Analytical Answers, Inc.

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THERMOGRAVIMETRIC ANALYSIS (TGA)

Thermogravimetric Analysis (TGA) is a technique which monitors changes in the mass of a sample as a function of time and temperature. The sample, usually a solid but liquids may be analyzed as well, is placed in an inert quartz sample vial inside a low volume, high temperature computer controlled oven. The quartz sample vial is suspended from one arm of a high sensitivity electrobalance, which is monitored along with the oven temperature during a TGA run. AAI's advanced TGA system also provides computer control of multiple reactant gases during the run, monitoring mass changes as different reactant gases are introduced. The entire system may also be run under vacuum, for low pressure studies of gas evolution from materials as a function of temperature.

AAI's advanced TGA system can be used to analyze samples up to ~10g, although sample masses on the order of 0.1g to 1g are typical. The sensitive electrobalance has a precision of 1ppm, which allows detection of a mass change of 0.000001g during an analysis.

TGA Applications Include:

1. **QUANTITATIVE LOADING OF INORGANIC FILLERS IN POLYMERS**

   Glass Fibers
   Metals and Metal Oxides
   Calcium Carbonate
   Talc
   Kaolin
   Carbon black (via computer controlled N_2/Air switching)
   Inorganic Pigments

2. **ORGANIC FILLER AND POLYMER BLENDS**

   Aramid fibers in polymers
   Oils in rubber
   Plasticizers in polymers

3. **RESIDUAL SOLVENTS IN PHARMACEUTICALS**

4. **LOW MOLECULAR WEIGHT MONOMERS IN POLYMERS**

5. **EVALUATION OF FLAME RETARDANTS IN POLYMERS AND FABRICS**

6. **MOISTURE LEVELS AND DRYING CHARACTERISTICS IN POLYMERS AND INORGANIC POWDERS**

7. **GAS ADSORPTION IN ZEOLITES AND CATALYSTS**

8. **OXIDATIVE STABILITY OF SOLIDS**

9. **INERT AND REACTIVE GAS STUDIES**

Principle Of Operation:

A small amount of a sample is placed in a quartz boat, which is suspended from one arm of a sensitive electrobalance. The suspended sample boat hangs inside a glass walled, computer controlled furnace, with gas tight fittings. A thermocouple, which is used to monitor the temperature at the sample, is also located in the furnace next to the sample boat. The furnace, electrobalance, thermocouple and gas flow controllers are monitored and commanded by PC software, allowing independent programming of constant temperature and temperature ramp segments, along with switching different gases during a run. Under the selected conditions, different organic and inorganic components are evolved or decomposed, leading to a mass loss which is measured by the electrobalance. An example of the initial temperature and mass change profiles collected during a TGA run are shown in the following example.

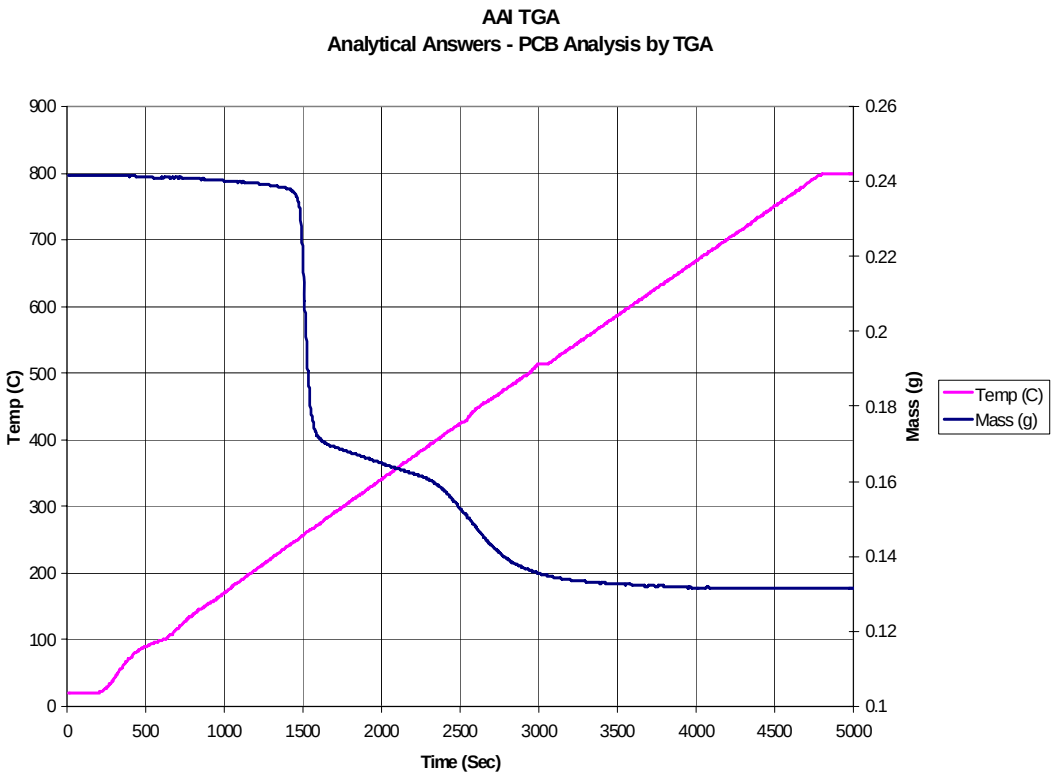

Figure 1 - Temperature and Mass Change Profiles from TGA Run of a Printed Circuit Board.

Combining the time-temperature and time-mass profiles generates a plot relating mass as a function of temperature, clearly showing the temperatures where different materials start to break down. In this example, the TGA results show an initial weight loss of 2%, most probably due to trapped moisture, low molecular weight coatings, residual soldering fluxes and other similar compounds. Starting at ~250ºC, a sharp weight loss, followed by a more gradual weight loss, occurs as the resin is boiled off and pyrolized out of the fiberglass mat. The ending mass shows the board is composed of 54% fiberglass by weight, 44% resin, and 2% other components.

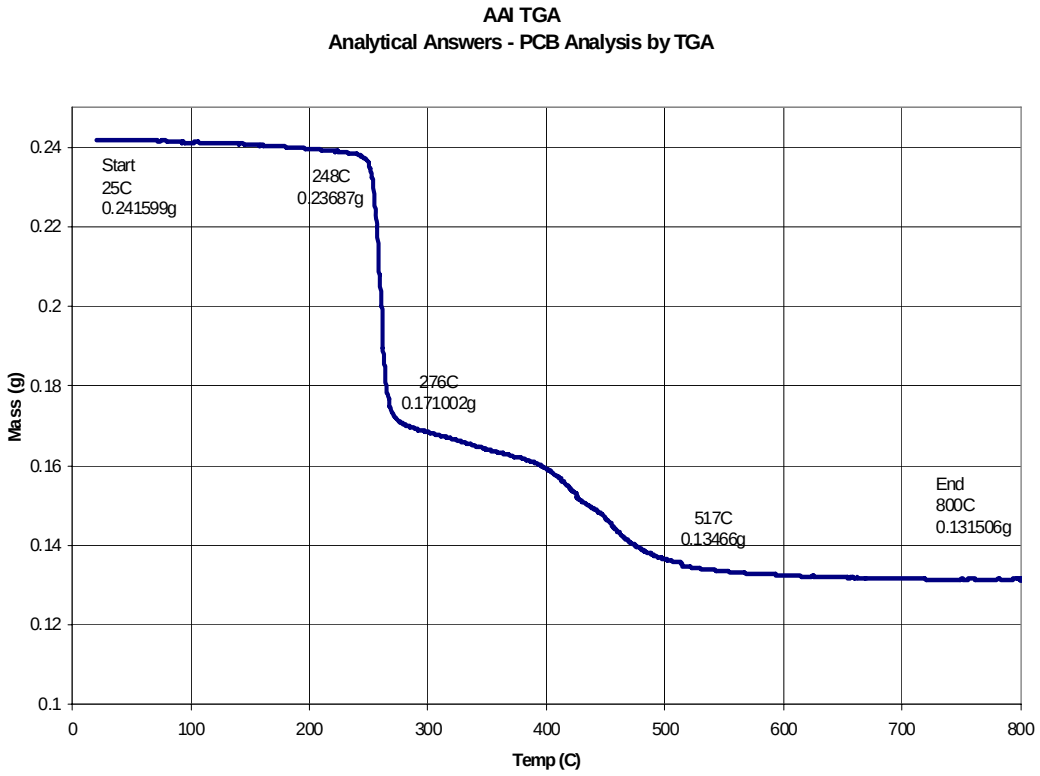

Figure 2 - Mass Change as a Function of Temperature from TGA Run of a Printed Circuit Board

Data Output:

The TGA generates plots showing temperature and mass as a function of time, along with mass as a function of temperature. Additional plots showing the derivative of mass as a function of temperature (dW/dT) may be used to deconvolve complex weight loss profiles. Data can be provided in hard copy format, as PDF images of the hard copy plots, ASCII X-Y data pairs (for Excel and other spreadsheet programs), ThermoGalactic's SPC format (for Grams/32 and Grams/AI), and others.

Sample Constraints:

Samples must be solids or liquids, stable at room temperature, and be provided or prepared to 10g mass or less and occupying a volume of < 1 cm^3.