



Techniques in **Thermal Analysis**

Hyphenated Techniques,
Thermal Analysis of the Surface,
and Fast Rate Analysis

WP. Pan
L. Judovits
Editors



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***Techniques in Thermal Analysis:
Hyphenated Techniques, Thermal
Analysis of the Surface, and Fast
Rate Analysis***

Wei-Ping Pan and Lawrence Judovits, editors

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Foreword

This publication, *Techniques in Thermal Analysis: Hyphenated Techniques, Thermal Analysis of the Surface, and Fast Rate Analysis*, contains papers presented at the symposium of the same name held at ASTM International Headquarters, W. Conshohocken, PA, on 24-25 May 2004, sponsored by the ASTM International Committee E37 on Thermal Measurements. The symposium chairmen were Prof. Wei-Ping Pan, Western Kentucky University, Bowling Green, KY and Dr. Lawrence Judovits, Arkema Inc., King of Prussia, PA.

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Overview (updated 1/12/2007)

In May 2004 a two day symposium titled “Techniques in Thermal Analysis: Hyphenated Techniques, Thermal Analysis of the Surface, and Fast Rate Analysis” was held at the ASTM Headquarters in West Conshohocken, PA. Twenty-two presentations were given at the symposium. Additionally, the presenters were given the opportunity to submit to the Journal of ASTM International and for their papers to be included into a special technical publication (STP), thirteen papers were received.

The symposium itself was timely and reflected leading edge research in thermal analysis. Of major interest now is fast scan calorimetry in both instrument development and techniques. Through the use of a thin film nanocalorimeter scanning rates as high as 10,000 °C/sec can now be achieved. This, for example, allows for the better study of semicrystalline polymers where the reorganization process can be inhibited and the original metastable crystal can now be analyzed. Through the use of current technology, fast heating rates were employed to study epoxy curing. Fast rate analysis allowed the separation of the glass transition and cure exotherm.

The Hyphenated Techniques session brought some interesting papers mostly using thermogravimetric analysis (TGA) with another technique. It should also be noted that other techniques that have hyphens were also presented such as a paper on temperature-modulated differential scanning calorimetry, which is more prevalently written modulated temperature differential scanning calorimetry without the hyphen. An interesting study of the combined use of TGA with DTA (differential thermal analysis) and Raman spectroscopy was presented. The spectroscopy was performed on the sample itself as it underwent physical changes. This allowed the more precise study of dehydration of pharmaceuticals. Also presented was a paper advocating improved modeling when using hyphenated techniques such as TGA/FTIR (Fourier transform infrared) allowing kinetic parameters to be determined using both sets of data. Also of note was a simple calibration method for the quantitative use of mass spectrometry with TGA for a variety of encountered off gases.

Finally, a number of papers were given on thermal analysis of the surface. Many of these papers centered on the use of a modified atomic force microscope (AFM), or Micro-Thermal Analysis, that uses the AFM probe as a thermal device. A technique that shows promise is the use of micro-thermal analysis in combination with other techniques such as FTIR. This technique is referred to as photo thermal micro-spectroscopy (PTMS). PTMS uses the AFM probe to detect temperature fluctuations after a sample has been exposed to IR radiation allowing the construction of an infrared spectrum. This permits for a fast identification of an unknown material with minimal sample preparation.

The symposium chairs would like to acknowledge and extend our appreciation for all who have helped with the organization of the symposium and subsequent publications. A special thanks goes out to the reviewers who took the time and provided the needed commentary. Finally, we would like to recognize the sponsorship of both ASTM International Committee E37 on Thermal Measurements and the Thermal Analysis Forum of the Delaware Valley.

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HYPHENATED TECHNIQUES

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