. The resin decomposes in two steps, first rapidly, then slows as the last remaining resin decomposes.



Q How can the TGA be used to ensure product safety?

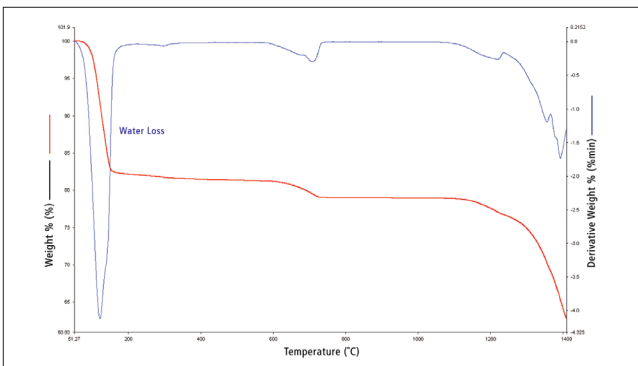
A Above is a three (3) component material, ABS – Acrylonitrile butadiene styrene. This ABS has a high butadiene concentration. The red weight loss curve shows three distinct weight losses representing each component. The blue derivative curve has 3 peaks. This confirms that there are three (3) distinct thermal events taking place in this experiment. The percent of each component must be consistent for this product to provide useful and safe functionality. An alternate TGA application is using TGA AutoStepwise analysis to examine ABS.*



Q Can the TGA be used to determine carbon content?

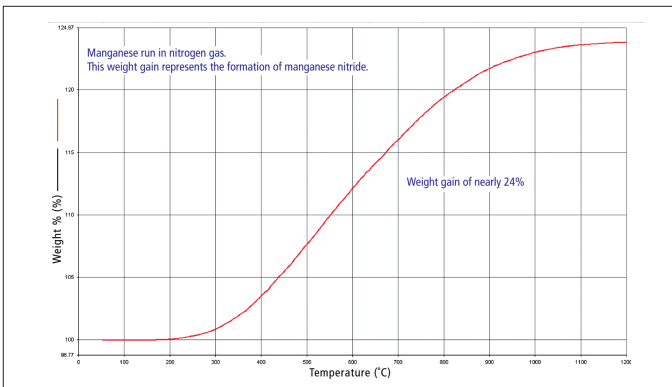
A Here is an ethyl cellulose sample; the experiment utilized gas switching. This was done on a TGA 4000. The sample gas purge rate was 30 mL/minute for nitrogen and air. The gas-switch from nitrogen to oxygen was triggered by a simple software command from within the sample method. The delta Y percentage after the gas switch is the carbon content of this material.

* <http://las.perkinelmer.com/content/applicationnotes/app-autostepwisetgaseparationabs.pdf>



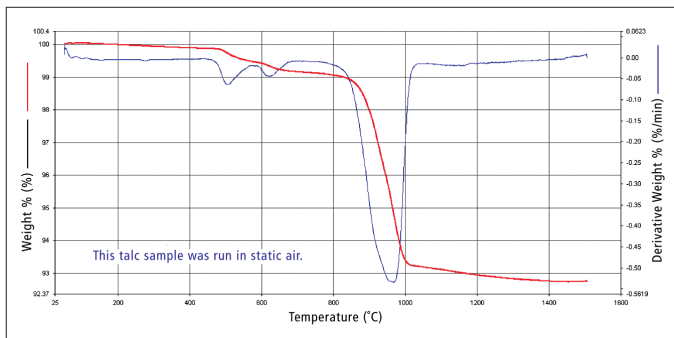
Q Can the TGA be used to identify a counterfeit products?

A Above is a sample of commercial grade gypsum run in static air from 50 °C to 1400 °C in the Pyris 1 TGA equipped with the High Temperature Furnace. The scanning rate was 10 °C/minute. The red curve is the weight loss curve and its 1st derivative is the blue curve. The water loss of 18.5% begins at the initial 50 °C mark and continues evolving up to 150 °C. The values were calculated using the onset calculation and the delta Y calculation.



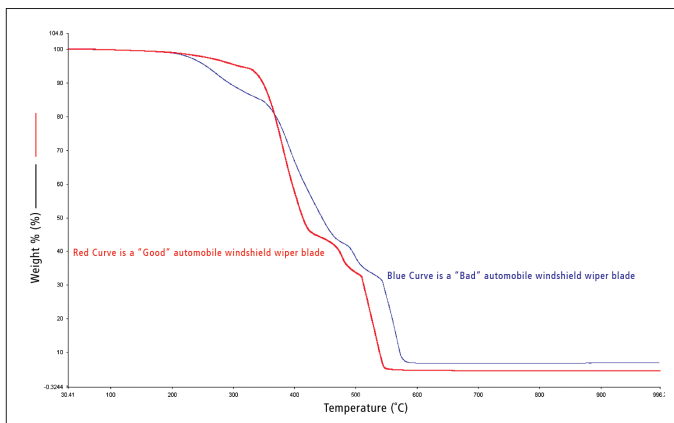
Q Can the TGA be used to identify safe operating temperatures in various gases?

A Above is an example of a weight gain experiment. A manganese sample was run in a nitrogen atmosphere on a Pyris 1 TGA with High Temperature Furnace. The sample purge rate was 30 mL/minute. The delta Y calculation with calculation limits were arbitrarily set at 115 °C and 1190 °C. In this experiment the weight gain is due to the formation of manganese nitride. When manganese is run in argon, there is no weight change.



Q How can the TGA be used to enhance product formulation processes?

A Above talc sample was run in static air from 50 °C to 1500 °C in the Pyris 1 TGA equipped with the High Temperature Furnace. Scanning rate was 10 °C/minute. The red curve is the weight loss curve and its 1st derivative is the blue curve. Static air was used as a purge gas because talc is processed into a personal healthcare product in that environment. A better understanding of when thermal events occur enables a process to be optimized.



Q How can the TGA be used to reverse engineer a product?

A The TGA is frequently used as a QA/QC tool. Here two products are being compared. Comparisons are done frequently by TGA. Many times competing products from different suppliers are distinguished by TGA.

Q How can I increase my laboratory's productivity?

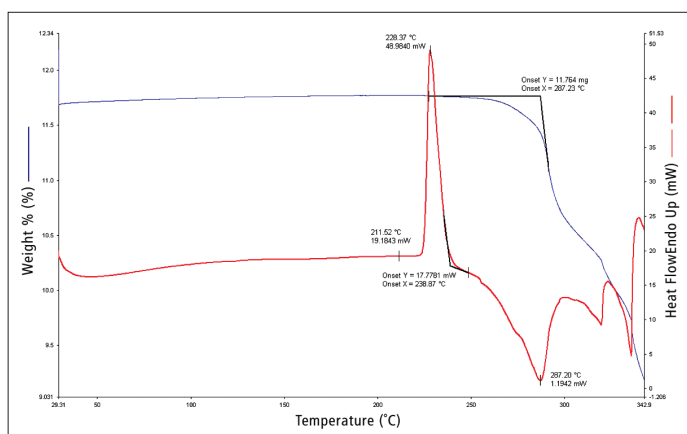
A The Pyris 1 TGA, TGA 4000, and STA 6000 have optional autosamplers that enable your instrument to run unattended.

Q What should I do if I have a limited amount of sample and little time to fully characterize the sample?

A The STA 6000 combines two analytical techniques together. It combines TGA and DSC. Simultaneously the STA 6000 collects DSC heat flow data and TGA weight loss data. DSC Heat Flow is measured in Joules/gram and milliwatts.

The STA 6000 is designed with routine and research applications in mind; the STA 6000 Simultaneous Thermal Analyzer applies leading edge sensor technology to yield higher accuracy and quality results. The patent pending SaTurnA™ sensor and proven compact furnace ensure better temperature control, more consistent measurements, and the fastest cool-down time than any other simultaneous TGA-DSC analyzer.

Simultaneous TGA-DSC application curve



This pharmaceutical sample was characterized by the STA 6000. The Red DSC thermal curve and the Blue TGA weight loss curve are displayed above.

This sample is a free base, small-molecule crystalline powder. The DSC curve indicates that there is a crystalline melt defined by the peak temperature at 228.37 °C. After the melt transition, the baseline returns to a slightly lower position than the pre-melt baseline. This change of 1.4 mW, 19.18 mW less 17.77 mW indicates that the liquid phase has a lower heat capacity (C_p) than the crystalline phase. The post-melt baseline changes slope as the sample begins decomposition. The DSC exothermic decomposition peak at 287.2 °C corresponds to the TGA extrapolated onset temperature of 287.2 °C as this sample decomposes.

Q What is evolved gas analysis?

A Several techniques are used to analyze the gas products from a TGA experiment. This approach is called evolved gas analysis, (EGA). These are a subset of hyphenated techniques where 2 or more instruments are combined.

The following are TGA – EGA techniques:

TGA-FT-IR – A Thermogravimetric Analyzer (TGA) combined with an Infrared Spectrometer (TG-IR) is the most common type of Evolved Gas Analysis (EGA) system. When you heat a sample on the TGA, the sample will release volatile materials or generate combustion components as it burns. These gases are then transferred to your IR cell, where the components can be identified. This technique is most useful when the evolved gases are known to be one of a small set, such as water, carbon dioxide or common solvents which have characteristic IR spectra.

TGA-MS – The combination of a TGA with a MS allows you to detect very low levels of impurities in real time. By heating a sample on the TGA, the sample will release volatile materials or generate combustion components as it burns. These gases are transferred to the MS where the components can be identified. Because of its ability to detect very low levels of material, the TG-MS is a powerful tool for quality control, safety, and product development. This technique is most useful when the evolved gases or breakdown products are known in advance but are few in number.

TGA-GC/MS – The combination of a TGA with a GC/MS is becoming increasingly popular. When you heat a sample on the TGA it causes gases to be released. These gases are then transferred to the GC where the components can be collected. The sample can then be run by GC to separate the material and the peaks identified by the MS. Because of its ability to detect very low levels of material in complex mixtures, the TG-GC/MS is a powerful tool for quality control, safety, and product development.



TGA-FT-IR



TGA-MS



TGA-GC/MS

Q Can TGA analyze Nanomaterials?

A TGA is widely used as a QA/QC tool in the manufacture and use of Carbon Nano Tubes (CNT). TGA is used in CNT manufacturing process to characterize the amount of metallic catalytic residue that remains on the CNT. This is done because CNT are classified by percent purity; in other words 100% less the percent of catalytic residue (carbon to metal content). TGA is used to characterize end products that contain Nanoparticles (NP) or CNTs as in their usual end product characterizations.

Characterization of coatings on NPs and CNTs by evolved gas analysis can be achieved using TGA-EGA techniques. Both NP manufacturers and manufacturers of end products that contain NPs and CNTs use these techniques. And with TGA-Hyphenated systems, the TGA can always be used alone as a simple TGA.

Conclusion

TGA analysis is widely used to characterize and verify materials. TGA is applicable to most industries. Environmental, food science, pharmaceutical, and petrochemical applications are the mainstay of Thermogravimetric Analysis and Evolved Gas Analysis.

Useful Internet Links

PerkinElmer Instrument Training

<http://las.perkinelmer.com/Trainings/Courses.htm>

PerkinElmer Corporate Website

<http://www.perkinelmer.com/>

PerkinElmer Nanomaterials Reference Library

<http://www.perkinelmer.com/nano>

PerkinElmer TGA Application Notes

<http://las.perkinelmer.com/Catalog/TechLibDetails.htm?expand=Application%20Notes&ObjectId=Thermogravimetry+%5bTGA%2c+TG%2fDTA%5d&CategoryId=Thermogravimetry+%5bTGA%2c+TG%2fDTA%5d&type=CATEGORY.com>

ASTM® Plastics TGA Methods

D2288	Test Method for Weight Loss of Plasticizers on Heating
D4202	Test Method for Thermal Stability of PVC Resin
D2115	Test Method for Volatile Matter (including water) of Vinyl Chloride Resins
D2126	Test Method for Response of Rigid Cellular Plastics to Thermal and Humid Aging
D3045	Recommended Practice for Heat Aging of Plastics Without Load
D1870	Practice for Elevated Temperature Aging Using a Tubular Oven
D4218	Test Method for Determination of Carbon Black Content in Polyethylene Compounds by a Muffle-Furnace
D1603	Test Method for Carbon Black in Olefin Plastics
D5510	Practice for Heat Aging of Oxidatively Degradable Plastics
E1131	Standard Test Method for Compositional Analysis by TGA
E1641	Standard Test Method for Decomposition Kinetics by TGA

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