TGA 2050
Thermogravimetric Analyzer
**TGA: The Technique**

Thermal analysis is the generic name used to describe a series of analytical techniques which measure physical and chemical changes in materials as a function of temperature and time. Thermogravimetric analysis (TGA) is one of the most widely used thermal analysis techniques and specifically measures the weight changes (gains and losses) in materials. Such measurements provide information about the material’s thermal stability as well as the material’s compositional analysis. TGA can be used to characterize both inorganic and organic materials (including polymers).

**What TGA Can Tell You**

TGA experiments provide important information that can be used to select materials, predict product performance, and improve quality. The technique is particularly useful for determining:
- Composition of multicomponent systems.
- Thermal stability of materials.
- Oxidative stability of materials.
- Estimated lifetime of a product (through reaction kinetics).
- Decomposition kinetics of materials.
- The effect of reactive atmospheres on materials.
- Moisture and volatiles content of materials.

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**TA Instruments TGA 2050**

The TA Instruments TGA 2050 is based on a unique vertical balance/horizontal purge design and provides a series of features which make it ideal for laboratories that want a high quality TGA, but that have a limited budget. These include:

- **Excellent TGA performance.** The unit’s high sensitivity (0.2 µg), high capacity (1 g), and broad temperature range (ambient to 1000°C) are equivalent to specifications normally associated with expensive research-grade instruments. The TGA 2050 has many automated features such as automated sample pan loading which make it ideal for less experienced operators. Furthermore, its design makes for easy interfacing to evolved gas analysis techniques (e.g., mass spectroscopy or FTIR).

- **Compact design** which makes efficient use of valuable laboratory bench space.

- **Modular design** which provides a cost-effective initial system price, but still allows future expansion into other thermal analysis techniques if laboratory needs change.

- **Complete applications and service support** including a variety of training aids for new TGA users and immediate access to knowledgeable applications and service specialists by telephone.

**Principle of Operation**

The electromechanical components of the TGA 2050 Thermogravimetric Analyzer are shown in Figure 1. The system operates on a null-balance principle, using a highly sensitive transducer coupled to a taut-band suspension system to detect minute changes in the mass of a sample. An optically actuated servo loop maintains the balance arm in the horizontal reference (null) position by regulating the amount of current flowing through the transducer coil. An infrared LED light source and a pair of photosensitive diodes detect movement of the beam. A flag at the top of the balance arm controls the amount of light reaching each photosensor. As sample weight is lost or gained, the beam becomes unbalanced, causing the light to strike the photodiodes unequally. The unbalanced signal is fed into the control program, where it is zeroed. This changes the amount of current supplied to the meter movement, causing the balance to rotate back to its null (zero) position. The amount of current required is directly proportional to the change in the mass of the sample.

Although the TGA 2050 has a vertical sample/furnace configuration, the purge gas flow across the sample is horizontal (Figure 2). The purge gas enters the furnace through a side arm and flows directly across the sample located in an open pan. A low volume of inert gas which flows through the balance chamber prevents back diffusion of the primary purge gas or sample effluents into the balance chamber. The furnace incorporates a quartz liner between a bifilar-wound heating element and the sample measurement area. This arrangement results in a small internal volume which further reduces the opportunity for back diffusion of the sample off-gases into the balance chamber by assuring rapid transfer of the gases out the furnace exit port. (This purge flow design is also ideal for evolved gas analysis.) The quartz liner also provides a “protective barrier” between the furnace core and the sample area when performing experiments where corrosive gases such as halogens are present.
Features and Benefits

The TGA 2050 is designed to provide performance usually associated with research-grade instruments, but at a lower price. In combination with a Thermal Analyst Controller, the TGA 2050 is the most cost-effective general purpose TGA system available. Key features and benefits include:

Exceptional Weight Sensitivity, permitting detection and measurement of very small weight gains or losses (less than 1 µg), and the use of small quantities of sample.

Five-Point Temperature Calibration, for maximum temperature accuracy. The operator can choose from one to five calibration standards. Temperature calibration is with either well characterized curie temperature materials, or high-purity metals with well documented melting points.

Wide Dynamic Range, permitting measurement of sample weight losses of up to 1 g.

Controlled Atmosphere, with horizontal flow of gas across the sample. The indirect flow pattern helps assure good sample/atmosphere interaction while minimizing the buoyancy and chimney effects. Purge gases can be inert or reactive. In addition, a positive flow of inert gas from the balance chamber into the furnace protects delicate components against back diffusion of furnace purge gases or sample effluents.

Methods Versatility, facilitating evaluation of organic or inorganic materials. A variety of sample pans in several different sizes is available. Platinum pans are usually preferred because they are easy to clean and do not react with most materials. Ceramic pans are available for samples that might amalgamate or react with platinum and when large capacity is required. Disposable aluminum pans are also available.

Ease of Use: Operator-oriented features include:
• Automated temperature calibration.
• Automated weight calibration.
• Automated taring and determination of sample weight.
• Automated pickup of the loaded sample pan by the balance.
• Automated collection, storage, and display of data.
• One-button operation. Once an experiment has been programmed and the sample loaded, a single keystroke initiates the complete sequence of operating functions – pickup of the loaded sample pan, raising the furnace, starting and running the method, collecting and storing the data, unloading the sample pan, and rapidly cooling the furnace.

Rapid Turnaround: Programmable end-of-run conditions include accelerated furnace cooling using a forced air purge.

Compatibility with Other Techniques: The TGA 2050 is one of many TA Instruments thermal analysis and rheology techniques that complement each other. These modules, together with TA Instruments controllers and software, constitute the most complete, versatile and cost effective systems available for characterizing materials.
Applications

The broad capability of the TGA 2050 for characterizing materials is illustrated by these representative applications.

Multipoint Temperature Calibration

The TGA 2050 uses a single thermocouple to both control the furnace and monitor sample temperature. This simplified design is particularly beneficial to the temperature calibration process, and contributes significantly to the precision and accuracy of the temperature measurements. Because the thermocouple is located adjacent to the sample pan, highly exothermic or endothermic reactions within the sample cause minor fluctuations in the programmed heating rate. While these fluctuations are not of sufficient magnitude to affect the results of an experiment, they do create an easily detected peak in the heating rate derivative. Hence, the melting of commonly used metal standards offers a rapid, convenient way to calibrate the instrument. Temperature calibration can also be achieved using Curie Point standards where the magnetic transition of ferro magnetic materials is characterized as shown in figure 3.

Thermal Stability of Materials

TGA is widely used to determine the thermal stability of materials. Figure 4 shows a series of decomposition curves for four different polymers with different thermal stability profiles: polyvinyl chloride, polystyrene, polypropylene, and polycarbonate. Defining such an important performance property helps design engineers select the best material for a given application. With suitable standard materials, these TGA curves also can be used to identify unknown polymers.

Evaluation of Polymer Flammability

The rapid decomposition and flammability of materials is important particularly when the materials are used in consumer-related applications such as building and transportation products. TGA provides a rapid method for comparing materials with and without flame retardants to determine their effectiveness. Figure 5 shows the TGA profiles for an unretarded polyester fiber versus the material with 6% by weight of added bromine retardant and a new experimental retardant. Both retarded materials exhibit different decomposition profiles than the unretarded material. The 6% Br-retarded sample degrades more rapidly initially than the unretarded sample. Nevertheless, both it and the other retarded sample have larger residues after the first weight loss is complete than does the unretarded sample. After the final decomposition, however, only the experimental retardant sample has any appreciable residue. In addition, the final degradation temperatures and overall degradation process occur at higher temperatures for the experimental retardant sample. These results suggest that the two flame retardants derive some of their respective efficiencies by different mechanisms. The Br-retardant derives its flame resistance from the liberation of hydrogen bromide gas during its thermal decomposition. The liberated gas extinguishes burning and prevents flame propagation. The experimental retardant, on the other hand, probably acts to form fewer flammable degradation products or to form surface barriers that interfere with combustion oxidation processes.

Composition Analysis of Materials

The ability to vary temperature as well as the atmosphere seen by the sample material makes TGA a convenient technique for rapidly evaluating composition. Figure 6 illustrates the results for an oil-extended elastomer. Initial heating up to 500°C in nitrogen produces two weight losses associated with the oil and polymer content in the elastomer. Switching to air and raising the temperature further produces an additional weight loss due to oxidation (carbon dioxide formation) of
the carbon black reinforcer present. The stable residue in air above 600°C is inert filler and ash. Figure 7 shows a similar analysis of coal where a complete proximate analysis (moisture, volatiles, fixed carbon and ash) is determined in a single TGA experiment. In this case, the analysis is automated by the addition of a gas switching accessory and also shortened by using isothermal temperature jumps instead of continuous temperature ramps, making it an ideal quality control test method.

**Predicting the Lifetime of a Product**

Estimating the lifetime of a product requires some form of accelerated testing. TGA decomposition kinetics can provide reliable aging stability information and lifetime predictions within hours, compared with the months required for oven-aging tests. As shown in Figure 8, a sample of polytetrafluoroethylene (PTFE) is heated through its decomposition region at several different heating rates (2.5, 5, and 10°C/min), and the weight loss as a function of temperature is recorded. Then the activation energy is calculated from a plot of the log heating rate versus the reciprocal of the temperature for a constant decomposition level (typically between 1% and 20%). This activation energy can subsequently be used to calculate kinetic parameters such as specific rate constant (k) or half-life times, as well as to estimate the lifetime of the material at a given temperature.

**Oxidative Stability of Edible Oils**

A critical quality parameter for edible oils is their resistance to oxidation. One widely used technique for predicting this resistance to oxidation (rancidity) involves heating the oil in an oven and periodically testing it for weight gain due to oxygen uptake. TGA allows the same measurement to be made continuously, as shown in Figure 9. The oil is heated to the temperature of interest in an inert atmosphere, then the purge gas is changed to oxygen. The time from oxygen introduction to the onset of weight gain is the oxidative stability. Excellent isothermal temperature stability and the ability to detect small weight changes makes the TGA 2050 ideal for obtaining reproducible results.

**Analysis of Evolved Gases**

The weight losses observed in TGA experiments are useful in determining gross compositional information about a material. However, because TGA cannot identify the specific evolved gaseous decomposition products, and because many TGA weight losses involve multiple components, the TGA module is often coupled with another analytical technique that can provide that additional information. A common example is FTIR (Fourier transform infrared) spectroscopy. Figure 10 shows a composite TGA/FTIR plot for polyvinylchloride. A total spectral absorbance (Gram-Schmidt) plot parallels the TGA first derivative curve, illustrating that the observed weight losses are multicomponent and contain degradation products that absorb in the IR spectral region. Individual FTIR spectra taken at selected times are subsequently used to identify specific degradation products. Coupling the TGA to a mass spectrometer provides additional confirmation of the identity of the degradation products, which may be critical when different organic compounds have similar FTIR spectra.
Specifications

Temperature Range: Ambient to 1000°C
Weighing Capacity (max): 1.0 g
Sensitivity: 0.2µg
Balance Accuracy: ±0.1%
Purge Gas Rate: Furnace 90 mL/min; balance 10 mL/min
Temperature Calibration: 1 to 5 points, based on metal or Curie Point standards
Heating Rate: 0.1 to 50°C/min in 0.01°C/min increments
Furnace Cooling: Forced air
Sample Pans: Platinum: 50µL, 100µL
Alumina: 100µL, 250µL, 500µL
Aluminum: 100µL

TA Instruments Commitment

The TGA 2050 is designed and engineered to assure easy, reliable, trouble free operation. It is supported by a full range of services, including an applications laboratory, publications, training courses, technical seminars, applications CD’s, an internet website, and a telephone Hotline for customer consultation. Highly qualified service personnel specializing in thermal analyzer/rheometer maintenance and service are available throughout the world. All of these items reflect TA Instruments commitment to providing thermal analysis & rheology products and related support services that deliver maximum value for your investment.

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