SII NanoTechnology Inc.

TGA AS PROBLEM-SOLVING TOOL: COMPOSITIONAL ANALYSIS OF PAINTS

Problem

A quality assurance chemist, working for a company that produces paints, has a need to determine the composition of the multi-component paints. Standard thermogravimetric analysis (TGA) yields continuous and severely overlapping weight losses and the data cannot be easily analyzed to provide compositional information on the paints. The chemist desires an easy to use test which provides a high degree of resolution on the paints in order to accurately determine the compositional characteristics of the materials with a high degree of reproducibility.

Solution

Auto stepwise isothermal TGA provides the best possible means of separating or resolving closely spaced decomposition or weight loss events. The approach taken is to heat the sample at a constant rate (generally 40°C/min) until the rate of mass loss becomes large and exceeds a user-defined 'entrance' threshold value. The TGA instrument automatically holds the sample under isothermal conditions until the rate of weight loss becomes negligible or less than a user-defined 'exit' threshold value. The TGA then resumes heating the sample at a constant rate until the next significant weight loss event is encountered. In this manner, the auto stepwise approach separates out overlapping decomposition events to the highest possible degree of resolution.

The Seiko Instruments TG/DTA provides the auto stepwise capability as a standard feature. The instrument also offers the following desirable features:

- High sensitivity
- Stable performance
- True horizontal purging
- Simultaneous DTA measurements
- Capability of direct conversion of DTA to DSC output
- Direct temperature calibration using high purity metal standards
- Up to 20-point, semi-automated temperature calibration
- State-of-the-art, add-on robotic capability
- Windows NT operation

In this study, the compositional characteristics of a paint sample were analyzed using the auto stepwise approach. The following experimental conditions were utilized:

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Instrument: Seiko TG/DTA
Mode of operation: auto stepwise
Heating rate: 40°C/min
Entrance threshold: 275 μg/min
Exit threshold: 20 μg/min
Sample mass: 14 mg
Sample container: open aluminum pan
Purge gas: nitrogen at a flow rate of 200 mL/min

The paint sample was quickly injected into the pan with a pipette and the purge gas was turned off to prevent evaporation of the solvent in the paint as the sample and balance equilibrated. After about 30 seconds, the purge gas was activated and the run was immediately started.

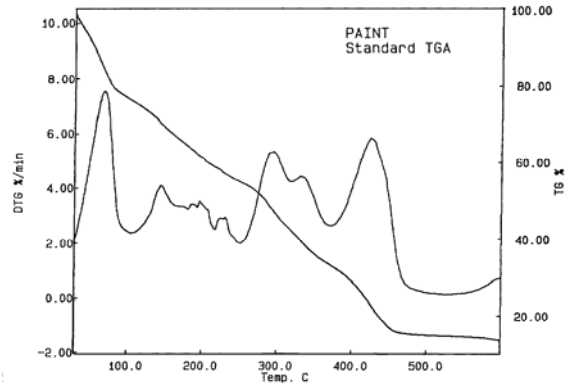

Figure 1

Displayed in Figure 1 are the results obtained by standard TGA on the paint sample as the material is heated at a rate of 20°C/min. The plot shows the percent mass (TG %) and the derivative or rate of mass loss (DTG %/min) as a function of sample temperature. A continuous weight loss is obtained from room temperature up to 500°C. The derivative trace shows numerous peaks indicating that the number of

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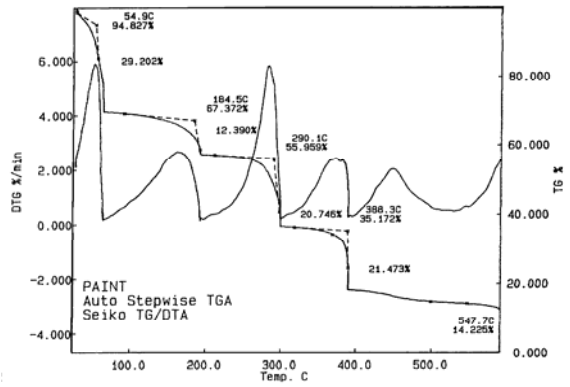

Figure 2

severely overlapping weight loss events take place as the paint sample is heated. However, it is impossible to quantify the individual mass losses which are obtained because of the severe overlapping generated with standard TGA.

The auto stepwise approach was employed and the results obtained from this technique on the paint sample are shown in Figure 2. Excellent resolution of the various mass loss events is produced using the auto stepwise TGA mode of analysis. The complex, multi-component nature of the paint material is clearly evident in the auto stepwise results. The first weight loss corresponds to the loss of solvent from the paint and this represents 29.2% of the total mass of the paint. Higher temperature weight loss events are observed at 184.5°C (12.4%), 290.1°C (20.8%) and 388.3°C (21.5%). The residue remaining at 550°C is 14.2%.

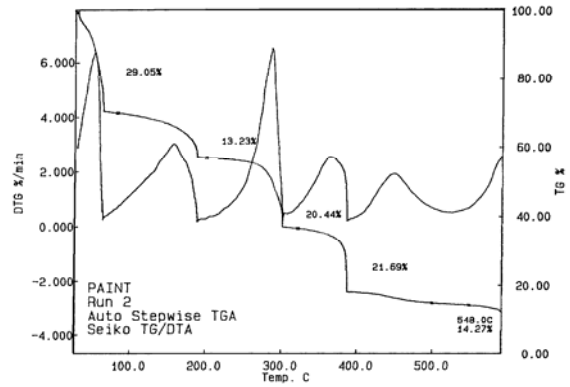

Figure 3

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In order to establish the reproducibility of the technique, a fresh sample of the paint was analyzed under the same experimental conditions and the results are displayed in Figure 3. The data from the second experiment agrees very well with that obtained from the first. A direct overlay of the duplicate runs on the paint sample is shown in Figure 4. Excellent reproducibility is observed.

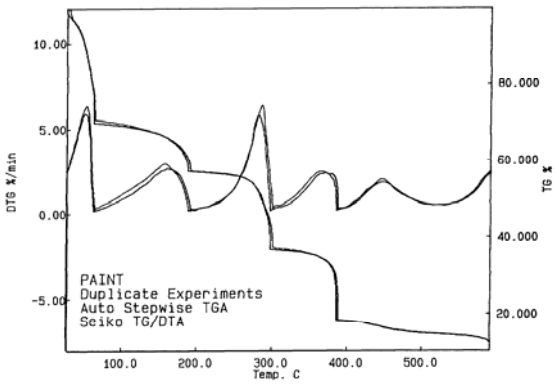

Figure 4

Summary

The auto stepwise TGA approach provides the highest possible degree of resolution or separation of closely spaced decomposition events. The technique permits the quantitative compositional characterization of multi-component materials, such as paints. The Seiko TG/DTA offers the auto stepwise mode as a standard feature.

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