Thermogravimetric analysis (TGA) is an established technique for studying the thermal stability of materials and for determining and understanding their decomposition profiles. While standard operating conditions (10–15 mg samples; 10–40 °C/min heating rates) are commonly used, a growing need exists for more advanced capability. In biological and pharmaceutical studies, analysts are increasingly faced with trying to determine trace materials (e.g., 1–5% of volatiles) in 1–2 mg samples, which are difficult or costly to prepare. This is usually difficult or impossible with current instruments. Many analysts want improved laboratory productivity, while others seek very high heating rates for more specialized analysis, such as studying energetic materials. Naturally, all desire advances in software and operational convenience. Accommodating these diverse needs in a single instrument presents significant technical challenges.

Design criteria

The ability to routinely detect very small weight changes in TGA requires minimizing baseline drift and ensuring that it is reproducible over the temperature range of the experiment. Of the factors contributing to TGA baseline drift, the most critical is maintaining good temperature control in the balance chamber.

For decades, thermogravimetric analyzers have employed resistance wound furnaces, since they deliver the stable and reproducible heating rates (1–40 °C/min) required for quantitative weight change measurements. However, their mass or other design constraints have limited their ability to provide significantly higher heating rates without extensive temperature overshoot. Clearly, a new style of heating is needed to meet the latest and diverse technical challenges. In addition, improvements in automatic sample processing, control, and data analysis software, as well as innovative user convenience features, have also been requirements of a next-generation, research performance TGA as commonly requested by users. These criteria are well met by the Q5000 IR (TA Instruments, New Castle, DE) (Figure 1).

**Instrumentation**

In this TGA, the electrically grounded balance mechanism is housed in a well-insulated, gas-purged, temperature-controlled chamber, which is isolated from the furnace. The furnace uses infrared heating and delivers not only the
heating rate accuracy and precision needed for optimal analytical performance, but also linear heating to 500 °C/min for rapid analysis, and ballistic rates in excess of 2000 °C/min for specialty applications. The furnace includes an integrated electromagnetic coil that simplifies Curie point temperature calibration. The autosampler can process up to 25 samples in platinum, ceramic, quartz, or aluminum pans in a sequential or random manner. The furnace can be readily coupled to a mass spectrometer.

**Discussion**

**Baseline flatness**
The Q5000 IR design has been found to deliver extremely low levels of baseline drift, due mainly to improvements in balance chamber temperature control. Proof of this has been demonstrated in heating ramp experiments from ambient to 1200 °C, where the chamber temperature varied by less than 0.005 °C. Figure 2 shows an overlay of eight repeat dynamic baselines performed at 20 °C/min from 40 to 1000 °C using empty platinum pans. The raw data indicate a worst-case drift of only 6 µg over the temperature range and demonstrate very good repeatability. This level of baseline flatness is nearly an order of magnitude improvement in the state-of-the-art and makes practical the detection of low or submicrogram weight changes.

**Sensitivity for low-level weight change detection**
The presence of unexpected volatiles can be a problem in the production and use of resin systems. When a plastic formulation contains even small amounts of water, solvent, or monomer, it may produce bubbles or imperfections during processing. Figure 3 depicts a high-sensitivity moisture determination in a 2.4-mg sample of polyethylene terephthalate (PET) bottle-stock. The raw data show evidence of 5.2 µg (0.21%) of moisture, and indicate that lower amounts could easily be detected.

A second good example of sensitivity and also resolution appears in Figure 4, an analysis of a commonly used over-the-counter (OTC) analgesic. The entire tablet (active ingredient, binder/fillers, and coating) was ground and a 0.543-mg sample was heated at 10 °C/min from ambient to 400 °C. Individual analysis of the core tablet and the outer coating reveals that the first and third weight losses are from the core tablet, and the second loss is due to decomposition of the coating. Of special note was the detection of less than 6 µg of adsorbed moisture.

**Heating rate reproducibility**
An important need in all TGA experiments is stability and reproducibility in heating rate. Figure 5 displays a triplicate analysis of the decomposition of a proprietary elastomer that produced a carbon black determination within 0.1% of the expected value. The method involved several
isothermal and ramp segments, the latter performed first at 500 °C/min in nitrogen to decompose the elastomer, followed in turn by an equilibration at 330 °C, an automatic gas switch to air, and finally a second 500 °C/min ramp to 850 °C. The overlay of three runs provides strong proof of the stability and reproducibility of the infrared furnace, even at an elevated heating rate.

Another demonstration of the heating rate stability and reproducibility from the infrared furnace has been from the well-known Detroit Diesel Soot Test. This involves heating used diesel engine oil to decomposition, initially in nitrogen and then in air, to make a measurement of the remaining “soot.” Data reproducibility of better than 0.1% has been reported from different laboratories in this analysis.

**Capability for rapid analysis**

Figure 6 shows two plots of the decomposition of a 40% calcium carbonate-filled polypropylene sample at heating rates of 40 °C/min and 500 °C/min, respectively. In the latter experiment, the analysis time was reduced (7x) with little or no loss in resolution. This demonstrates that the combination of rapid heating, forced air cooling (<10 min from 1200 to 50 °C), and an automatic sample processor can deliver significantly improved productivity in routine sample analysis where the resolution can be maintained at the higher heating rates. The furnace’s capability to generate very high ballistic heating rates (>2000 °C/min) has drawn high interest from scientists wishing to use the TGA to simulate actual combustion processes (i.e., propellants, tobacco, pyrotechnics).

**Automatic sample processing**

The capability for automatic sample processing, analysis, and data reduction in TGA experiments is a necessity in busy thermal analysis laboratories. It permits improved productivity, minimizes operator error, and releases staff for other tasks. Time-dependent integrity of some materials while awaiting analysis has been an issue and is addressed in two ways. The first is the ability to preweigh samples awaiting analysis and then compare the value with the weight recorded at the actual time of analysis. This allows detection of any sample weight change while in the autosampler tray. The second and more comprehensive solution to the issue of environmentally sensitive samples is encapsulation in aluminum pans, which are opened just prior to analysis. A key advance in this autosampler is in the mechanism for opening the sealed pan. A stainless steel “punch” is used that does not “pierce” the lid and thus
avoids contact with the sample. This eliminates the possibility of cross-contamination of other samples awaiting analysis.

Good laboratory efficiency requires that the instrumentation be maintained properly calibrated and ready for use. This takes time and is often postponed. A solution is now available in which the autosampler and Advantage software (TA Instruments) combine in a way that automatically ensures the operational readiness of the analyzer. This allows the user to schedule key performance tests such as weight and temperature calibration, verification, and system diagnostics on the TGA and automatically receive post-test notification of the results by e-mail. Further test sequences can be automatically performed based upon the data obtained. These operations can be scheduled at quiescent periods, such as overnight or on weekends. In addition, a separate software program permits the analyzer to assist the user in complying with the U.S. FDA regulations as specified in Document 21 CFR 11.

Summary
The above design features and many others permit the Q5000 IR to set new standards in performance and user convenience in thermogravimetric analysis.

References

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