APPLICATION BRIEF

Thermogravimetric Analysis – GC Mass Spectrometry

TG-GC/MS Technology – Enabling the Analysis of Complex Matrices in Coffee Beans

Introduction

The combination of a thermogravimetric analyzer (TGA) with a mass spectrometer (MS) to analyze the gases evolved during a TGA analysis is a fairly well-known technique. In cases of complex samples, TG-MS often results in data in which it is nearly impossible to differentiate gases that evolve simultaneously.

Combining TGA with gas chromatography mass spectrometry (GC/MS) allows for a more complete characterization of the material under analysis and precisely determines the products from the TGA.

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Experimental

This analysis was performed on a PerkinElmer® Pyris™ 1 TGA using alumina pans and the standard furnace. The instrument was calibrated with nickel and iron and all samples were run under helium purge. Heating rates varied from 5 to 40 °C/min, depending on the sample under test. The furnace was burned off between runs in air. Samples were approximately 10-15 mg. Data analysis was performed using Pyris 9.0 Software.

During the TG-GC/MS analysis, the PerkinElmer Clarus® 680 C GC/MS was used. A 0.32 mm I.D. deactivated fused-silica transfer line was connected to the GC injector port. The transfer line was heated to 210 °C and connected to the Elite™-1ms capillary GC column. In both cases, data analysis was performed using TurboMass™ GC/MS Software.

Results

In this TG-GC/MS application, coffee beans were analyzed. The TGA resulted in a complex thermogram with many different transitions (Figure 1).

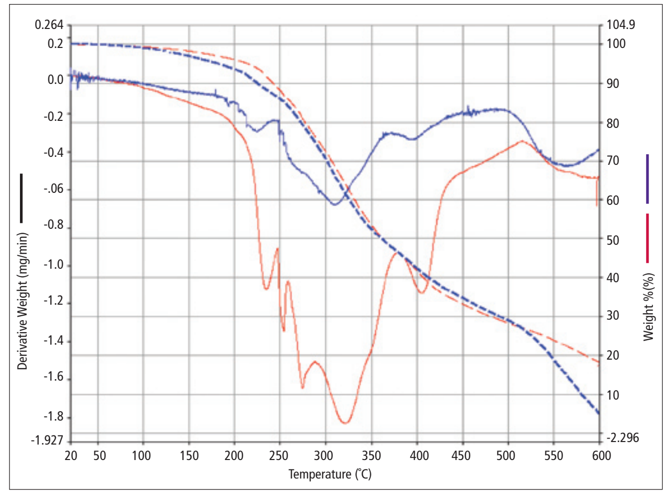

Figure 1. Resultant thermogram from the analysis of coffee beans. The blue curve is unroasted beans from Africa; the red curve is unroasted beans from Sumatra. The weight loss and the first derivative are shown.

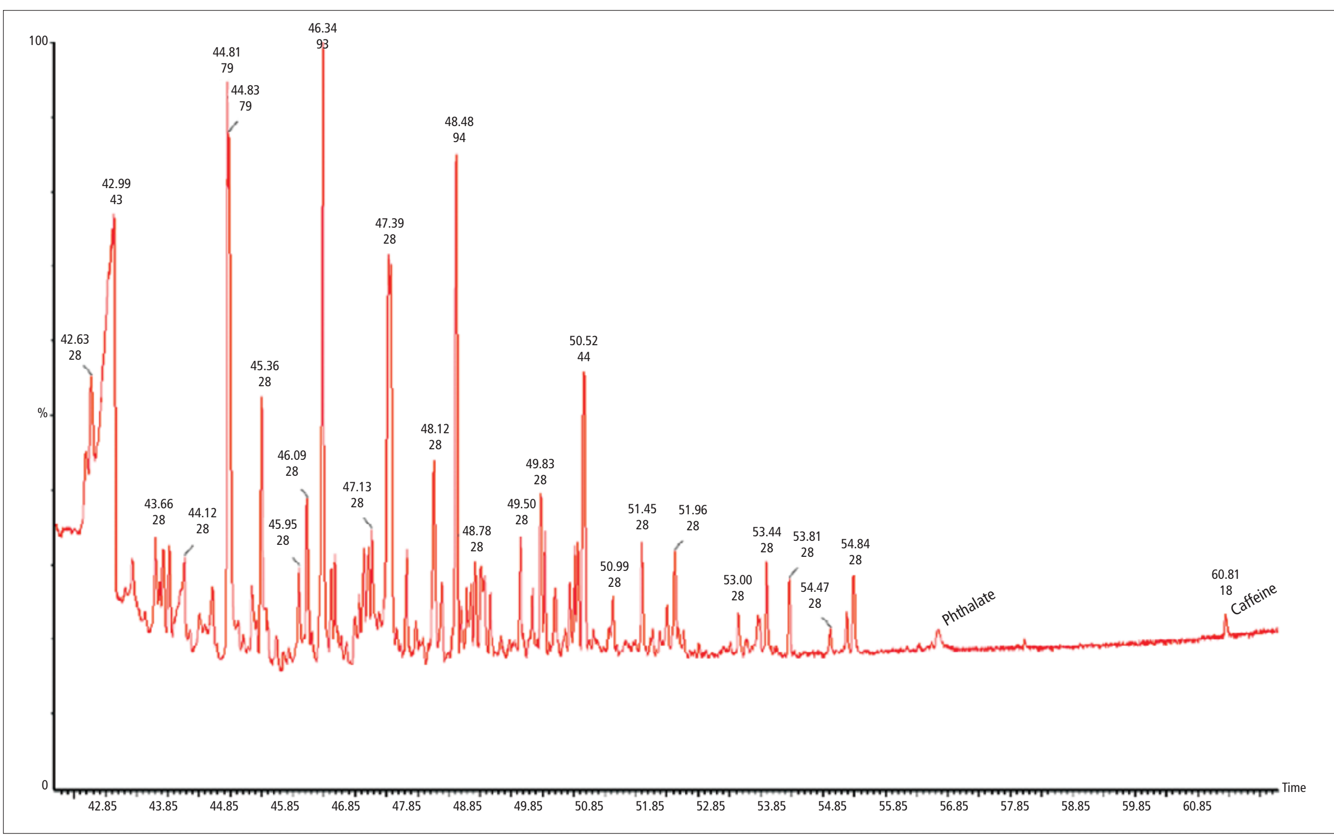

Figure 2. The GC/MS data resultant from the TGA of African coffee beans.

Complex data was expected as coffee beans are known to contain many different compounds. As a result, it was determined that the evolved gas would likely be too complex for TG-MS, thus TG-GC/MS was determined to be a better approach for this matrix.

The goal of the analysis was to search the complex data for two compounds that would be expected in a coffee sample, caffeine (*m/z* 194) and phthalates (*m/z* 149). The caffeine is obviously present in coffee, while the phthalates were a possibility as a result of storage in and contact with plastics. The resultant GC/MS data is shown in Figure 2 (Page 2), demonstrating a very complex chromatogram.

A search for significant peaks resulted in a spectral match for a phthalate (56 minutes), while a search for *m/z* 194 resulted in a spectral match for caffeine at 60 minutes. As expected, the TGA of coffee beans results in the simultaneous evolution of a large number of gases – TG-GC/MS is able to resolve many of these compounds enabling deeper investigation.

Conclusions

TGA analysis allows quantification of the weight loss of a material at specific temperatures. MS increases the power of the technique by providing the ability to identify the species evolved during thermal analysis. TG-GC/MS adds the additional capability of chromatographic separation of co-evolved gases. While not realtime, the improved separation by the GC/MS makes data interpretation easier than TG-MS. This allows the separation of fairly complex mixtures with minimal sample preparation by using the TGA to volatilize components.

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