Thermal Analysis Method Development

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INTRODUCTION

This poster guides the new thermal analyst through the most demanding areas of analytical science. The difficulty of defining the appropriate thermal analytical technique and how to develop a thermal method for that technique are simplified.

Method Development Steps to follow

- Step 1 -- Ask a series of questions.
- Step 2 -- Identify the Thermal Analysis Techniques to use
- Step 3 -- If applicable, select the appropriate ASTM method to use
- Step 4 -- Enlist expert knowledge to assist and interpret data.

Step 1 -- Ask a Series of Questions. This is the ground work for the best thermal technique and method.

Question 1 -- What is the physical state of the material?

Is the material state a powder?
Is the material state a pellet?
Is the material state a paste?
Is the material state a foam?
Is the material state a thin film?
Is the material state a fiber?
Is the material state a liquid?
Is the material state a rectangular bar?
Is the material state an irregular shape?
Is the material state a tube?
Is the material state a gel?
Is the material state a cylinder?

These questions begin to identify what thermal techniques and accessories should be used.

Question 2 -- What are the material's physical properties to be analyzed by Thermal Analysis?

Do they include:

Soften points?
Effects of plasticizer?
Glass transition temperate?
Melting point?
Heat of fusion?
Specific Heat?
Purity?
Oxidative stability
Rates of Reaction (Kinetics)
- Curing?
- Cross linking?

Degree of cure?
Decomposition?
Optimum Crystallization temperature?
Pyrolysis?
Weight percent filler?
Weight loss vs. time?
Weight loss vs. temperature?
- Loss of water?
- Loss of solvent?
- Loss of plasticizer?

Impact properties?
Viscoelastic behavior?
Mechanical behavior?
Long term behavior?
Modulus vs. temperature?
Damping vs. temperature?
Chain branching?
Molecular weight?
Molecular weight distribution?
Liquidus or Solidus temperatures?

Question 3 -- What type of information is required?

Quantitative?
Qualitative?
A mixture of both?

Question 4 -- What analytical accuracy and precision is required?

Accuracy is the extent to which the results of a measurement approach the true values.
Precision is measure of the range of values of a set of measurements.
Example: When five darts are thrown at a bull's eye hit the bull's eye, then the accuracy is said to be good. The fact that all five darts are close to each other indicates good precision.

Question 5 -- Are the events of interest minor or major?

Question 6 -- What do you already know about the material or sample?

Question 7 -- What are the important properties of the material?

Question 8 -- Is the material a single component or a complex mixture?

Question 9 -- What is the material's history?

Question 10 -- What is the material's future?

Question 11 -- Is there a limitation on sample size?

Question 12 -- How many samples must be run?

Question 13 -- What is the required analysis turn-around time?

Question 14 -- Are there any safety hazards to be concerned about?

Step 2 – Use the applications listed below to help identify appropriate Thermal Analysis techniques for your investigation. Many times a combination of techniques are required to solve the problem.

Differential Scanning Calorimetry (DSC) is the workhorse of thermal analysis laboratory. DSC can examine materials between -180C to 750C.

Definition: DSC is a technique upon which the heat flow to or from a sample specimen is measured as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere.

Some DSC applications are listed below:

- *Identify the softening point of a material (glass transition).*
- *Compare additive effects on a material.*
- *Identify the glass transition temperature.*
- *Identify a material's minimum process temperature.*
- *Identify the amount of energy to melt a material.*
- *Quantify a material's specific heat.*
- *Perform oxidative stability testing (OIT).*
- *Characterize a material as it cures under UV light.*
- *Optimize the crystallization temperature upon cooling.*
- *Study polymorphic stability in pharmaceuticals.*

Some more DSC applications are listed below:

- *Study amorphous content.*
- *Understand the reaction kinetics of a thermoset material as it cures.*
- *Compare the degree of cure of one material to another.*
- *Examine thermal events that are very week or masked by other material characteristics by employing very fast scanning rates (Hyper-DSC ®).*

Thermogravimetric Analysis (TGA) examines materials between ambient and +1500C.

Definition: TGA is a technique in which the mass of a substance is monitored as a function of temperature or time as it is subjected to a controlled temperature program in a controlled atmosphere.

Some TGA applications are listed below:

- *Identify the filler content of a material by weight percent.*
- *Identify the ash content of a material by weight percent.*
- *Characterize a material's weight loss within a certain temperature range.*
- *Characterize a material's weight loss vs. time at a given temperature.*
- *Quantify a material's loss of water, solvent, or plasticizer within a certain temperature range.*
- *Examine flame retardant properties of a material.*
- *Examine the combustion properties of a material.*
- *Perform oxidative stability testing.*

Dynamic Mechanical Analysis (DMA) examines materials between -170C and +400C.

Definition: DMA is a technique in which a substance while under an oscillating load is measured as a function of temperature or time as the substance is subjected to a controlled temperature program in a controlled atmosphere.

Some DMA applications are listed below:

- *Quantify impact properties of a material.*
- *Examine the Viscoelastic behavior of a material.*
- *Examine a material's mechanical behavior.*
- *Examine a material's long term behavior while under load.*
- *Ability to extrapolate material behavior on a time basis.*
- *Identify a material's modulus vs. temperature.*
- *Examine a material's damping qualities vs. temperature.*
- *Examine the effects of temperature on molecular chain branching.*
- *Compare a material's molecular weight and weight distribution.*
- *Examine additive effects on a material's mechanical properties.*
- *Material -humidity studies from 10% RH to 80% RH*

Step 3 – Research ASTM methods. There may be a traditional ASTM method that is suited for your investigation.

- There are more than 25 ASTM methods for each thermal technique.
- There are several ASTM methods that can utilize more than one technique of thermal analysis.

Step 4 – If required, seek out expert knowledge to review your data, suggest additional testing, and recommend possible solutions to your problem.

For expert knowledge look to:

- Local Universities
- Consultants
- Instrument manufacturers

Summary:

Thermal Analysis is a simple and fast way to analyze a material problem. DSC, TGA, and DMA techniques complement each other and bring fast and accurate results.

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