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THERMAL ANALYSIS

application note

High Sensitivity Volatiles Analysis by TGA

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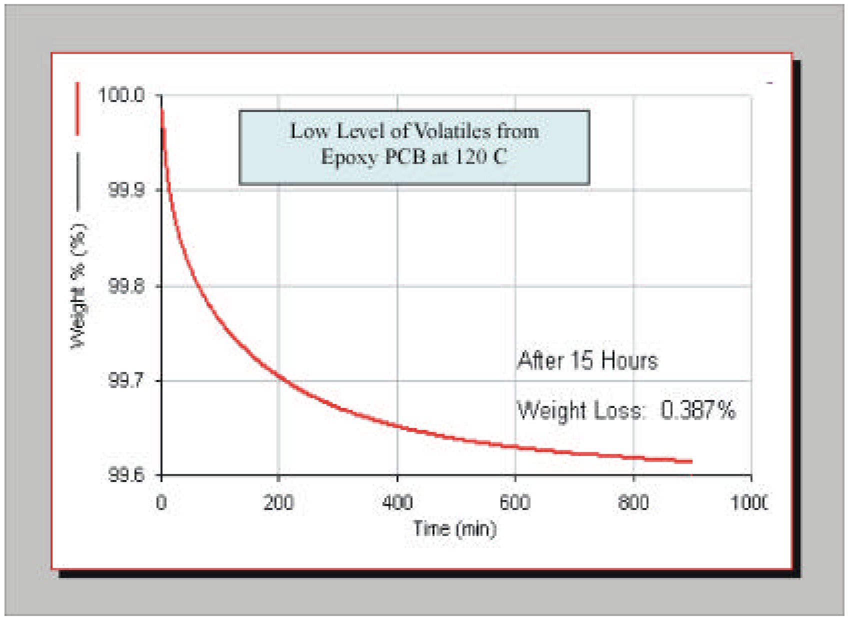
Thermogravimetric analysis (TGA) is generally used to study large decomposition or weight loss events associated with polymers, elastomers, electronics, pharmaceuticals, foods and other materials. However, there are cases where it is desired to measure very small or low levels of volatiles evolved from a sample during heating or holding under isothermal conditions. On a large production basis, even a small level of volatiles (e.g., less than 1%) can have a major impact on the processing of the polymer. It has been known that low levels of volatiles can affect the injection molding or blow molding processing of polymers. The shelf-life and cosmetic properties of pharmaceutical materials are strongly affected by the low levels of moisture contained in the compounds. Small variation in moisture content can have a significant affect upon these properties.

It is desired to have a high performance TGA instrument which is capable of reproducibly measuring such small weight loss events, which are on the order of 1% or less. The Pyris 1 TGA is a state-of-the-art instrument which provides the necessary high degree of performance for the quantitative measurement of small levels of volatiles, associated with polymers, electronics, pharmaceuticals and foods. The Pyris 1 TGA offers the following features to achieve its high performance operation:

Figure 1. Measurement of low level of volatiles from epoxy printed circuit board at 120 C using the Pyris 1 TGA.



- Low mass, ultra-light balance for ultra-low noise and outstanding sensitivity
- Separate thermostatically controlled temperature environment of balance to provide highest possible stability
- Iris shutter assembly to isolate balance chamber from sample/furnace
- Automated ion stream to eliminate troublesome static effects
- High performance heat/sensor furnace technology
- Reduced furnace volume for more efficient switching of purge gases and elimination of oxygen during pyrolysis
- Autosampler accessory for unattended operation
- Acupik accessory for better handling of volatile samples

PerkinElmer™ instruments.

Figure 2. High sensitivity TGA measurement of volatiles from PET resin at 130 C.

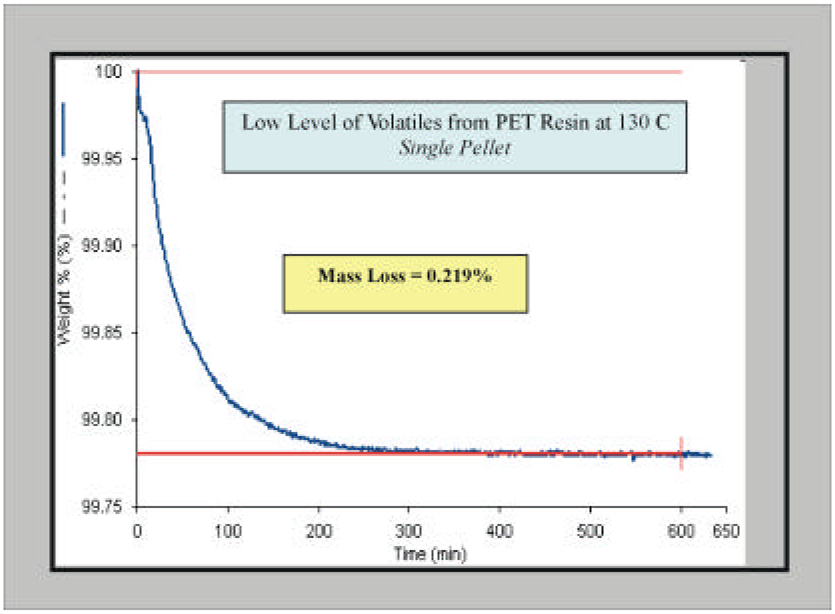
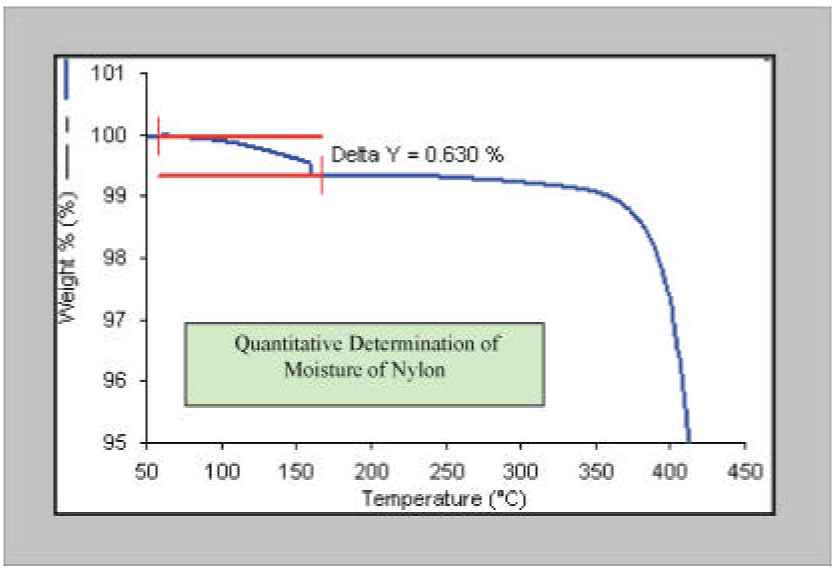


Figure 3. High sensitivity TGA measurement of water loss from nylon 6.



without uncontrolled and unmeasured weight loss while waiting to run

- Environmental autosampler carousel to avoid exposure of queued samples to ambient air and moisture

Shown in the Figure 1 is the measurement of the very low level of volatiles evolved from an epoxy printed circuit board (PCB) as measured by the Pyris 1 TGA under isothermal conditions at 120 C. The sample was held for 15 hours under the 'gentle' isothermal hold and the total weight loss of the volatiles was only 0.387%.

In Figure 2, the low level of volatiles evolved from a sample of polyester (PET) feedstock using the Pyris 1 TGA were measured at a relatively low temperature of 130 C. The mass loss observed after 10 hours was only 0.219% for the polyester resin.

When dealing with immense quantities of polymer for injection molding or spinning operations, small mass losses, as shown in the previous example, become quite important. Small quantities of absorbed water or moisture can have a significant effect upon PET resin due to hydrolytic degradation and can affect the spinning of fibers or the production of films.

Nylon represents another example where small amounts of absorbed water can have a major effect upon the end-properties of the polymer. Nylons are hydrophilic and will absorb moisture from the immediate environment. Because of the plasticizing effects of the water, the Tg of nylon 6 can shift by over 60 C between totally 'wet' nylon and the completely dry polymer. The stiffness (modulus) and impact properties of nylon can vary dramatically at room temperature as a function of the material's moisture content. Figure 3

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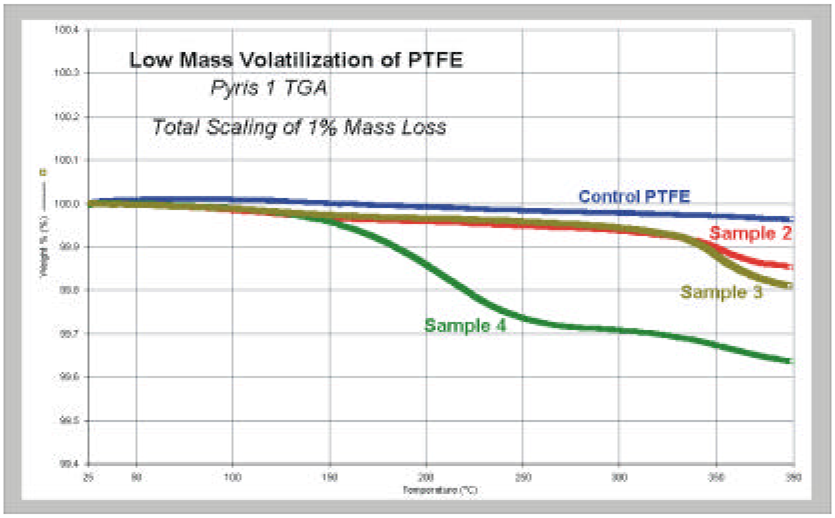
Figure 4. High sensitivity TGA measurement of loss of volatiles from PTFE resins.



shows the quantitative determination of the moisture content of nylon (only 0.630%) using the Pyris 1 TGA.

The results presented here demonstrate that a high performance TGA instrument, such as the Pyris 1 TGA, is required to measure the very small mass losses associated with 'wet' nylon samples.

In the next example, a manufacturer of components made from PTFE resins needed to quantify the low level of volatiles associated with the resins. Practical experience demonstrated that the amount of these low temperature volatiles had a major effect upon the production of the PTFE components.

Shown in the Figure 4 are the Pyris 1 TGA results generated on 4 different PTFE resins.

The results show that the control PTFE resin evolved very little volatiles (less than 0.03%) up to 390 C while Sample 4 yielded 0.29% weight loss between room temperature and 300 C. Although 0.29% may not seem like an overly significant amount, it was found that this was unsatisfactory and PTFE Sample 4 was unsuitable.

Summary

The Pyris 1 TGA provides outstanding performance for the quantitative measurement of low levels of volatiles evolved by materials including polymers, electronic components, elastomers, pharmaceuticals and foods. The volatiles can have a major effect upon the end-use and long-term properties of materials.

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