SII NanoTechnology Inc.

OPERATING TIPS FOR THE SEIKO TG/DTA

- **Sample mass**. It is recommended that a mass between 10 and 20 mg be used for the analysis of most samples. If a sample tends to foam significantly during heating, a smaller mass should be used to give better results. For samples which yield a small weight change during heating, a larger mass should be used (e.g., 20 to 40 mg). For the study of very small weight losses (e.g., 1%), a large mass, on the order of 40 mg, should be used since this larger mass enhances the signal to noise ratio.
- **Heating rate**. For most experiments, a heating rate of 20°C/min is recommended. Slower heating rates will give better resolution between successive weight loss events. The degradation onset temperature will be affected by the selected heating rate, as described in the 'Kinetic Effects' section.

- **Purge gas**. The TGA instrument must be purged in order to give meaningful results. It is important to sweep away the generated sample volatiles with an adequate purge flow rate. The standard recommended purge gas is nitrogen at a flow rate of 200 mL/min. When the purge gas is activated, the flow should be monitored for one or two minutes to ensure a stable flow rate has been achieved. When wishing to burn off the polymer or to perform complete compositional analysis, it is recommended to switch from nitrogen to an oxidizing atmosphere (air or oxygen) at temperatures between 600 and 1000°C using the Gas Switching Accessory. The flow rate of the oxidizing purge should be the same as for the nitrogen, which is recommended to be 200 mL/min. For samples which generate viscous, organic volatiles (such as elastomers with oil extenders), the use of the air or oxygen purge above 600°C is always recommended for each experiment. When performing normal TGA experiments, ensure that the special purge port located on the rear of the TG/DTA module is blocked off with a sealed tube (see page B-1 of the TG/DTA6000 operator's manual).
- **Sample pans**. Open aluminum pans can be used at temperatures below 600°C. Above 600°C, open platinum pans must be utilized. When using platinum pans above 1200°C with the high temperature TG/DTA, alumina powder should be sprinkled onto the sample and reference platforms before the pans are placed onto the balance.
- **Sample preparation**. The TGA results are sensitive towards the particular configuration of the sample, in many instances. Powdered samples can give different results as compared to a solid chunk because of the larger surface to volume ratio associated with powders. It is important to keep the sample configuration as consistent as is possible when performing quality assurance measurements or performing comparative experiments. When analyzing samples such as plaques, films, or elastomers, very good

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

consistency can be obtained by using a cork borer with the same diameter as the sample pan to extract the samples.

- **Counterbalancing**. For most samples, which lose most of their weight during a TGA experiment, counterbalancing the reference side is not necessary. For samples which yield a small weight loss (e.g., less than 20%), then counterbalancing the reference side with an inert material (such as alumina powder) to match the mass of the sample is recommended to provide enhanced data. When counterbalancing, add the counterbalancing inert material to the reference before zeroing.
- **Auto Stepwise Isothermal Settings**. The use of the auto stepwise mode of operation provides excellent separation or resolution between overlapping decomposition events. It is recommended to use an initial sample mass of approximately 12 mg. A heating rate of 20 or 40°C/min can be used in the auto stepwise mode. The recommended parameters for the auto stepwise operation are: Start Threshold of 300 µg/min and Stop Threshold of 20 µg/min.
- **DTA Baseline Subtraction**. When attempting to study or analyze small transitions in the simultaneous DTA signal, enhanced results can be obtained by performing a baseline experiment (i.e., two empty pans at the same heating rate) and then subtracting the baseline DTA results from the sample DTA signal.
- **Kinetic Effects**. It should be remembered that the TGA decomposition data is strongly affected by the heating rate that is selected. Decomposition is a chemical reaction which is time dependent. The TGA heating rate also has a time dependency (B = dT/dt) which significantly affects the TGA decomposition onset temperature. Slower heating rates will move the decomposition onset temperature to lower values. It is important to keep this in mind when comparing results from different TGA experiments. The effects of the heating rate with regards to the TGA onset temperature can be used to assess the lifetime of the material or to establish the sample's degradation kinetics using the Seiko TGA Kinetics software.
- **Calibration**. The simultaneous DTA signal provided with the Seiko TG/DTA permits direct temperature calibration to be performed using high purity metal standards. Indium is the preferred, ASTM-endorsed melting standard with an onset melting temperature of 156.6°C. Tin can be used as a secondary melting standard (M.P. = 232.0°C), but tin can only be heated once and then it must be discarded. Do not allow tin and indium to come in contact with each other since they will alloy rendering the calibration erroneous. The TGA instrument should be calibrated for temperature at the same heating rate, and under the same purge conditions that will be utilized to run the samples.

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- **Special or corrosive purge gases**. When attempting to analyze samples using purge gases other than nitrogen, oxygen, air, helium or argon, the special purge gas should be connected to the special purge port inlet on the back of the TG/DTA module. This introduces the special purge gas directly into the furnace and bypasses the balance system and electronics. A nitrogen or inert gas purge should still be placed into the standard purge port in order to protect the balance assembly and electronics. A listing of the acceptable corrosive purge gases is given on page 6-5 of the TG/DTA operator's manual.
- **Samples which boil over or foam excessively**. For samples which tend to boil over or foam significantly, use the special 5 mm deep containers and keep the sample mass as small as is possible.
- **Inactive TG/DTA instrument**. If the TG/DTA module has not been operated or has been left idle for more than a week, moisture and/or dust can accumulate on the balance beams. If the instrument has been idle, it is recommended to preheat the TG/DTA to 200°C with the purge gas activated before analyzing any samples to bake off the moisture and dust.

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