

High Gravimetric Sensitivity TGA for Volatiles Determination in Nylon Airbag Fabric

Keywords: Automotive, Thermogravimetric Analysis, fog test, textile, volatiles

TA397

Components used in the passenger compartment of a car or truck contain semi-volatile materials which can fog the windows. Upholstery, dash and door coverings, nobs and switches, carpet, electrical components, lubricants and adhesives all contain semi-volatile materials. On a hot day the semi-volatile materials will vaporize and then upon cooling condense and fog the windows. Vehicle owners are annoyed by this fog or haze on the windows. Auto manufacturers and their suppliers take significant care to minimize this problem by selecting components with low fogging tendency. A laboratory fog test has been used by the industry for several decades to quantitate the tendency of different materials to fog glass. But due to the testing complexity and cost, a simpler analytical approach is needed. Gas chromatography (GC), GC/Mass Spec, and thermogravimetric analysis (TGA) have all been evaluated as a proxy for the fog test with differing degrees of success. High gravimetric sensitivity TGA is a viable alternative. The TA Instruments Discovery TGA provides the necessary high gravimetric sensitivity for determination of extremely small weight changes and detecting minute differences between samples when quantitating the presence of semi-volatile material.

The modern car can have over ten airbags which are concealed in the passenger compartment. The airbag contains many different components with Nylon fabric being used to construct the bag. Six samples of Nylon airbag fabric with known fogging tendency were evaluated with a simple TGA method. Three samples were low fog and the other three were high fog. Since Nylon is hydrophilic, the TGA method needed to differentiate the semi-volatile material from the adsorbed water. This was accomplished by initially drying the sample at a low temperature in the TGA, and then heating to a higher temperature to vaporize and quantitate the semivolatile material. Since the water and semi-volatile materials are trapped in the Nylon fiber, isothermal periods at the two temperatures were needed to resolve the water and the semivolatiles based on their volatility. A ramp – isothermal – ramp - isothermal TGA method was used where the isothermal temperatures were determined from a preliminary TGA linear ramp experiment. A 10 minute isothermal period at 80 °C was sufficient to remove the water, and a 10 minute period at 160 °C was used to evolve the semi-volatiles. The semi-volatiles were quantitating based on the weight change between the ends of the two isothermal periods.

The gravimetric sensitivity for a TGA is also enhanced by using large samples. Since the gravimetric drift and baseline noise of a TGA are constants, using a large sample increased the gravimetric sensitivity. The air bag fabric was punched into 4 - 5 mm disks which were then stacked on the TGA pan to provide a 65 mg sample. The weight change between the ends of the two isothermal periods is easily quantitated with a 65 mg sample, but would be non-detectable using a 6 mg sample.

The six Nylon fabrics were run on a Discovery TGA in a nitrogen atmosphere using the following ramp – isothermal – ramp – isothermal method.

- 1) Ramp 10.00°C/min to 80.00°C
- 2) Mark end of cycle 0
- 3) Isothermal for 10.00 min
- 4) Mark end of cycle 0
- 5) Ramp 10.00°C/min to 160.00°C
- 6) Mark end of cycle 0
- 7) Isothermal for 10.00 min

Typical results for a high fog sample are shown in Figure 1.



Figure 1: Discovery TGA Results for a High Fog Nylon Airbag Fabric

The content of semi-volatile material was quantitated as the weight change between the end of the two isothermal periods, i.e. Points B and C in Figure 1. The moisture content can also be quantitated as the difference in the initial sample weight to the end of the first isothermal period, i.e. Points A and B in Figure 1. For the high fog fabric in Figure 1 (i.e. High Fog #2), a minute loss of 25.5 µg of semi-volatile material was observed which for the 65 mg sample size equates to 0.0382 wt% or 382 parts-per-million (PPM). Results for all six fabric samples and an empty pan blank are shown in Figure 2.



Figure 2: Discovery TGA Results for Nylon Airbag Fabric and Empty Pan Control

The results are tabulated in Figure 1 and an empty pan control is compared against a typical air bag sample in Figure 3. The high fog fabrics have 382 to 453 PPM of semi-volatile materials as compared to the lower 291 to 334 PPM for the low fog fabrics. So in conclusion, the high fog fabrics have at least 48 PPM more semi-volatile material as compared to the low fog fabrics.

Sample Identification	Semi-Volatiles, PPM
High Fog #1	453
High Fog #2	382
High Fog #3	387
Low Fog #1	334
Low Fog #2	304
Low Fog #3	291
Empty Pan Control	22 est.



Figure 3: Comparison of Empty Pan Control with Typical High Fog Air Bag Fabric

The empty pan control has a 1.4 µg difference between the ends of the two isothermal periods. Assuming a 65 mg sample size this equates to a 22 PPM systematic bias in the quantitated results for the fabric samples. In other words, the results in Table 1 are overstated by 22 parts per million. Although the empty pan blank run can be used to correct for the bias, the correction will not change the conclusion that the high fog fabrics have at least 48 PPM more semivolatile material as compared to the low fog fabrics.

SUMMARY

The TA Discovery TGA provides the high gravimetric sensitivity needed to quantitate differences in the fogging tendency of air bag fabrics. The concentration of the semi-volatiles materials in the airbag fabric was easily quantitated at several hundred parts-per-million, and high fog Nylon fabrics contained at least 48 PPM more semi-volatile material than did the low fog fabrics.

For more information or to place an order, go to http://www.tainstruments.com/ to locate your local sales office information.

ACNKOWLEDGEMENTS

This note was written by Charles Potter, Ph.D., TA Instruments.