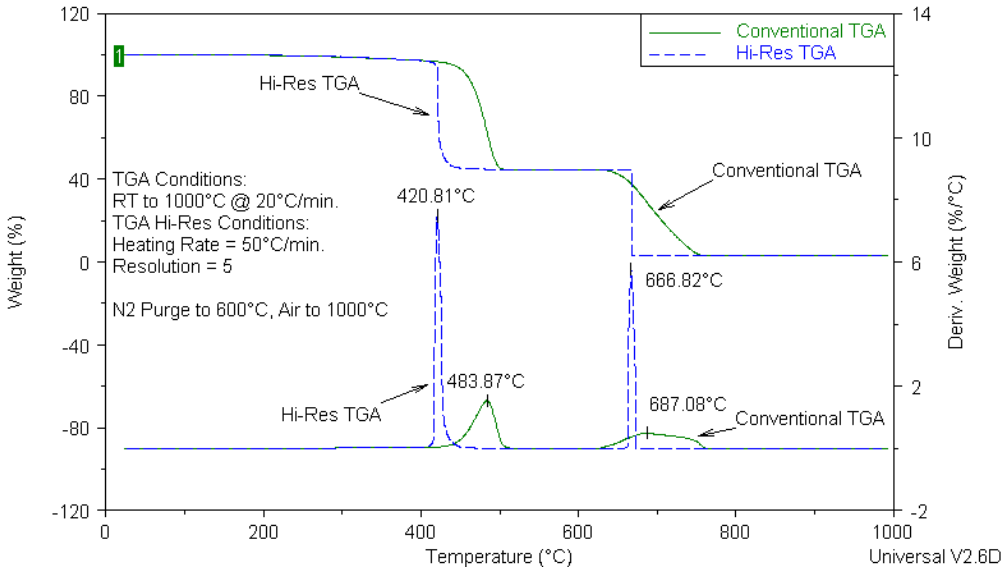
THERMAL SOLUTIONS

Characterization of EPDM Rubber by TGA and Hi-Res‹ TGA

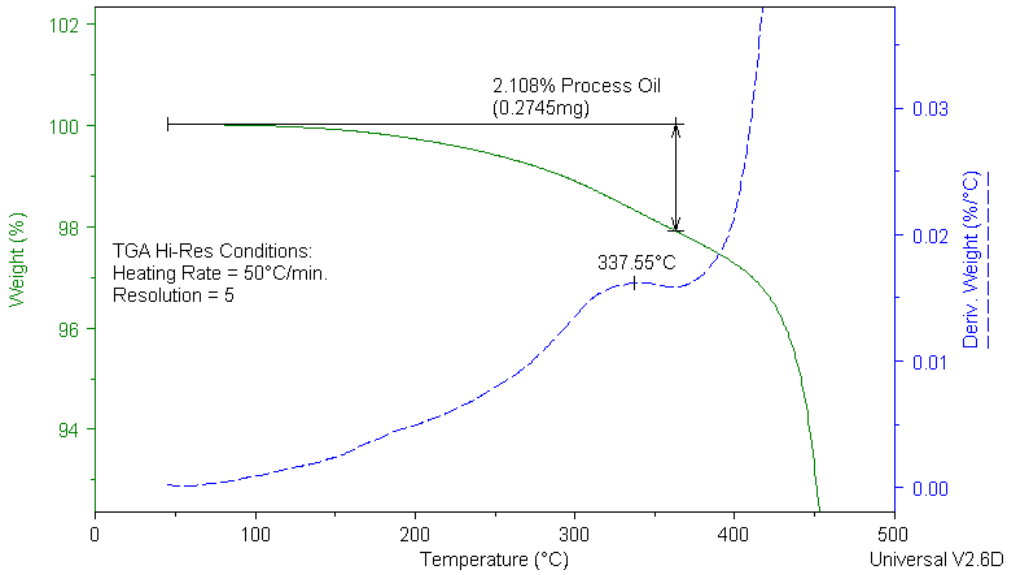
Figure 1



Rubber compounds are widely used in many applications because of the properties they exhibit. The properties of the material are highly influenced by the composition of the compounded material. Typically, the rubber compound will contain extenders (which are light to heavy oil(s), a polymer(s), carbon black, and filler. These components can be quickly quantified using thermogravimetric analysis (TGA). As the rubber compound is heated in a controlled environment, the components evolve. The oil evolves first followed by the polymer. In a nitrogen atmosphere, all of the oil and polymer have evolved by about 600°C. At that point, the TGA is programmed to switch to an oxidizing atmosphere (typically air). With continued heating (or an isothermal hold), the carbon black begins to burn off and the amount of carbon black is quantified. The filler content will be the residue remaining at the end of the scan.

In some compounds, calcium carbonate is used as an additive in rubber compounds. Calcium carbonate exhibits decomposition to calcium oxide with a weight loss in the region of 700°C. To quantify the carbonate additive, the scan would continue to be heated to approximately 900°C in a nitrogen atmosphere. A weight loss in the 700°C to 800°C region under nitrogen would indicate a carbonate material is present. Stoichiometric calculations can be done to ascertain the quantity of calcium carbonate present based on the weight loss amount observed. After reaching 900°C the TGA would be programmed to cool in nitrogen to 500°C and then switch over to an oxidizing atmosphere to observe the burn off of the carbon black material and resultant residue.

Figure 2



Thermogravimetric Analysis (TGA) measures the amount and rate of change in sample weight as a function of temperature or time. TGA experiments use linear heating rates to detect weight loss events. Overlapping or unresolved weight loss events may not be well resolved using conventional TGA. High Resolution TGA (Hi-Res™) is an extension to conventional TGA whereby the heating rate is varied as a function of the rate of sample weight loss. This approach allows the use of high heating rates during no weight loss regions, then automatically reduces the heating rate during a weight loss transition. This often yields faster experiment times, improved separation of overlapping or poorly defined weight loss events, and sharper derivative peaks.

Figure 1 above shows a sample of an EPDM rubber compound run by both techniques. Note how the polymer decomposition (in nitrogen) followed by the carbon black oxidation (in air) weight loss events are better resolved by using the high resolution TGA technique than by conventional TGA. Also, as expected, due to the reduced heating rate during the weight loss, the derivative peaks have shifted to lower temperatures.

Figure 2 is an expanded portion of the high resolution TGA parent weight loss curve showing the small amount of process oil lost during the lower temperature regions before the major polymer decomposition occurs.

This example clearly shows how TGA can be used to characterize rubber compounds. This example also shows that Hi-Res™ TGA can be used to improve weight loss resolution, often in a shorter amount of time, compared to standard TGA.