HUMAN HEALTH

ENVIRONMENTAL HEALTH

# POLYMER RECYCLING COMPENDIUM

Thermal Analysis and FT-IR Spectroscopy

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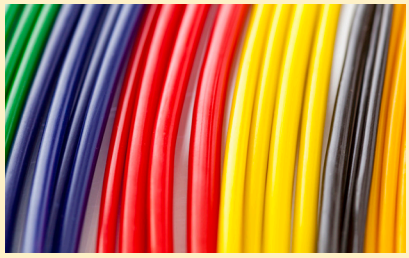
# INTRODUCTION TO THE POLYMER RECYCLING PACK



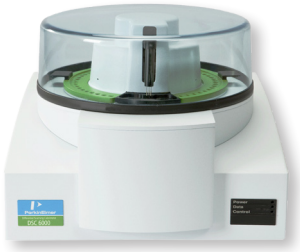
The use of recycled plastic requires rigorous testing to ensure proper separation and identification as some polymers are not compatible when mixed. Poor incoming raw material inspection can lead to multiple production issues. To address this, polymer products are "coded" as one of seven codes. This greatly assists in the separation when the code is visible on the product. This may not always be the case when a material is ground or bundled. The coding system also fails to account for fillers and additives, which may be detrimental to finished products. An improperly characterized polymer source can ruin an entire day of production due to not only incorrect formulation but also the cleaning required for the processing machinery.

The analytical techniques, mostly commonly implemented in the analysis of polymers are Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FT-IR). DSC is primary utilized in the determination of melting and/or glass transition temperatures. DSC can also allow for polymer ratios to be determined in blended materials. TGA is often utilized to detect polymer, filler, and carbon black content. TGA also affords the opportunity to determine moisture content, and in some instances, polymer ratios. FT-IR is a first-line measurement to assist in the identification of polymers, fillers, and additives. DSC, TGA, and FT-IR are key techniques to ensure polymer identification, and the quality of the materials received and produced.

This compendium provides example data and interpretations for the seven codes of recyclable polymers. DSC and TGA raw data files are provided so that the user can easily overlay their data with the expected results for new unprocessed plastics. This allows for a conformation of identity and assists in the analysis of incoming materials. An FT-IR library is provided that contains the most commonly encountered plastics along with some of the more prevalent additives and polymer blends. Additionally, method files are provided for the three instruments to ensure that the data collected is in accordance with polymer processing standards.

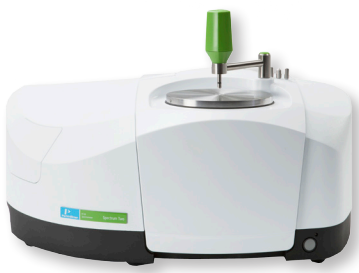
# Table of Contents





DSC 6000
Differential Scanning Calorimeter

TGA 4000
Thermogravimetric Analyzer

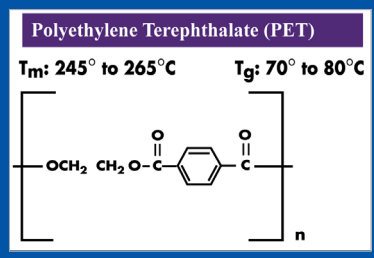


Spectrum Two™
FT-IR Spectrometer

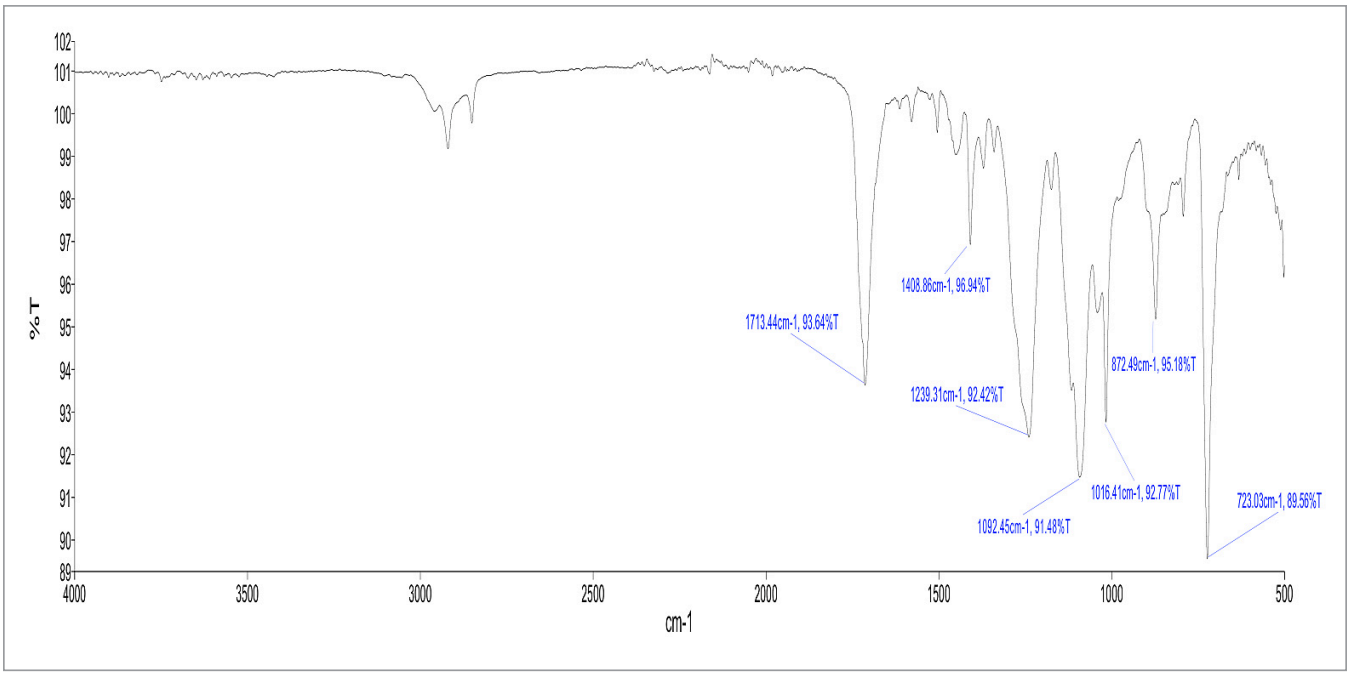
APPLICATION BRIEF

## Pure PET Analysis

# Code 1a
# Polyethylene Terephthalate




## FT-IR:



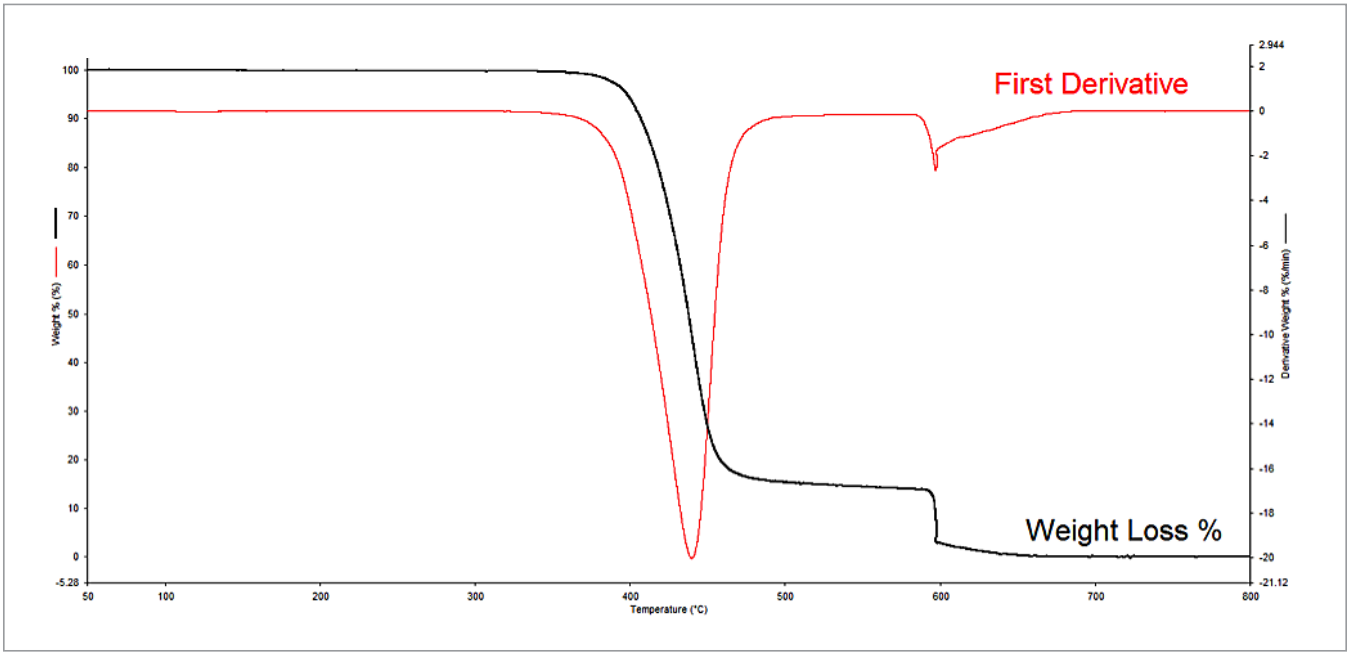

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 1713 | 94 |
| 2 | 1409 | 97 |
| 3 | 1239 | 92 |
| 4 | 1092 | 91 |
| 5 | 1016 | 93 |
| 6 | 872 | 95 |
| 7 | 723 | 90 |

Polyethylene Terephthalate (PET) Applications

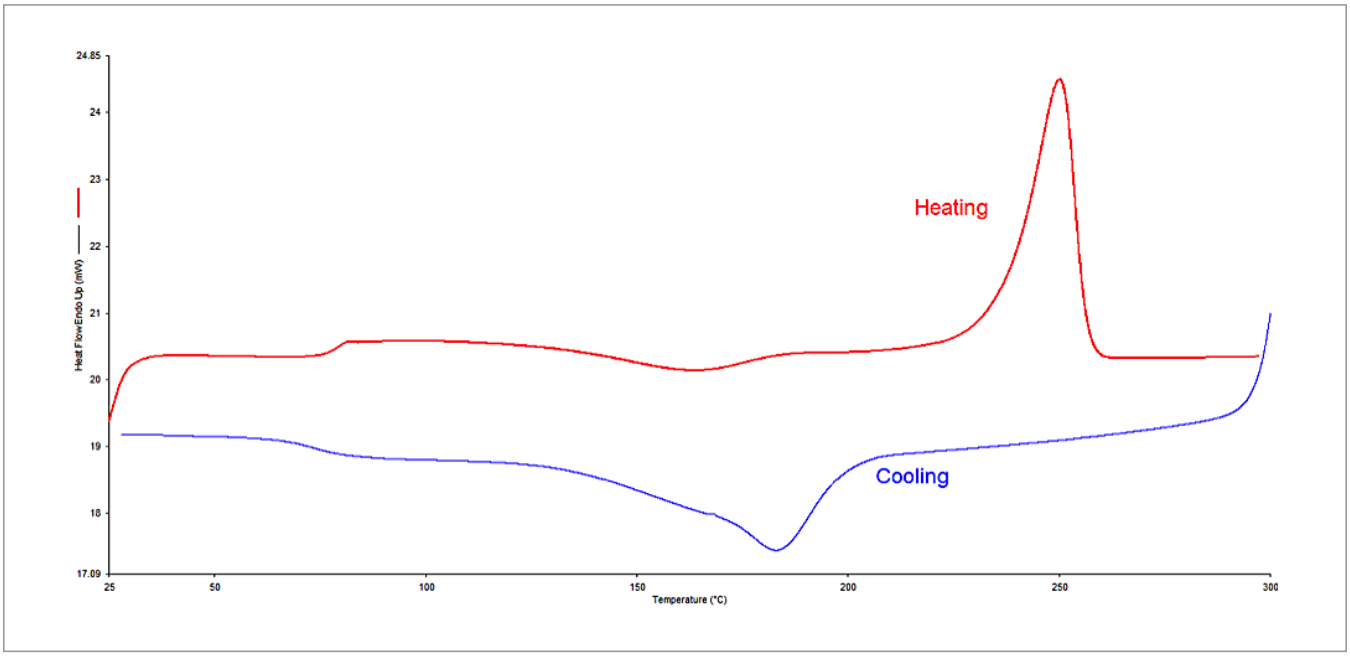
- Food packaging
- Fibers/clothing
- Films
- Electronics

# Pure PET Analysis

## TGA:




## DSC:

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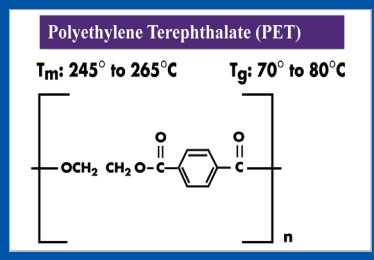
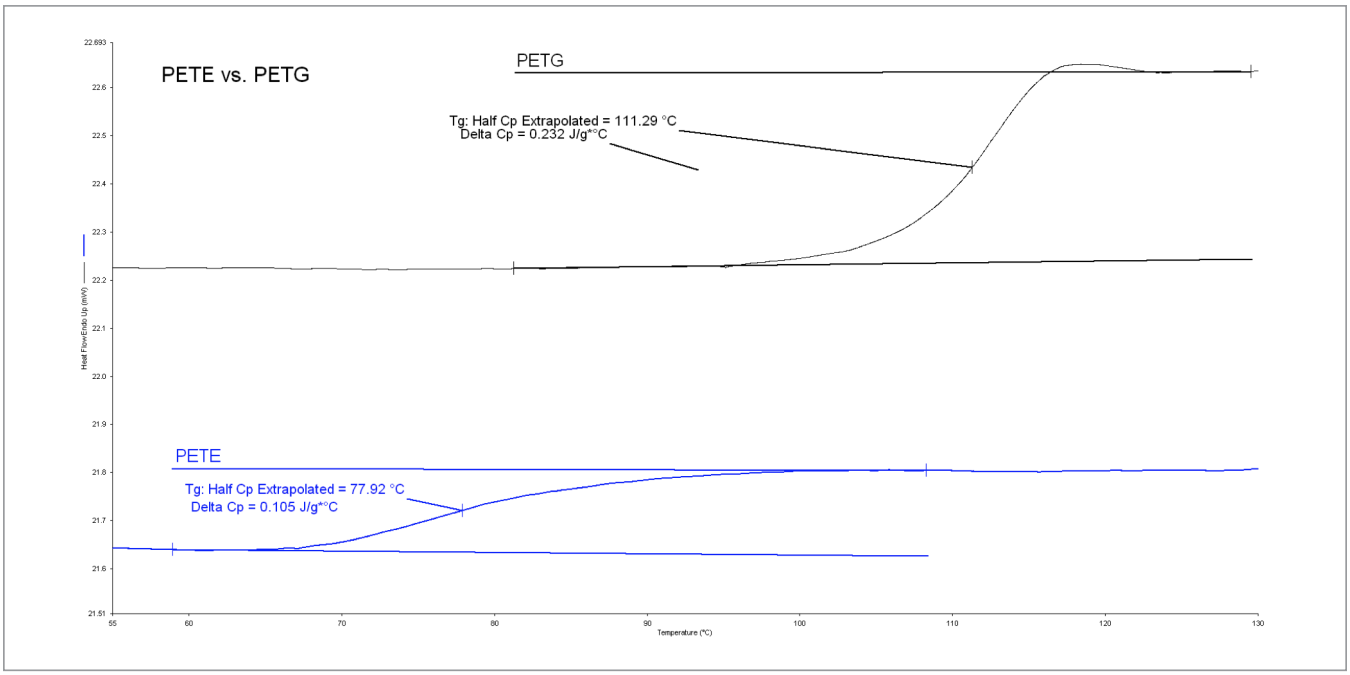
011519_01

APPLICATION BRIEF

# Method for Differentiating PETE from PETG by DSC

## Code 1b
## Polyethylene Terephthalate

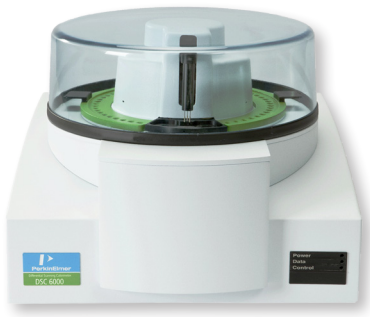



### Method:

DSC 4000 or 6000
Nitrogen 20 mL $min^{-1}$

1. Heat from 25.00 °C to 300.00 °C at 10.00 °C $min^{-1}$
2. Hold for 2.0 min at 300.00 °C
3. Cool from 300.00 °C to 25.00 °C at 10.00 °C $min^{-1}$
4. Hold for 2.0 min at 25.00 °C
5. Heat from 25.00 °C to 300.00 °C at 10.00 °C $min^{-1}$



Polyethylene Terephthalate (PET) Applications

- Food packaging
- Fibers/clothing
- Films
- Electronics

# Method for Differentiating PETE from PETG by DSC

## Discussion:

Polyethylene Terephthalate (PET) is a commonly recycled thermoplastic. There are numerous formulations available for PET which are optimized for specific applications. In this example, two types of PET are analyzed to demonstrate the DSC's ability to separate polymers based on their glass transition temperatures.

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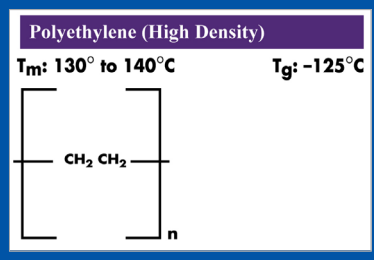
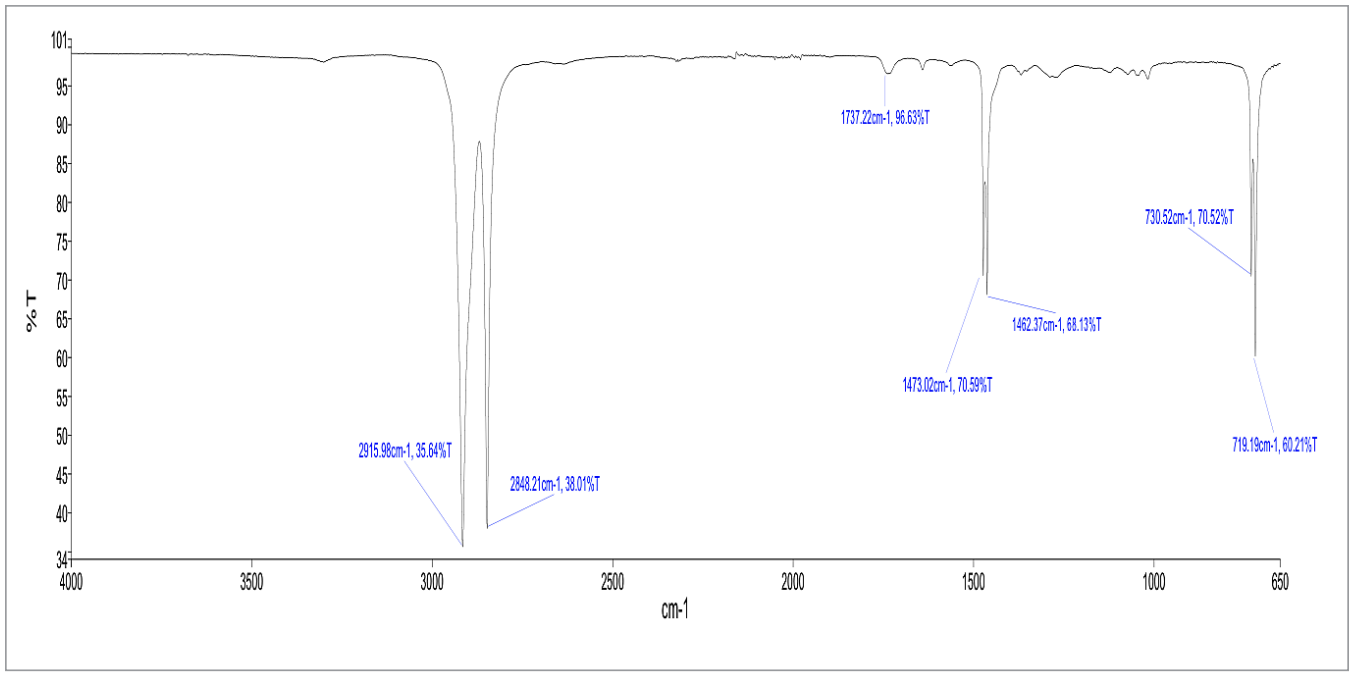
APPLICATION BRIEF

## Pure HDPE Analysis

# Code 2a
# High Density Polyethylene




## FT-IR:



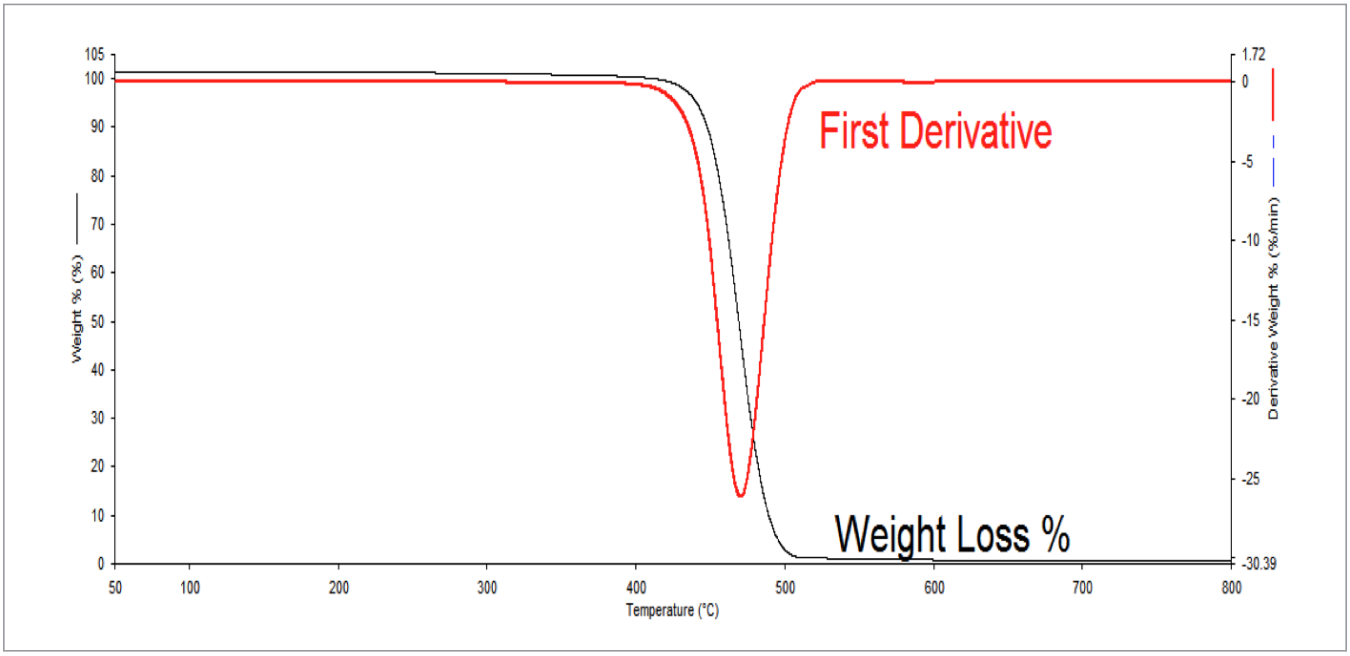

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 2916 | 36 |
| 2 | 2848 | 38 |
| 3 | 1737 | 97 |
| 4 | 1473 | 71 |
| 5 | 1462 | 68 |
| 6 | 731 | 71 |
| 7 | 719 | 60 |

High Density Polyethylene (HDPE) Applications

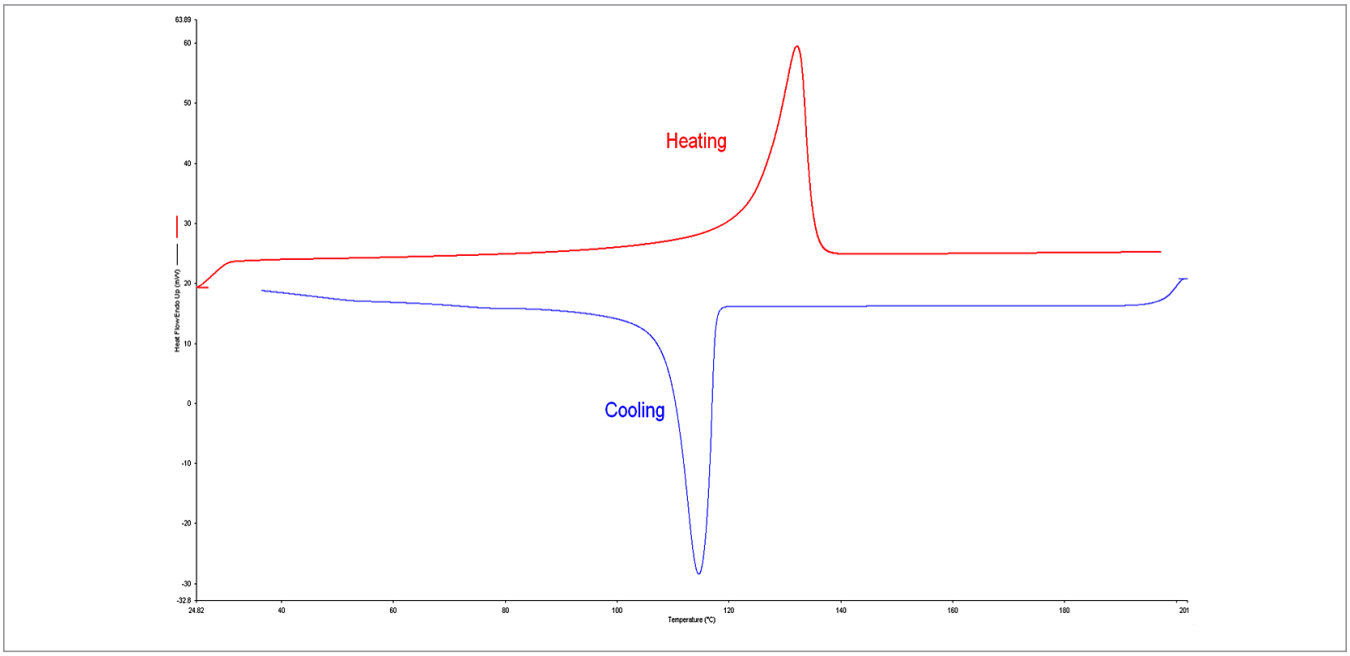
- Toys
- Packaging
- Pipes
- Cabling

# Pure HDPE Analysis

## TGA:




## DSC:

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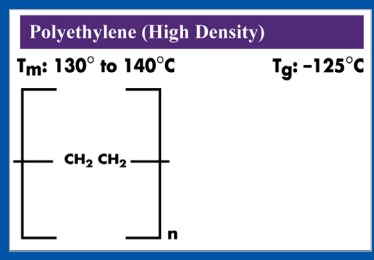
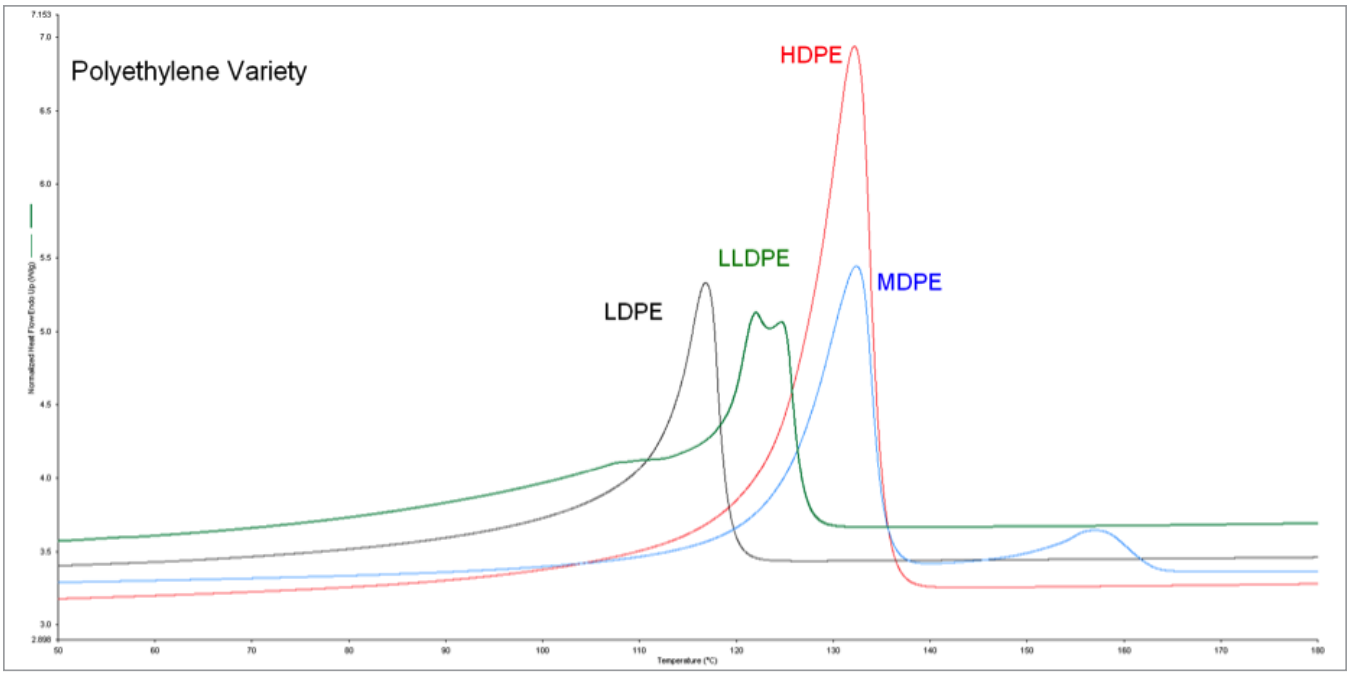
APPLICATION BRIEF

# Exemplifying the Variety of PE by DSC

## Code 2b
## High Density Polyethylene

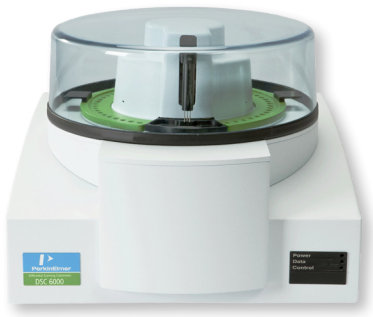



### Method:

DSC 4000 or 6000
Nitrogen 20 mL $min^{-1}$

1. Heat from 25.00 °C to 200.00 °C at 10.00 °C $min^{-1}$
2. Hold for 2.0 min at 200.00 °C
3. Cool from 200.00 °C to 25.00 °C at 10.00 °C $min^{-1}$
4. Hold for 2.0 min at 25.00 °C
5. Heat from 25.00 °C to 200.00 °C at 10.00 °C $min^{-1}$



High Density Polyethylene (HDPE) Applications

- Toys
- Packaging
- Pipes
- Cabling

# Exemplifying the Variety of PE by DSC

## Discussion:

There are many different types of Polyethylene (PE). The codes for polyethylene are 2 (for high density) and 4 (for low density). Linear Low Density Polyethylene (LLDPE) and Medium Density Polyethylene (MDPE) can be inadvertently added to the code 2 and 4 recycling streams. DSC is a very effective method in separating the different types of Polyethylene.

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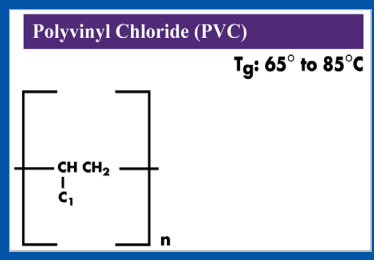
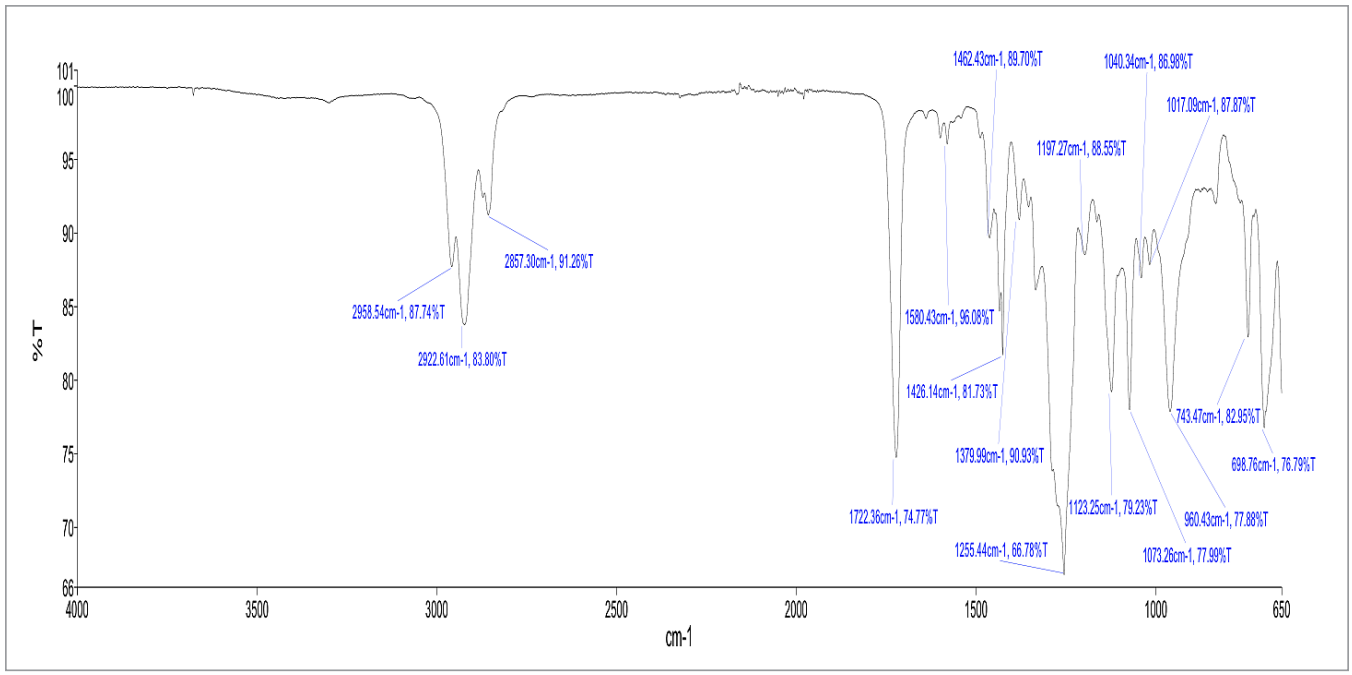
APPLICATION BRIEF

## Pure PVC Analysis

# Code 3a
# Polyvinyl Chloride




## FT-IR:



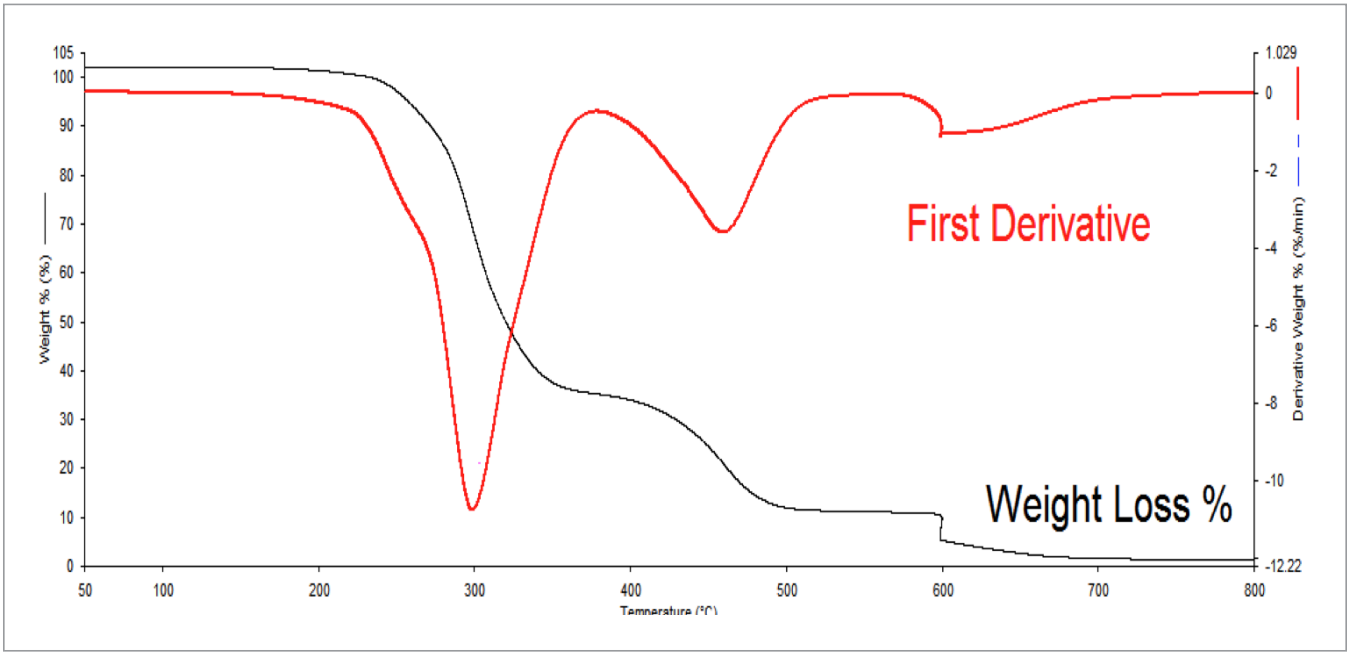

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 2959 | 88 |
| 2 | 2923 | 84 |
| 3 | 2857 | 91 |
| 4 | 1722 | 75 |
| 5 | 1580 | 96 |
| 6 | 1462 | 90 |
| 7 | 1426 | 82 |
| 8 | 1380 | 91 |
| 9 | 1255 | 67 |

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 10 | 1197 | 89 |
| 11 | 1123 | 79 |
| 12 | 1073 | 78 |
| 13 | 1040 | 87 |
| 14 | 1017 | 88 |
| 15 | 960 | 78 |
| 16 | 743 | 83 |
| 17 | 699 | 77 |

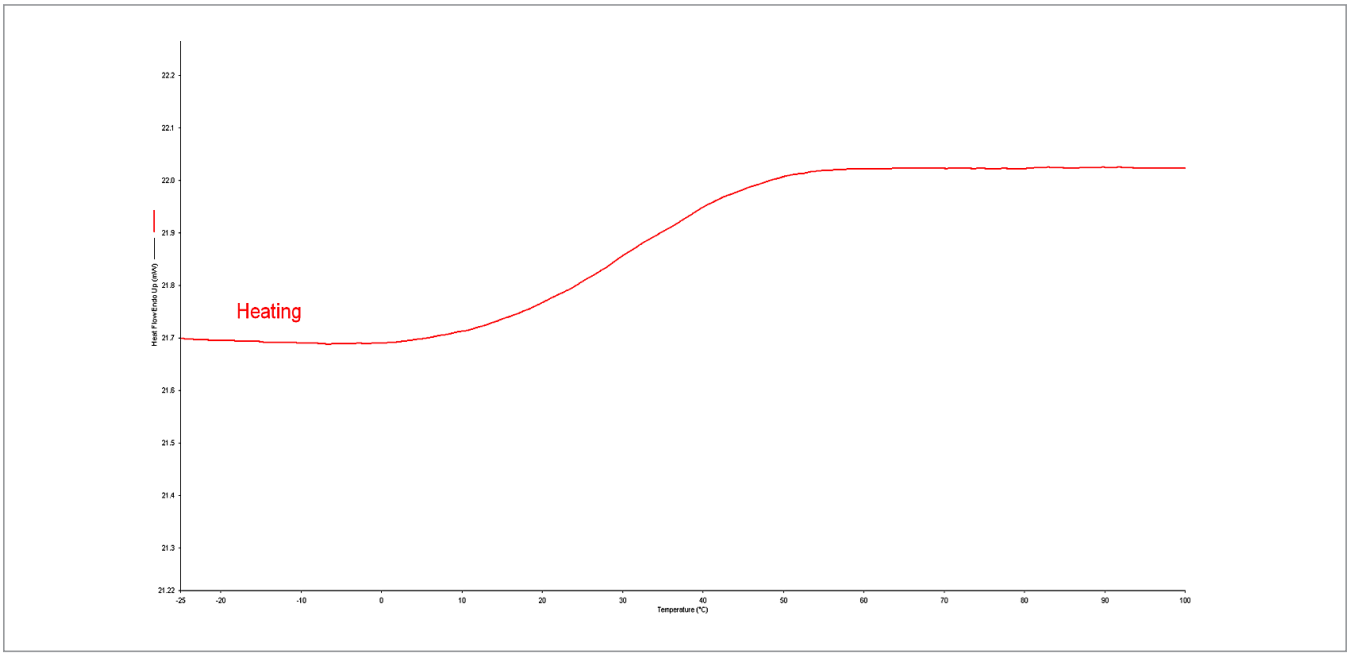
Polyvinyl Chloride (PVC) Applications
- Pipes
- Cabling
- Construction Materials
- Medical Tubing and Bags

# Pure PVC Analysis

## TGA:




## DSC:

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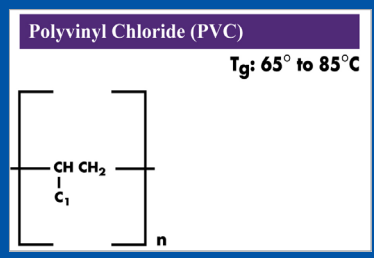
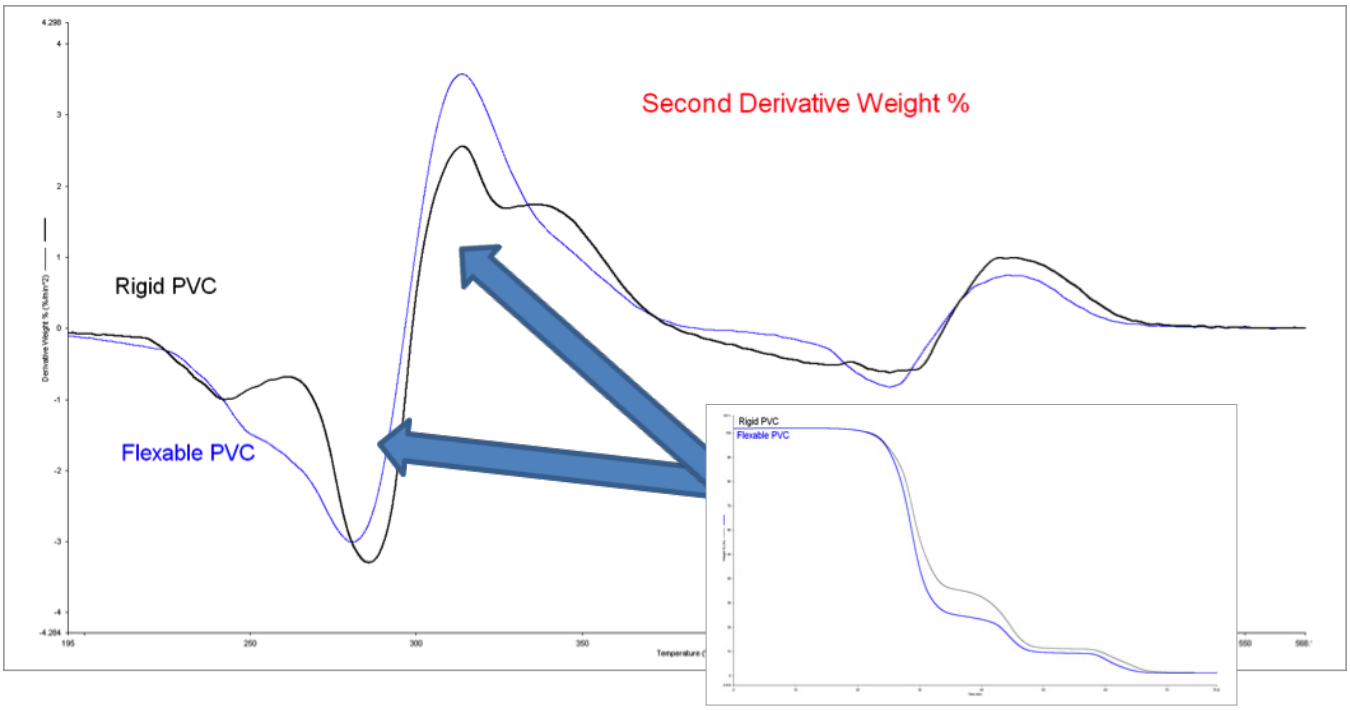
011523_01

APPLICATION BRIEF

# Detection of Additives in PVC by TGA

## Code 3b
## Polyvinyl Chloride (PVC)




### Method:

TGA 4000/STA 6000
Nitrogen purge 20 mL $min^{-1}$
1. Heat 30 to 900 °C at 10 °C $min^{-1}$
   a. Swap to air at 600 °C at 30 mL $min^{-1}$

Polyvinyl Chloride (PVC) Applications
- Pipes
- Cabling
- Construction Materials
- Medical Tubing and Bags

# Detection of Additives in PVC by TGA

## Discussion:

The functionality of materials can be greatly enhanced through additives and design. In some cases, such as Polyvinyl Chloride (PVC), utilizing the second derivative helps to identify subtle differences in formulation such as plasticizer content. This data analysis method assists in identifying shoulders that may otherwise be overlooked.

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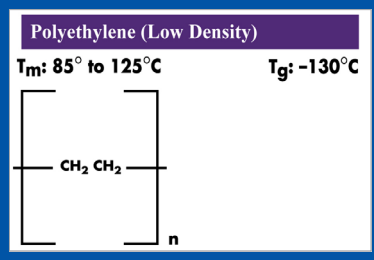
011524_01

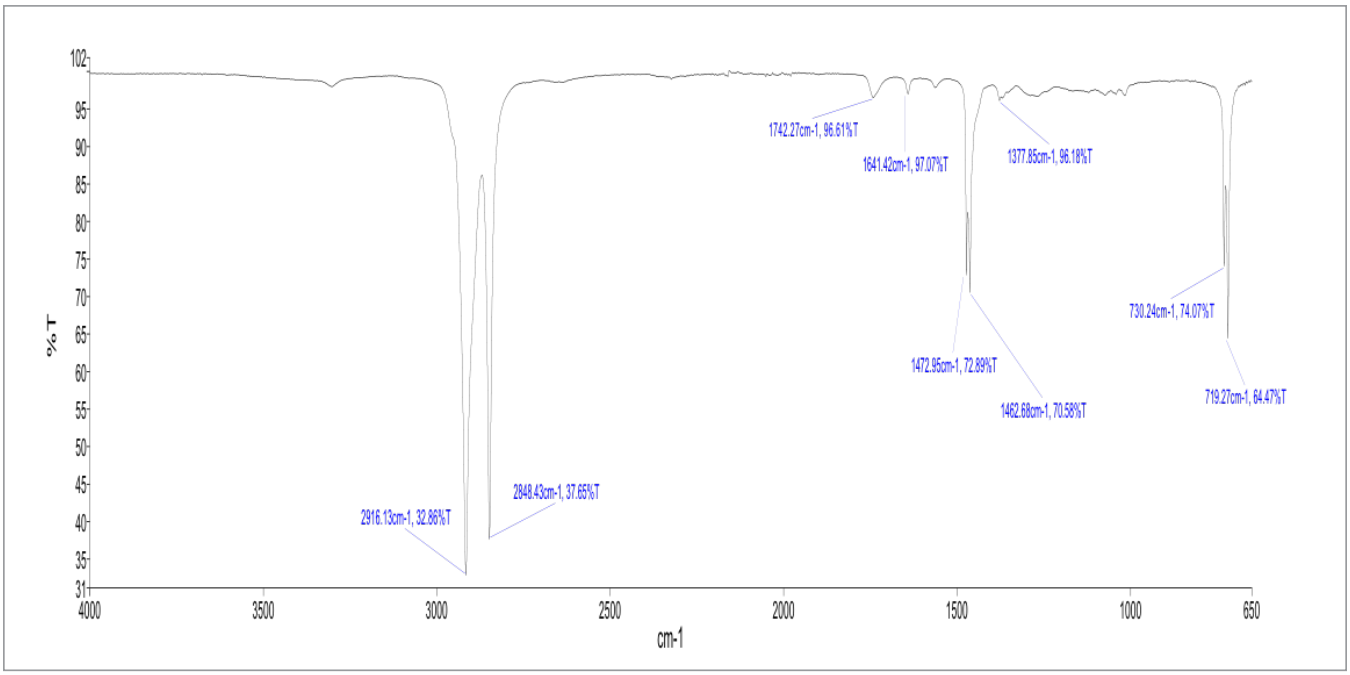
APPLICATION BRIEF

## Pure LDPE Analysis

# Code 4a
# Low Density Polyethylene




## FT-IR:



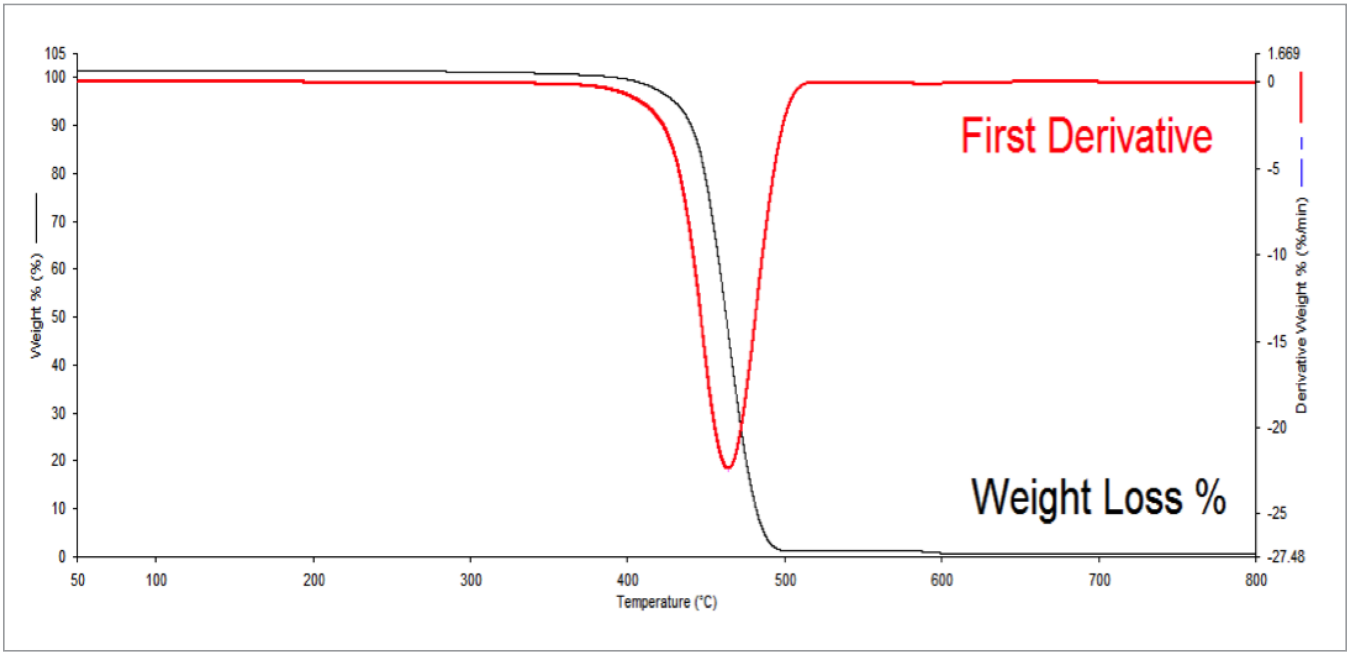

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 2916 | 33 |
| 2 | 2848 | 38 |
| 3 | 1742 | 97 |
| 4 | 1641 | 97 |
| 5 | 1473 | 73 |
| 6 | 1463 | 71 |
| 7 | 1378 | 96 |
| 8 | 730 | 74 |
| 9 | 719 | 64 |

Low Density Polyethylene (LDPE) Applications

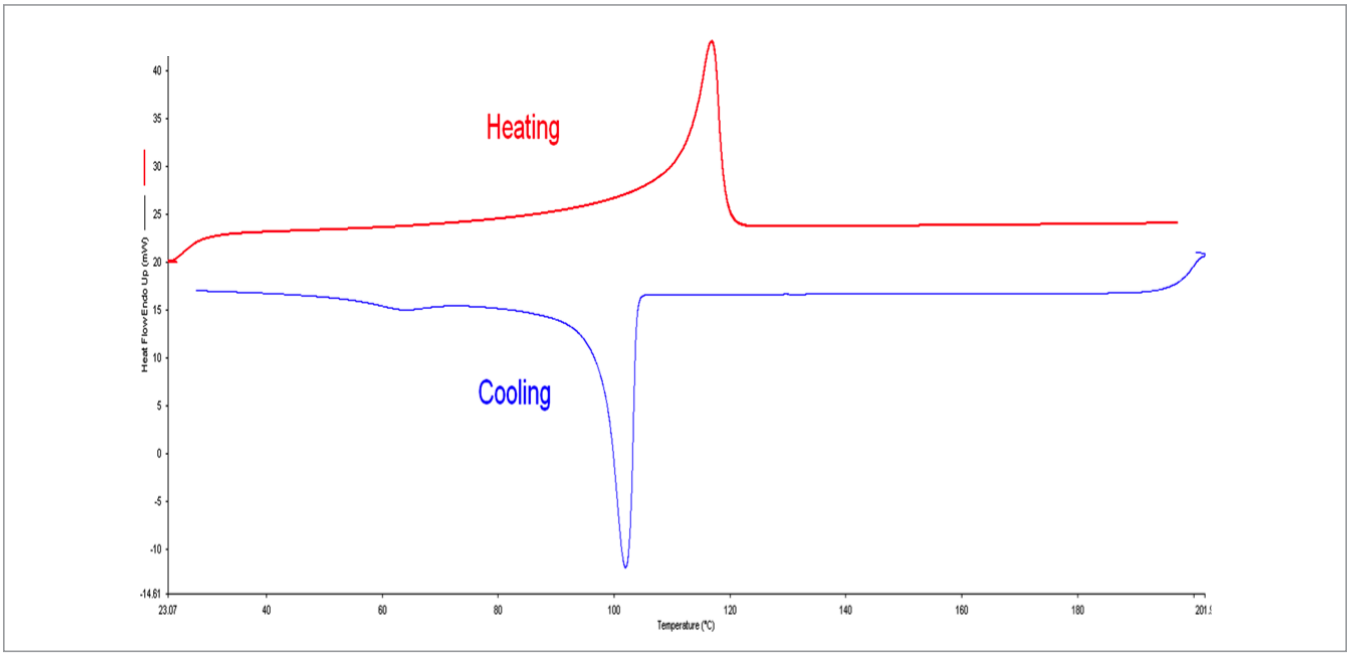
- Containers
- Food Packaging
- Toys
- Cabling

# Pure LDPE Analysis

## TGA:




## DSC:

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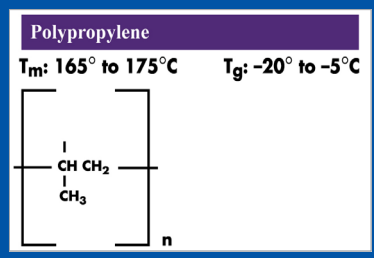
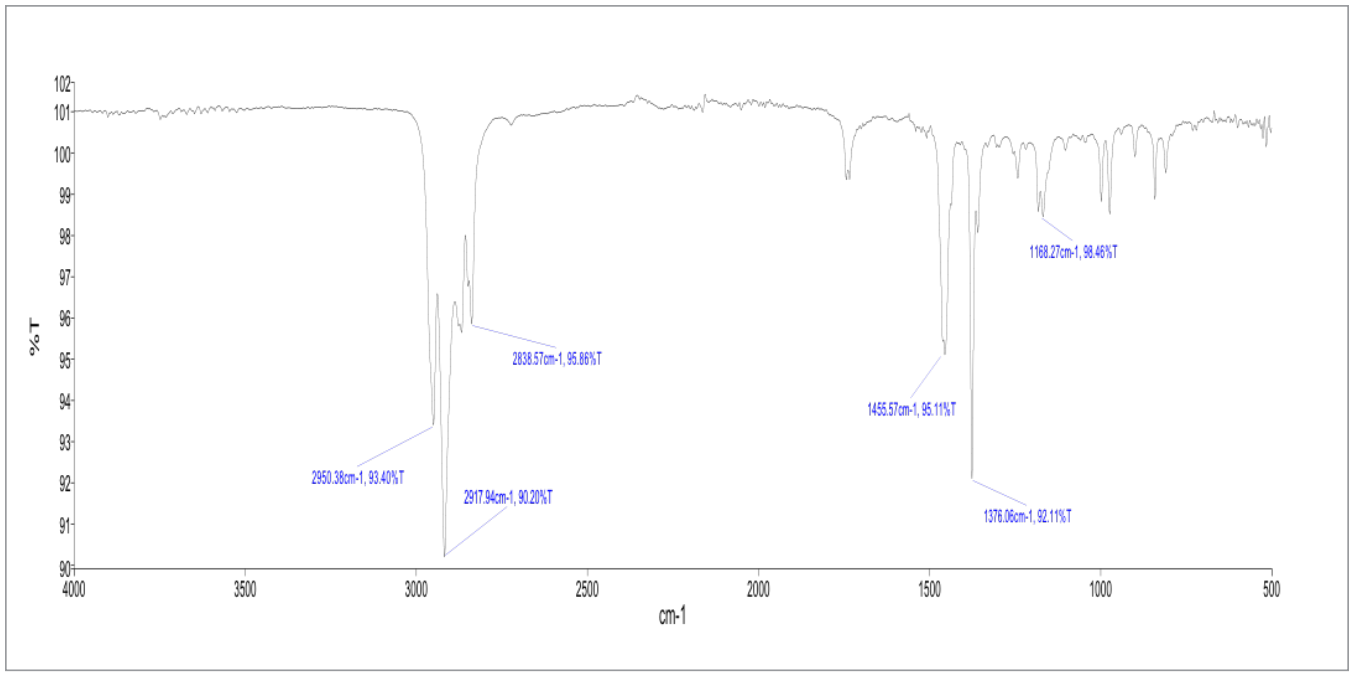
APPLICATION BRIEF

## Pure PP Analysis

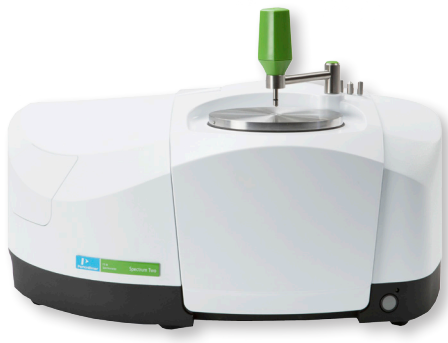
# Code 5a
# Polypropylene




## FT-IR:



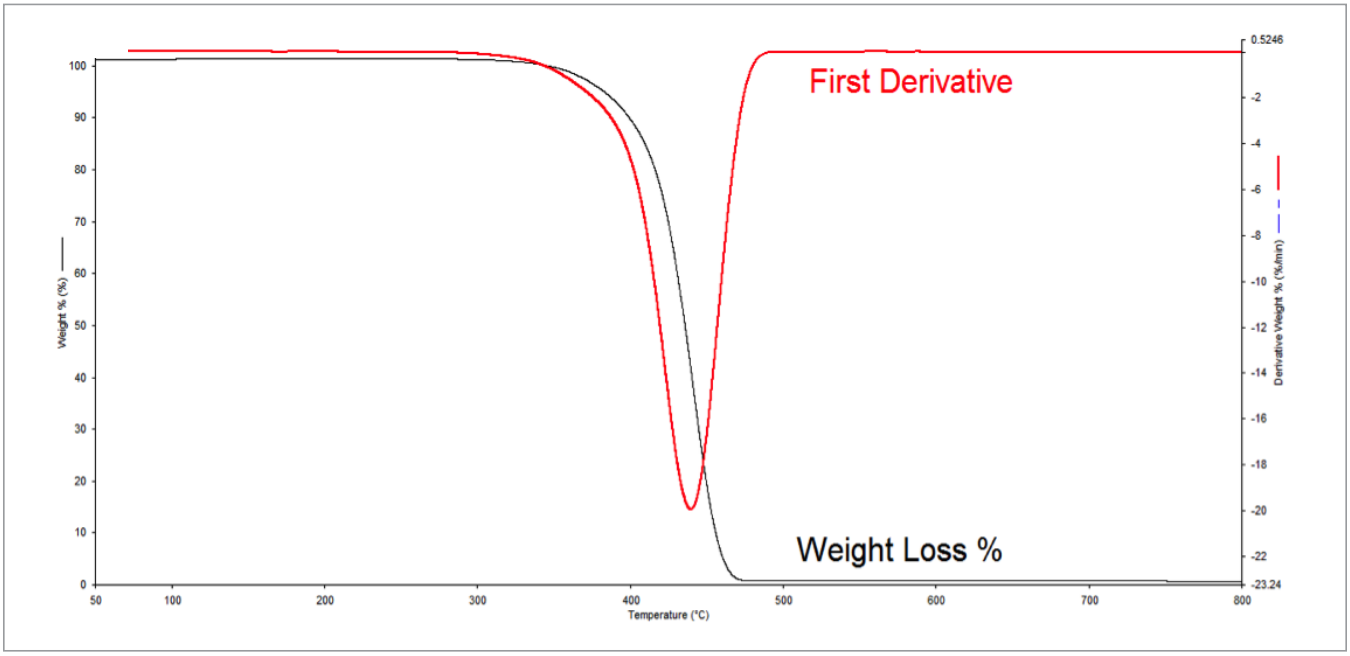

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 2950 | 93 |
| 2 | 2918 | 90 |
| 3 | 2839 | 96 |
| 4 | 1456 | 95 |
| 5 | 1376 | 92 |
| 6 | 1168 | 98 |



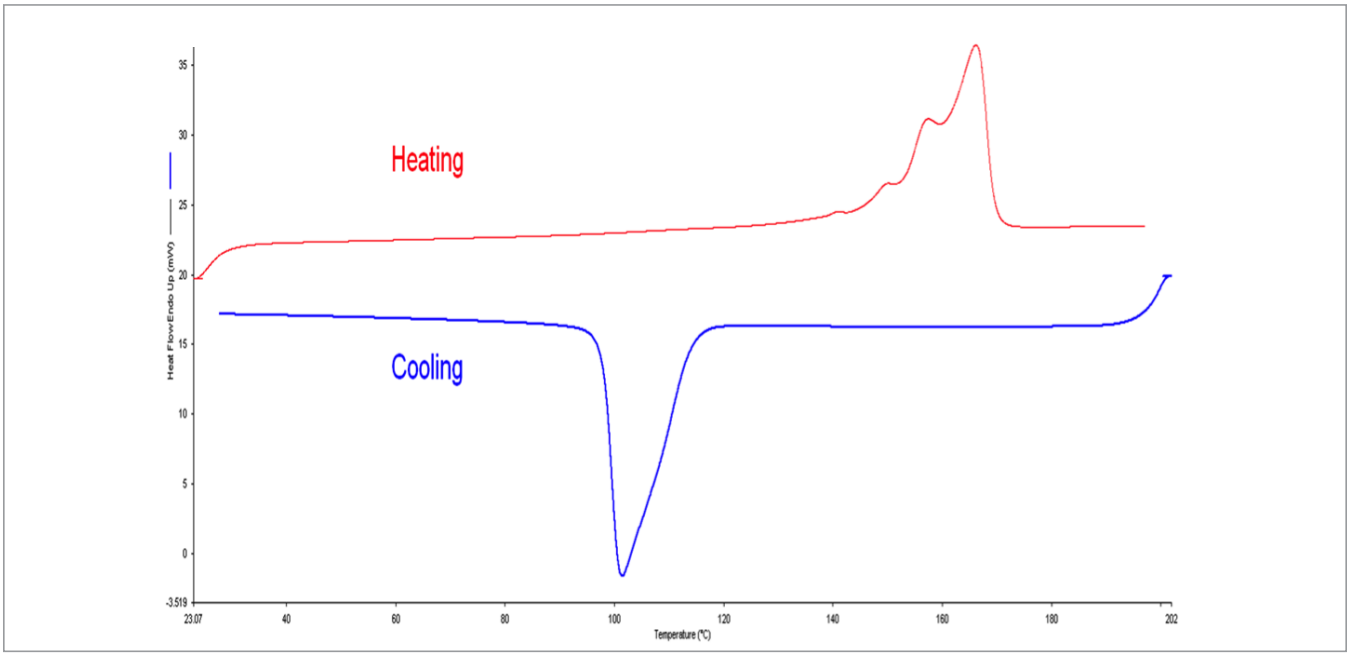
Polypropylene (PP) Applications
- Containers
- Piping
- Fibers/Clothing
- Packaging

# Pure PP Analysis

## TGA:




## DSC:

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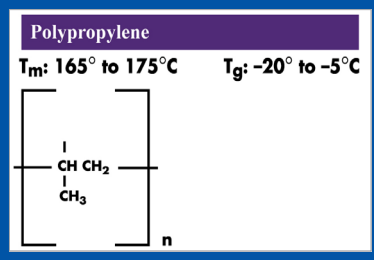
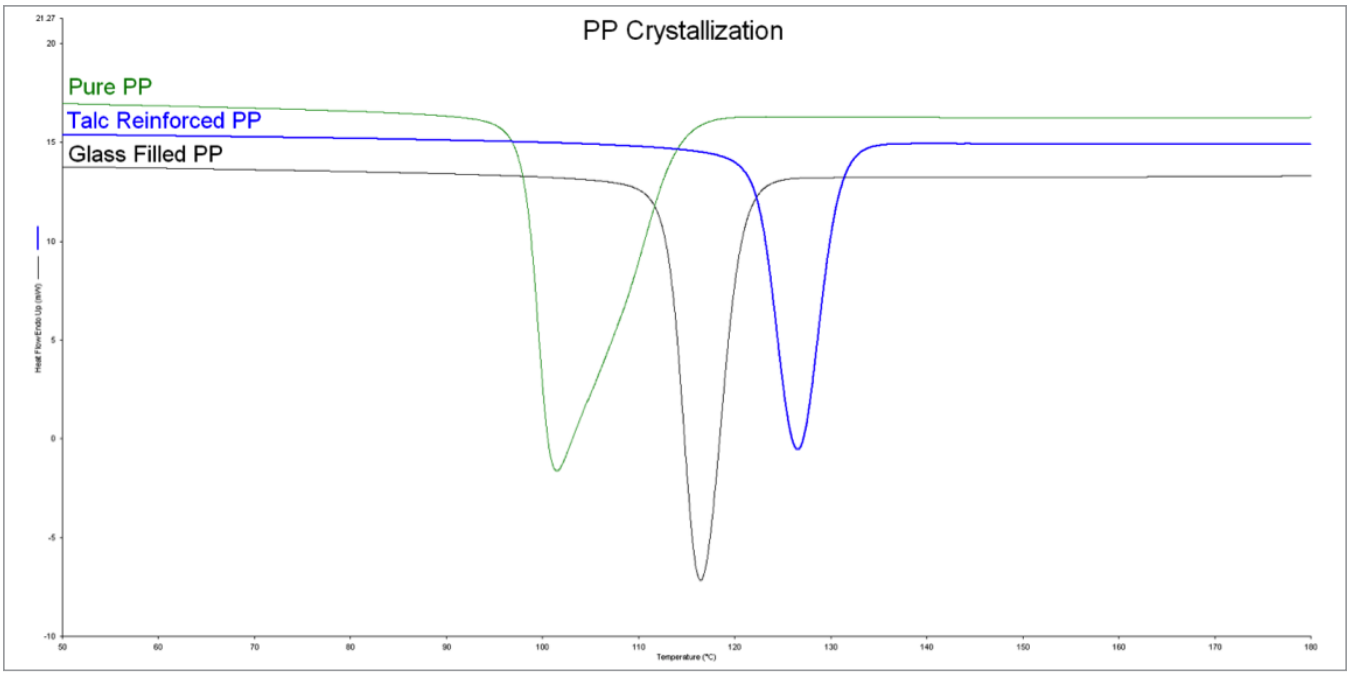
011526_01

APPLICATION BRIEF

# Analysis of the Effect of Filler on PP by DSC

## Code 5b
## Polypropylene

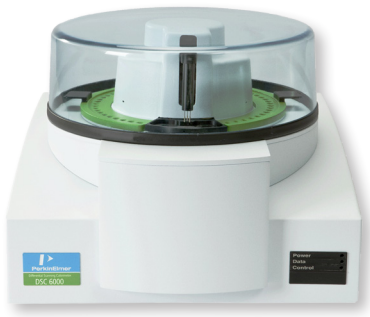



### Method:

DSC 4000 or 6000
Nitrogen 20 mL $min^{-1}$

1. Heat from 25.00 °C to 200.00 °C at 10.00 °C/min
2. Hold for 2.0 min at 200.00 °C
3. Cool from 200.00 °C to 25.00 °C at 10.00 °C/min
4. Hold for 2.0 min at 25.00 °C
5. Heat from 25.00 °C to 200.00 °C at 10.00 °C/min



Polypropylene (PP) Applications

- Containers
- Piping
- Fibers/Clothing
- Packaging

# Analysis of the Effect of Filler on PP by DSC

## Discussion:

Fillers can have a significant impact on the crystallization of Polypropylene. The way a material crystallizes impacts its mechanical performance and clarity. DSC allows for the characterization of the crystallization properties of Polypropylene.

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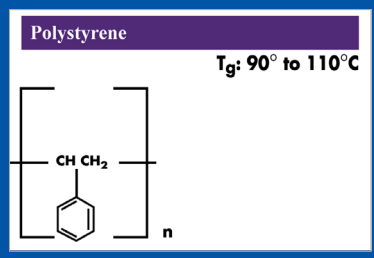
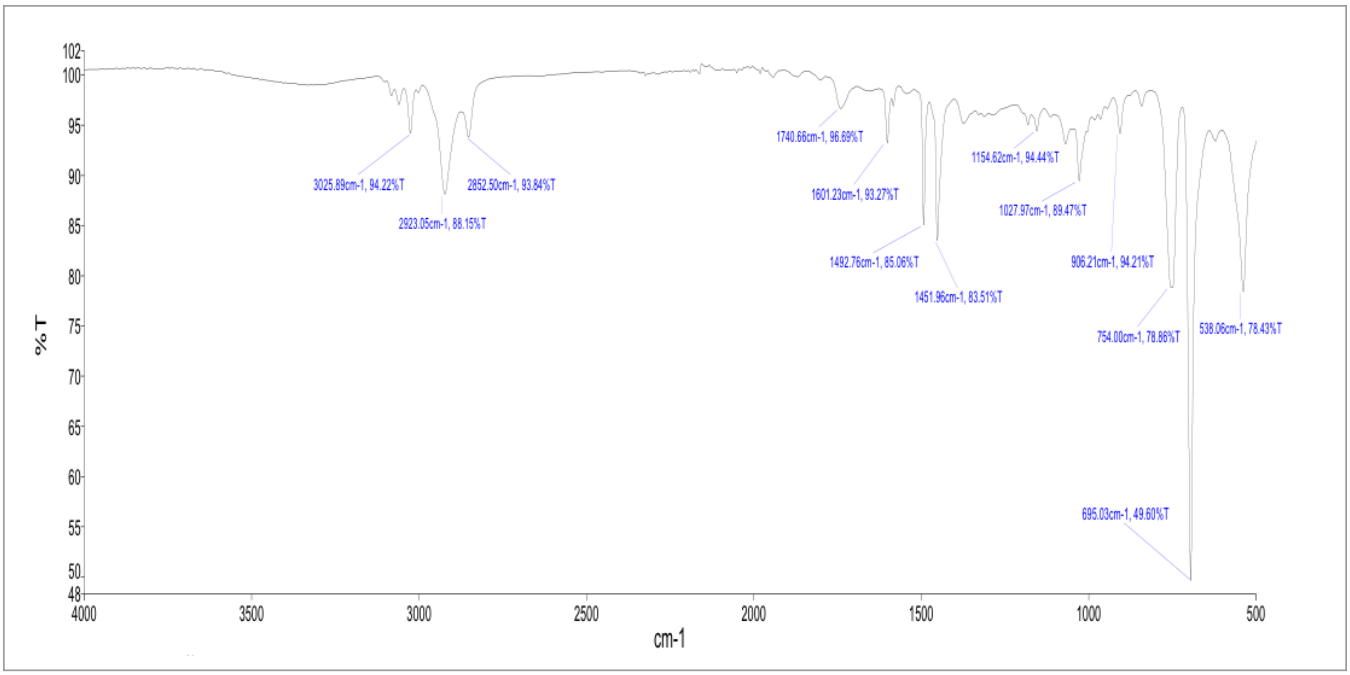
APPLICATION BRIEF

## Pure PS Analysis

# Code 6a
# Polystyrene




## FT-IR:



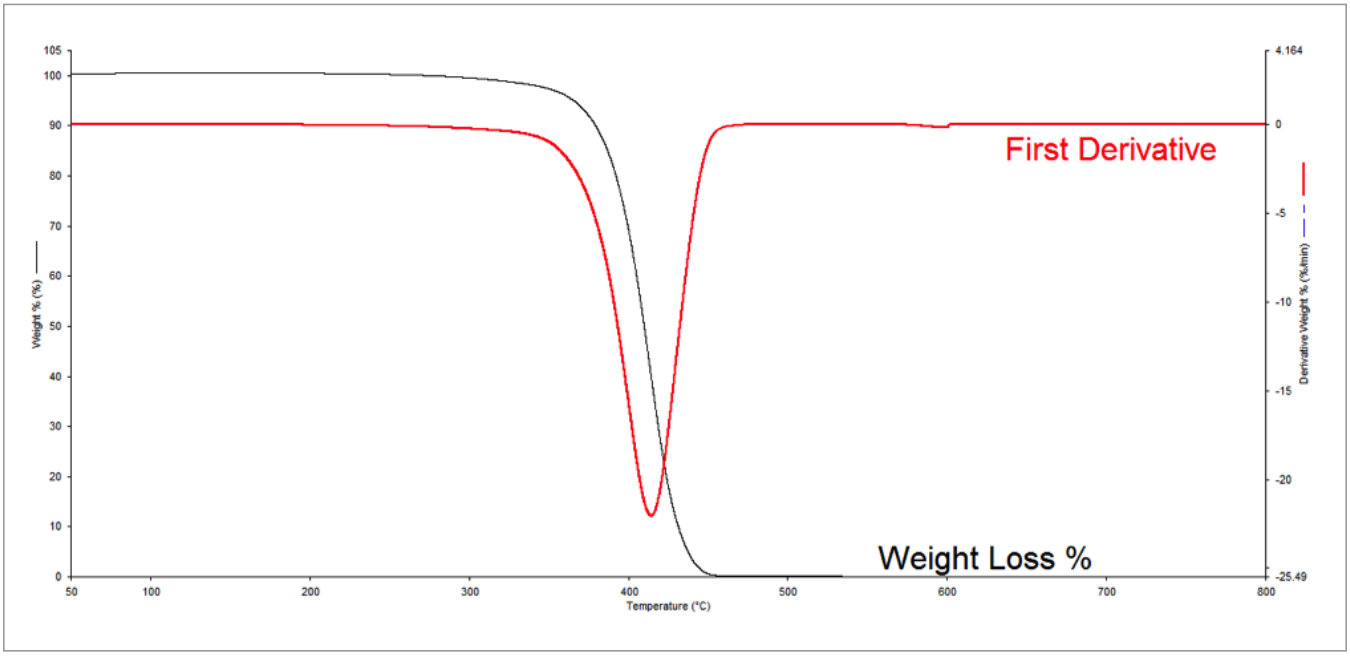

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 3026 | 94 |
| 2 | 2923 | 88 |
| 3 | 2853 | 94 |
| 4 | 1741 | 97 |
| 5 | 1601 | 93 |
| 6 | 1493 | 85 |
| 7 | 1452 | 84 |

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 8 | 1155 | 94 |
| 9 | 1028 | 89 |
| 10 | 906 | 94 |
| 11 | 754 | 79 |
| 12 | 695 | 50 |
| 13 | 538 | 78 |

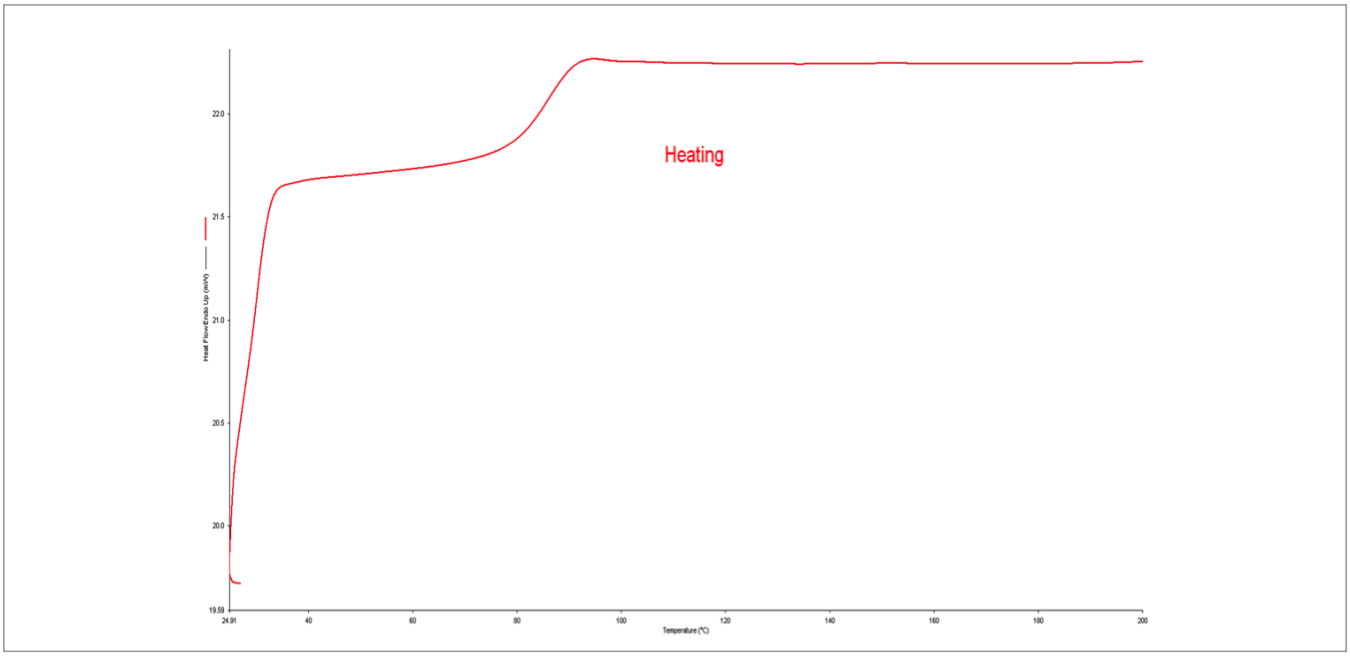
Polystyrene (PS) Applications
- Food packaging
- Electronics housing
- Insulation
- Disposable dining utensils

# Pure PS Analysis

## TGA:




## DSC:

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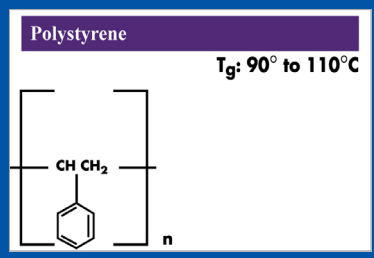
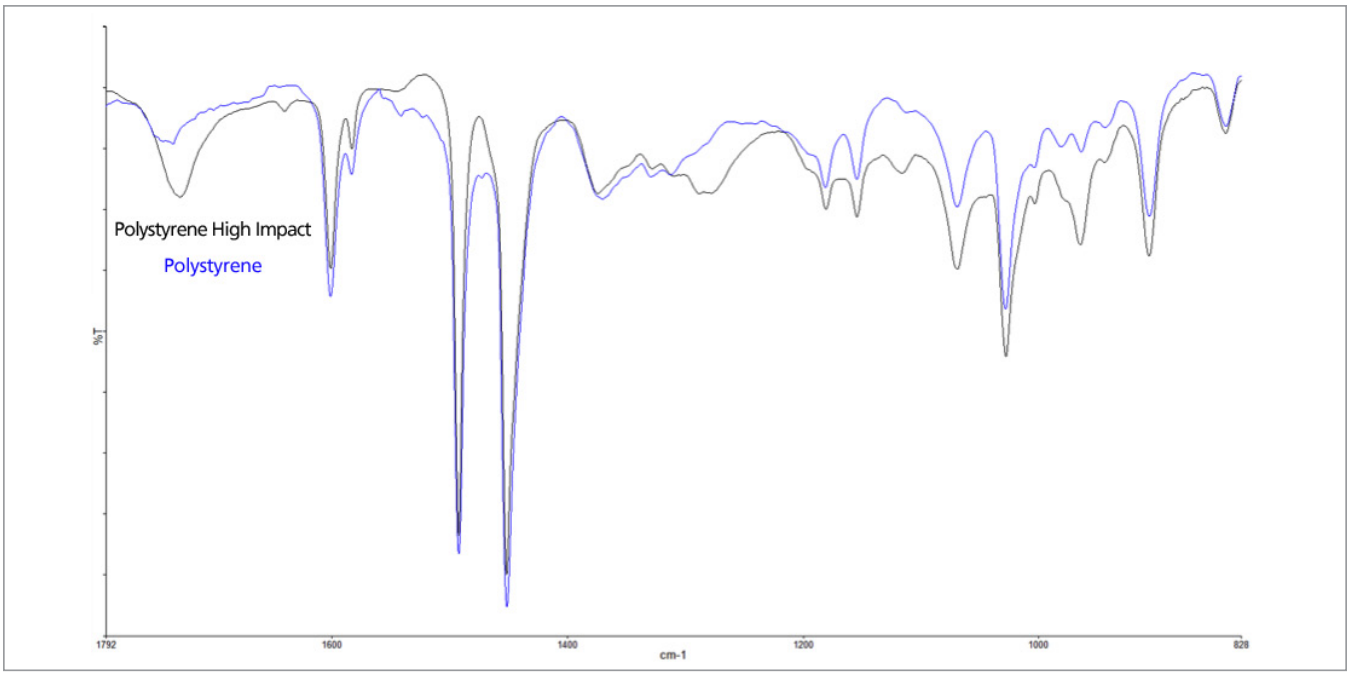
APPLICATION BRIEF

# Code 6b
# Polystyrene

## Comparing HIPS to PS by FT-IR




### Method:

Spectrum Two FT-IR
- 4000-450 $cm^{-1}$
- 4 $cm^{-1}$ resolution
- 4 scans

Polystyrene (PS) Applications
- Food packaging
- Electronics housing
- Insulation
- Disposable dining utensils

# Comparing HIPS to PS by FT-IR

## Discussion:

High Impact Polystyrene is fairly difficult to separate from Polystyrene. One has to focus on the finger print region shown above. One of the best ways to separate the two is through PerkinElmer's proprietary Compare algorithm.

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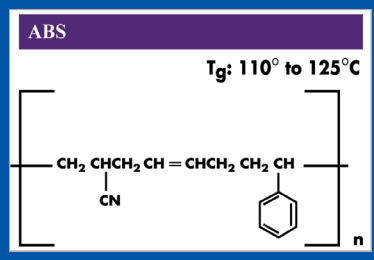
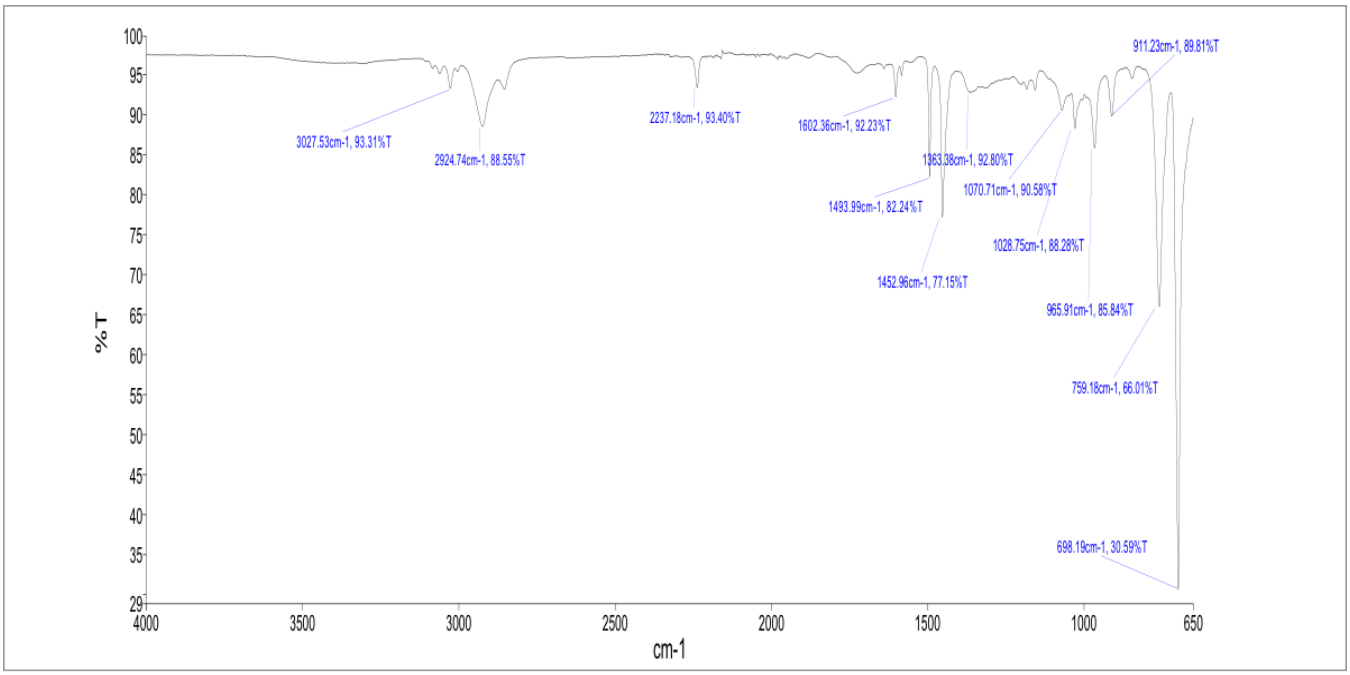
APPLICATION BRIEF

# Pure ABS Analysis

## Code 7a i
## ABS




## FT-IR:




| PEAK NAME | X (CM$^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 3028 | 93 |
| 2 | 2925 | 89 |
| 3 | 2237 9 | 3 |
| 4 | 1602 | 92 |
| 5 | 1494 | 82 |
| 6 | 1453 | 77 |
| 7 | 1363 | 93 |

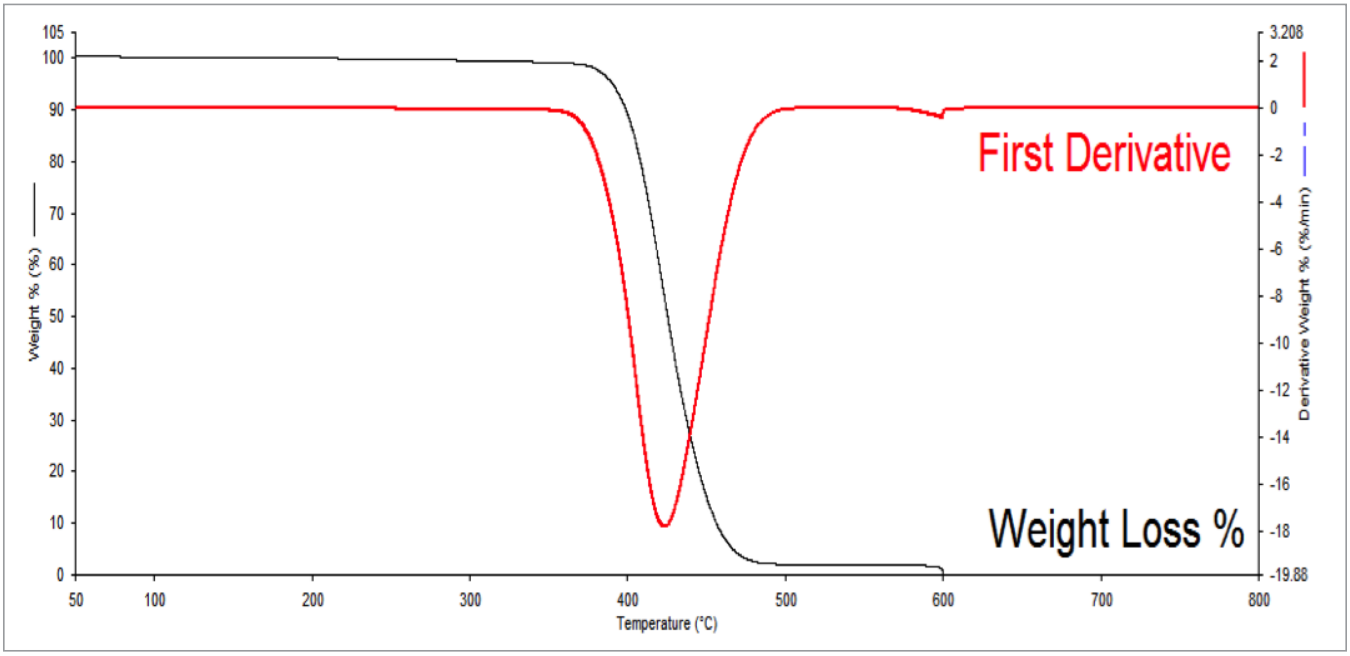
| PEAK NAME | X (CM$^{-1}$) | Y (%T) |
|---|---|---|
| 8 | 1071 | 91 |
| 9 | 1029 | 88 |
| 10 | 966 | 86 |
| 11 | 911 | 90 |
| 12 | 759 | 66 |
| 13 | 698 | 31 |

Acrylonitrile Butadiene Styrene (ABS) Applications

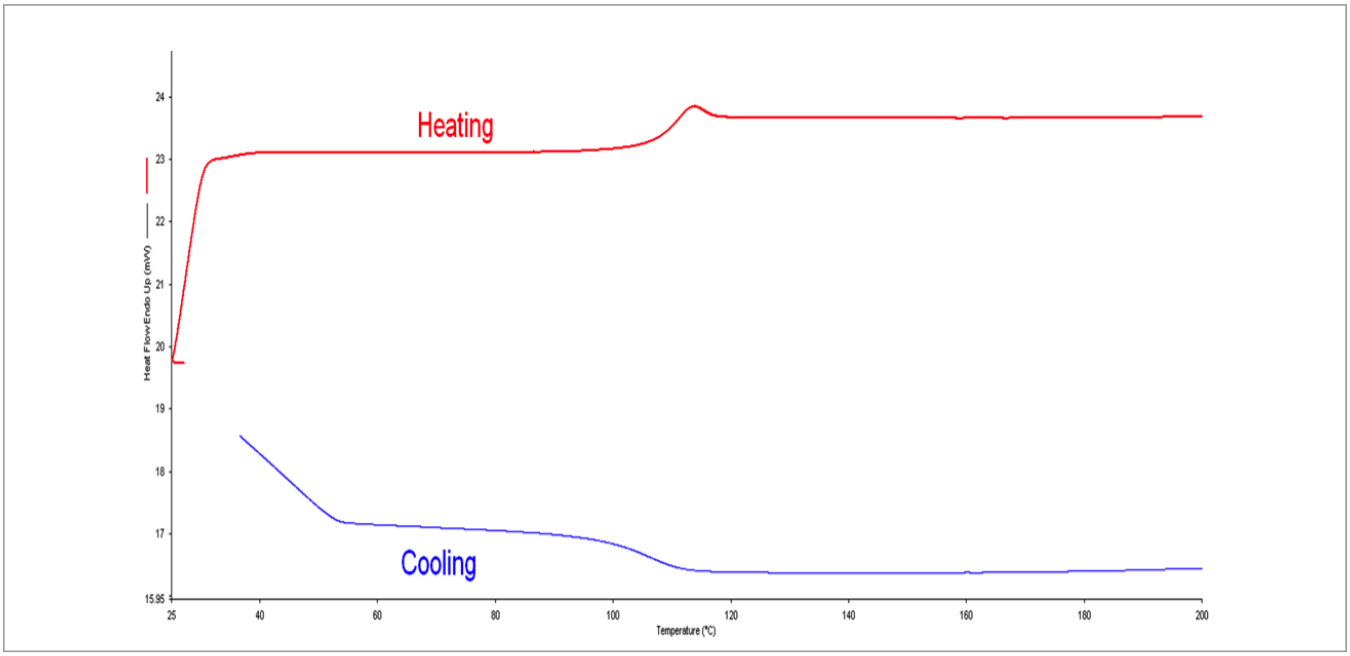
- Automotive
- Protective Equipment
- Toys
- Electronics Housing

# Pure ABS Analysis

## TGA:




## DSC:

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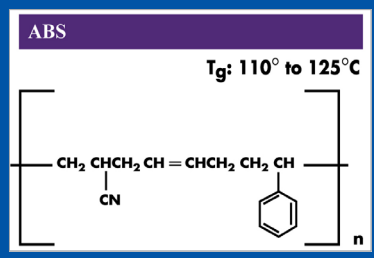
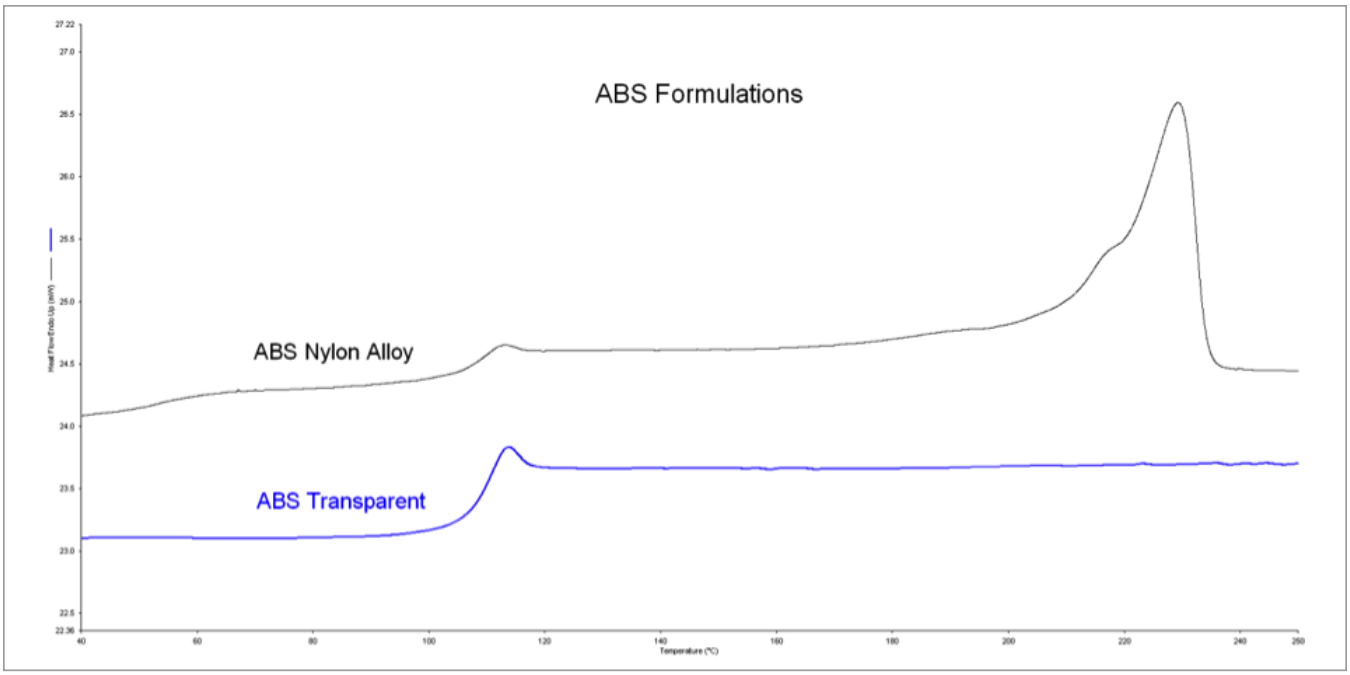
APPLICATION BRIEF

# Detecting ABS Nylon Formulations by DSC

## Code 7a ii
## ABS

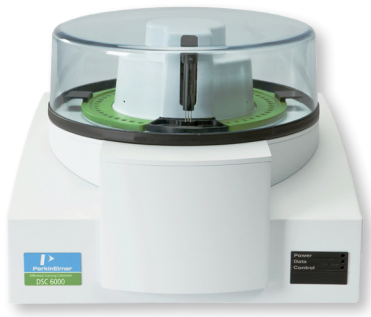



### Method:

DSC 4000 or 6000
Nitrogen 20 mL $min^{-1}$

1. Heat from -50.00 °C to 200.00 °C at 10.00 °C $min^{-1}$
2. Hold for 2.0 min at 200.00 °C
3. Cool from 200.00 °C to -50.00 °C at 10.00 °C $min^{-1}$
4. Hold for 2.0 min at -50.00 °C
5. Heat from -50.00 °C to 200.00 °C at 10.00 °C $min^{-1}$



Acrylonitrile Butadiene Styrene (ABS) Applications

- Automotive
- Protective Equipment
- Toys
- Electronics Housing

# Detecting ABS Nylon Formulations by DSC

## Discussion:

There are many different Acrylonitrile Butadiene Styrene (ABS) formulations. Typically, they are characterized by their upper glass transition. In this particular case, a blend of nylon and ABS is detected.

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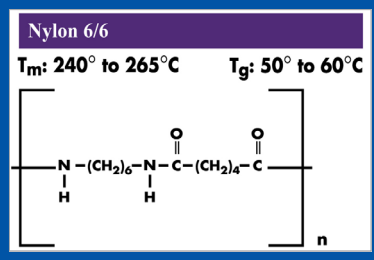
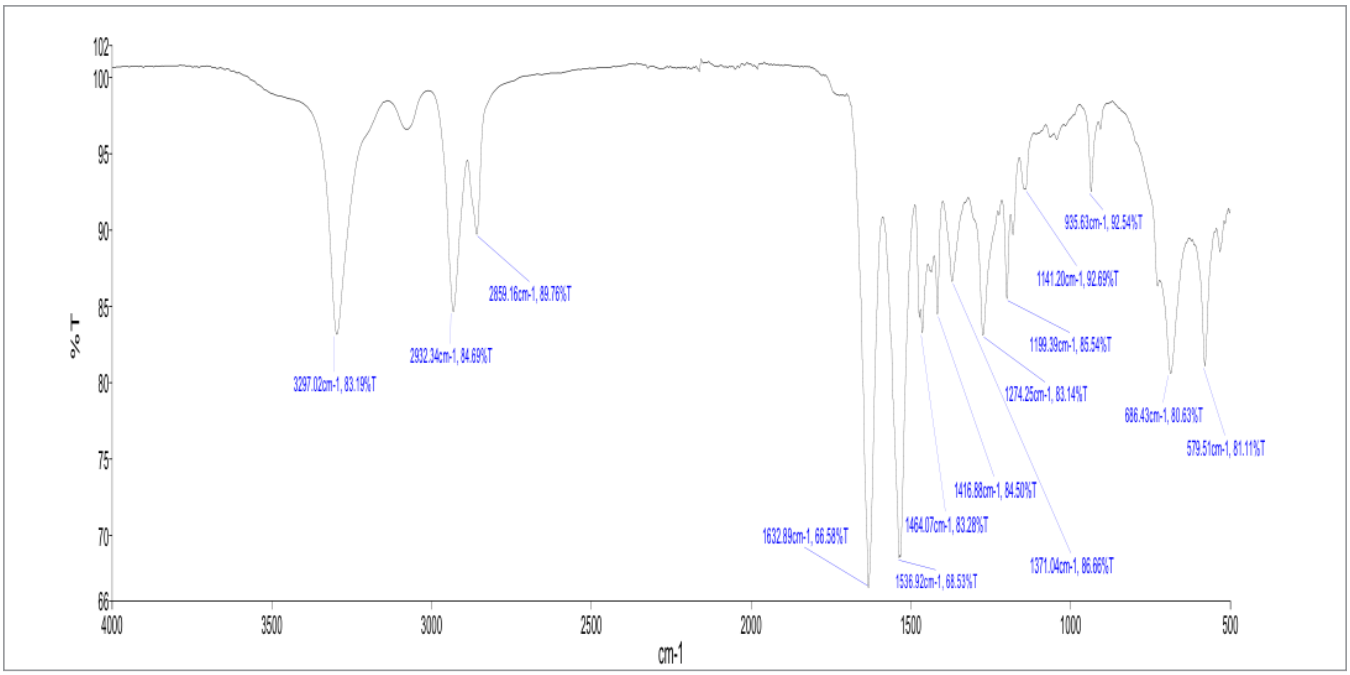
APPLICATION BRIEF

## Pure Nylon Analysis

# Code 7b i
# Nylon




## FT-IR:



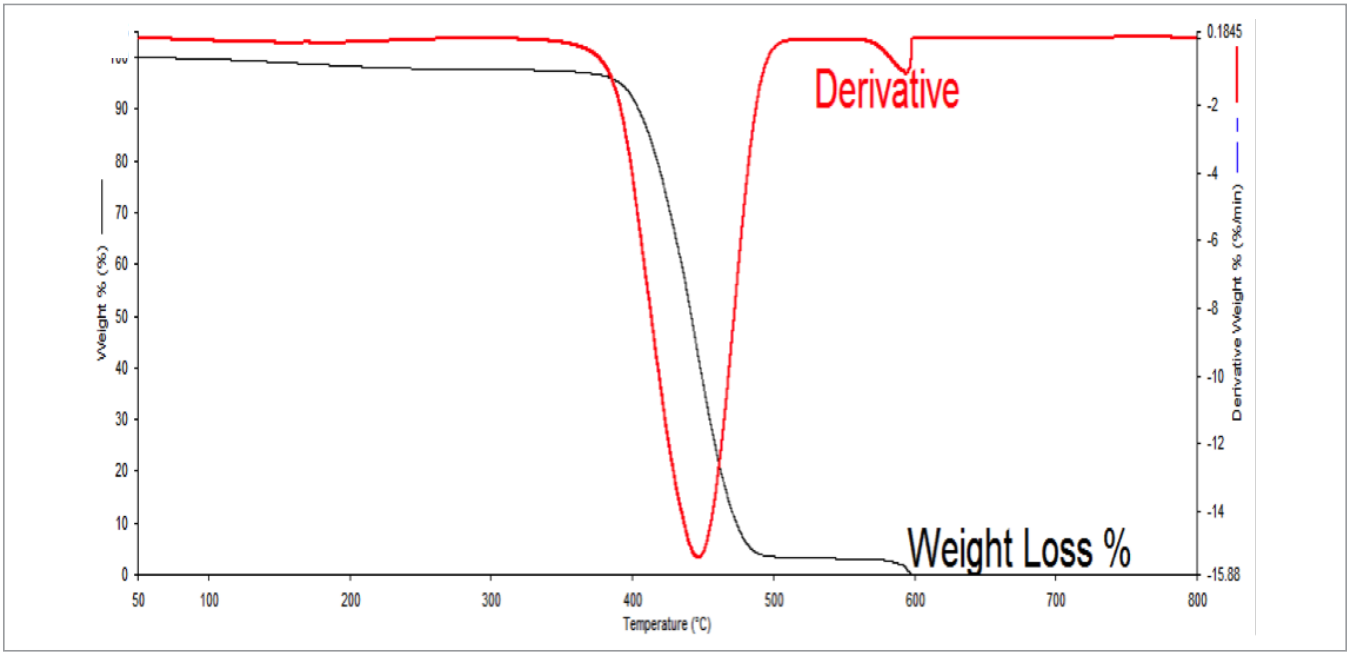

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 3297 | 83 |
| 2 | 2932 | 85 |
| 3 | 2859 | 90 |
| 4 | 1633 | 67 |
| 5 | 1537 | 69 |
| 6 | 1464 | 83 |
| 7 | 1417 | 85 |

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 8 | 1371 | 87 |
| 9 | 1274 | 83 |
| 10 | 1199 | 86 |
| 11 | 1141 | 93 |
| 12 | 936 | 93 |
| 13 | 686 | 81 |
| 14 | 580 | 81 |

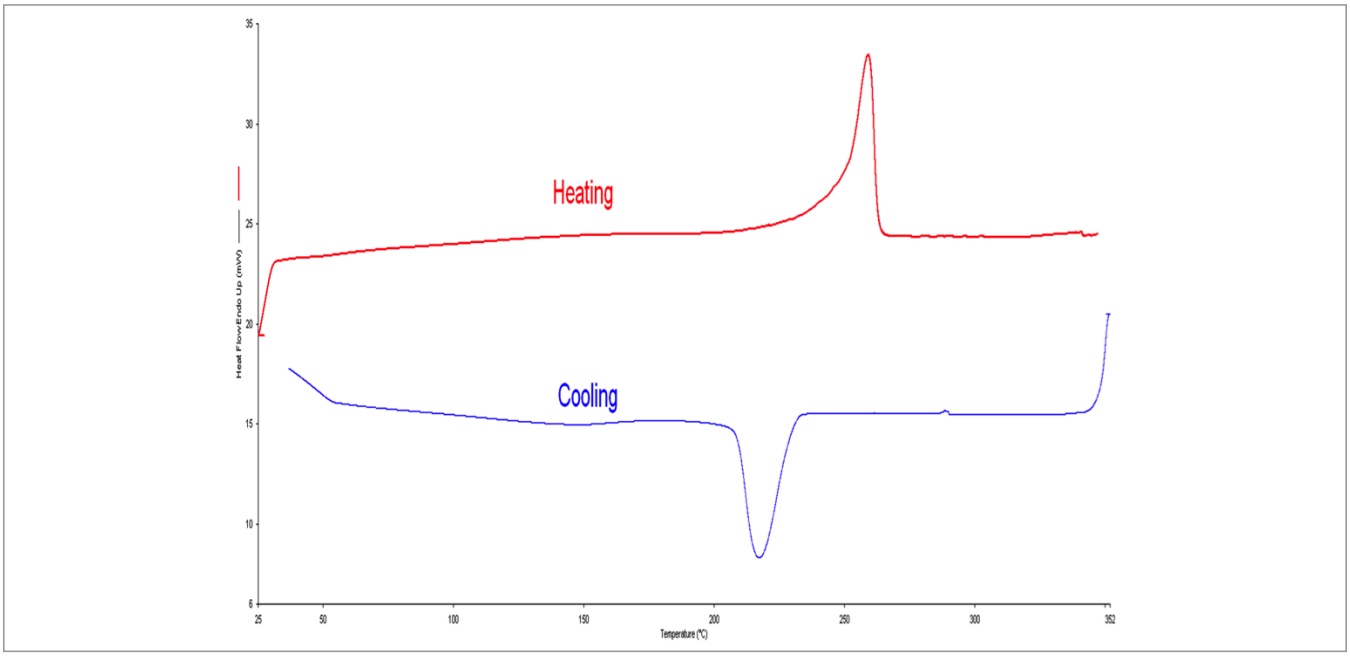
Nylon Applications
- Fibers/Clothing
- Automotive
- Pipes/Tubing
- Machine Parts

# Pure Nylon Analysis

## TGA:




## DSC:

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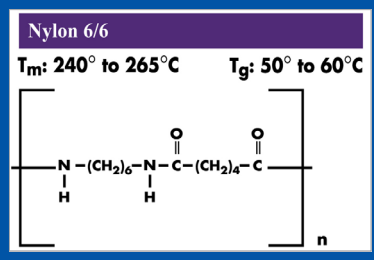
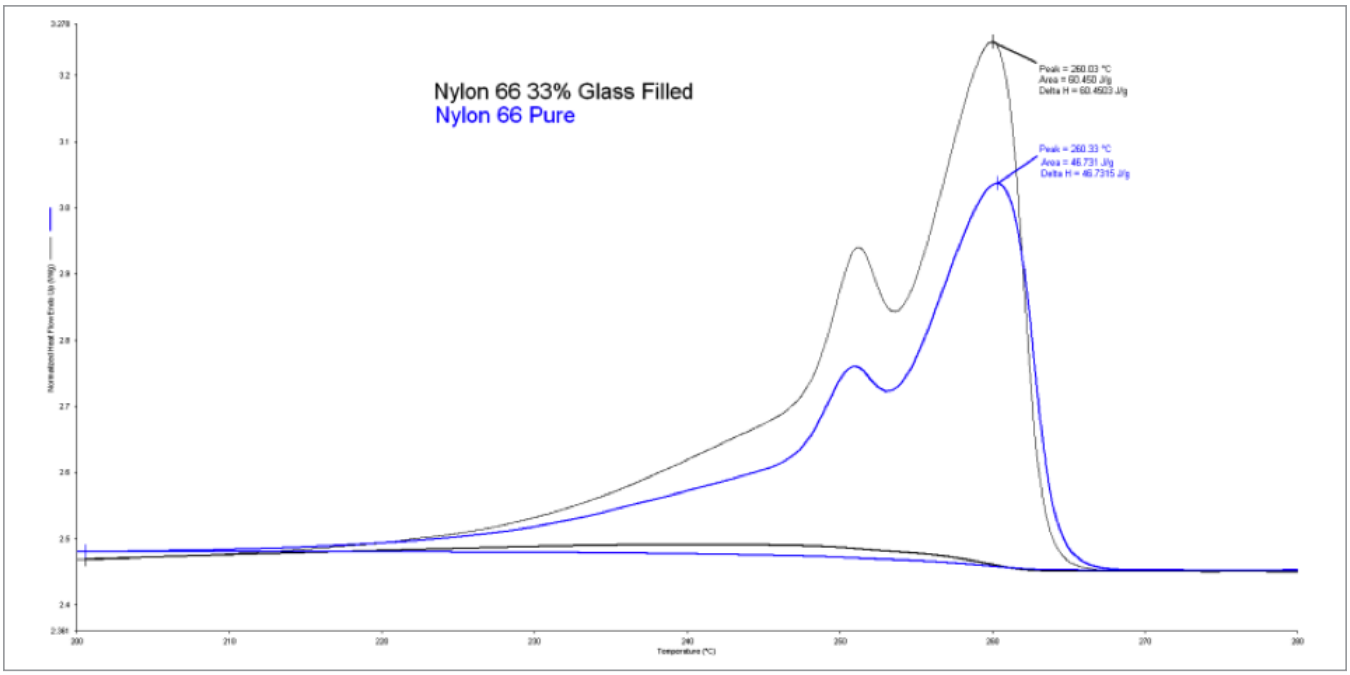
APPLICATION BRIEF

# Filled Nylon Comparison by DSC

## Code 7b ii
## Nylon

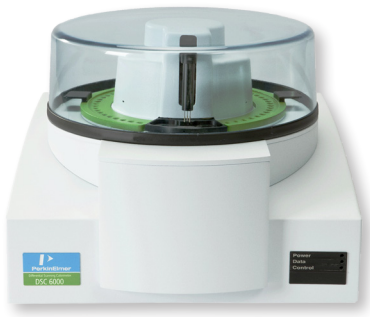



### Method:

DSC 4000 or 6000
Nitrogen 20 mL $min^{-1}$

1. Heat from 25.00 °C to 290.00 °C at 10.00 °C $min^{-1}$
2. Hold for 2.0 min at 290.00 °C
3. Cool from 290.00 °C to 25.00 °C at 10.00 °C $min^{-1}$
4. Hold for 2.0 min at 25.00 °C
5. Heat from 25.00 °C to 290.00 °C at 10.00 °C $min^{-1}$



Nylon Applications

- Fibers/Clothing
- Automotive
- Pipes/Tubing
- Machine Parts

# Filled Nylon Comparison by DSC

## Discussion:

By using DSC, one can approximate the polymer content by using a known standard to ratio against or by generating a concentration versus enthalpy calibration curve.

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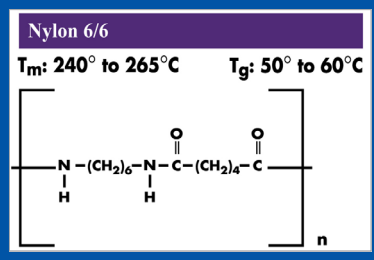
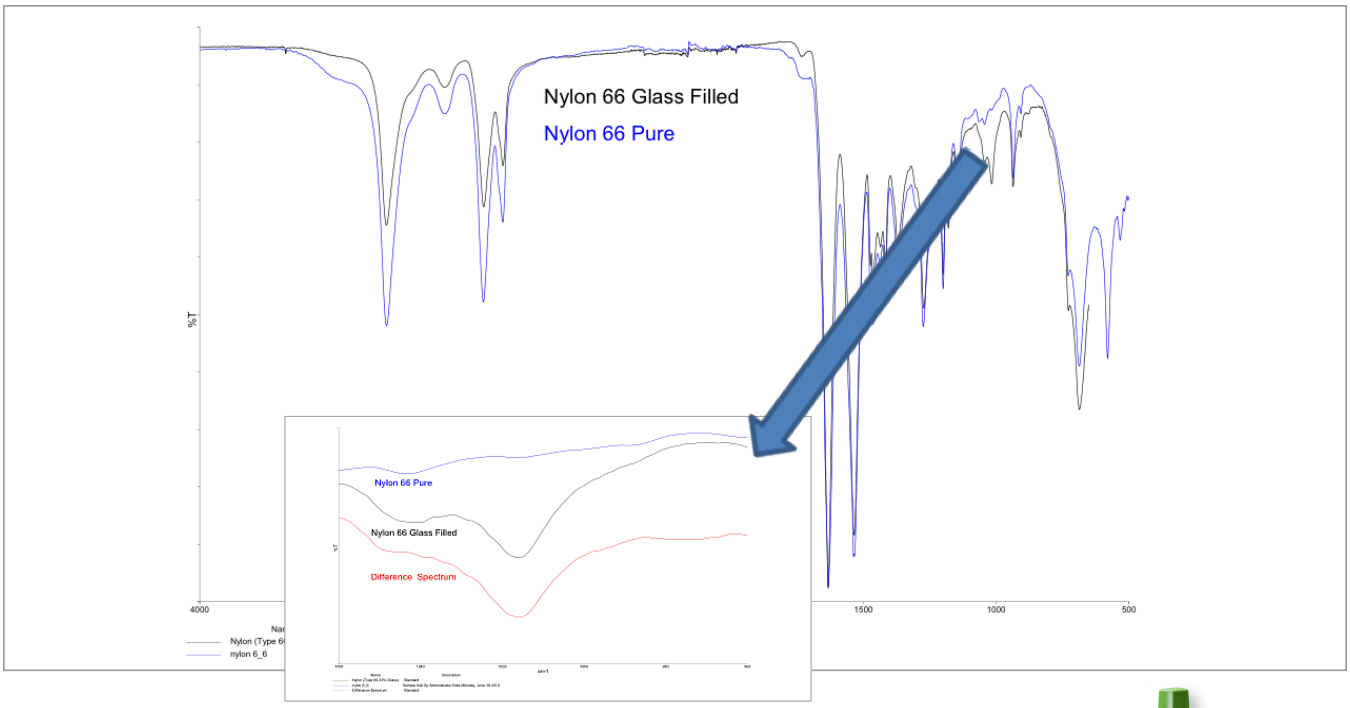
APPLICATION BRIEF

# Filled Nylon Comparison by FT-IR

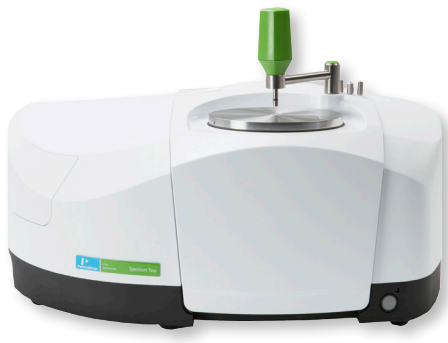
## Code 7b iii
## Nylon




**Method:**

Spectrum Two FT-IR
- 4000-450 $cm^{-1}$
- 4 $cm^{-1}$ resolution
- 4 scans



Nylon Applications
- Fibers/Clothing
- Automotive
- Pipes/Tubing
- Machine Parts

# Filled Nylon Comparison by FT-IR

## Discussion:

The addition of inorganic fillers to thermoplastics is common. This is either to reduce cost, improve stability, or to enhance mechanical properties. In this particular case, nylon has a glass filler to enhance its strength. There is one peak associated with the glass filling, around 1019 $cm^{-1}$. Using PerkinElmer's Spectral Subtraction algorithms, it's possible to identify and isolate additive packages (see insert).

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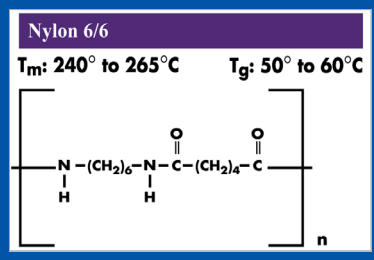
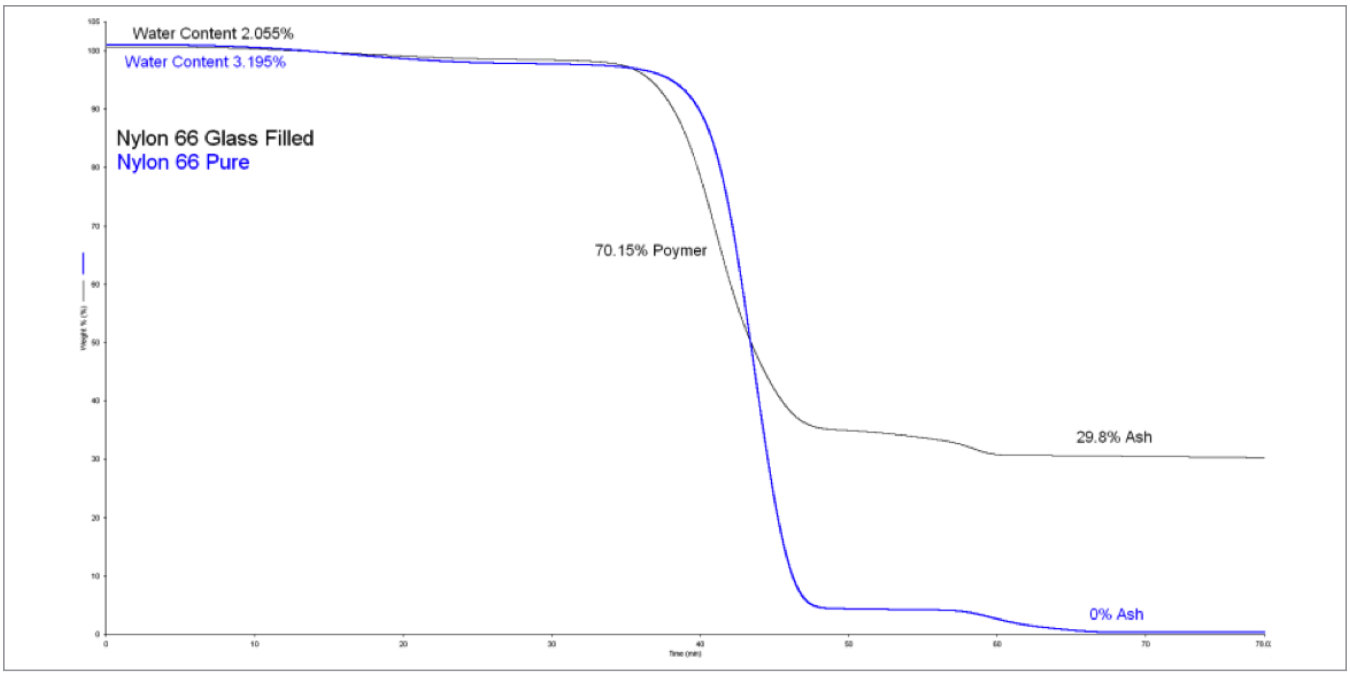
APPLICATION BRIEF

# Filled Nylon Comparison by TGA

## Code 7b iv Nylon




### Method:

TGA 4000/STA 6000
Nitrogen purge 20 mL min$^{-1}$
1. Heat 30 to 900 °C at 10 °C min$^{-1}$
   a. Swap to air at 600 °C at 30 mL min$^{-1}$

Nylon Applications
- Fibers/Clothing
- Automotive
- Pipes/Tubing
- Machine Parts

# Filled Nylon Comparison by TGA

## Discussion:

One of the most effective ways to quantify fillers is through the TGA. In this example, pure nylon is over laid with a filled nylon sample. Since pure nylon has 0% ash, all of the ash can be accounted for in the unknown as glass filler. The identity of the filler can be determined through the use of FT-IR or ICP-OES on the ash.

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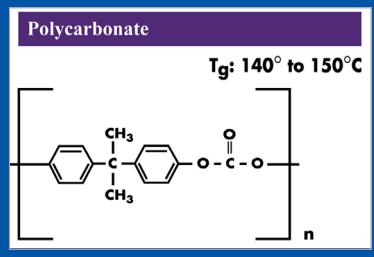
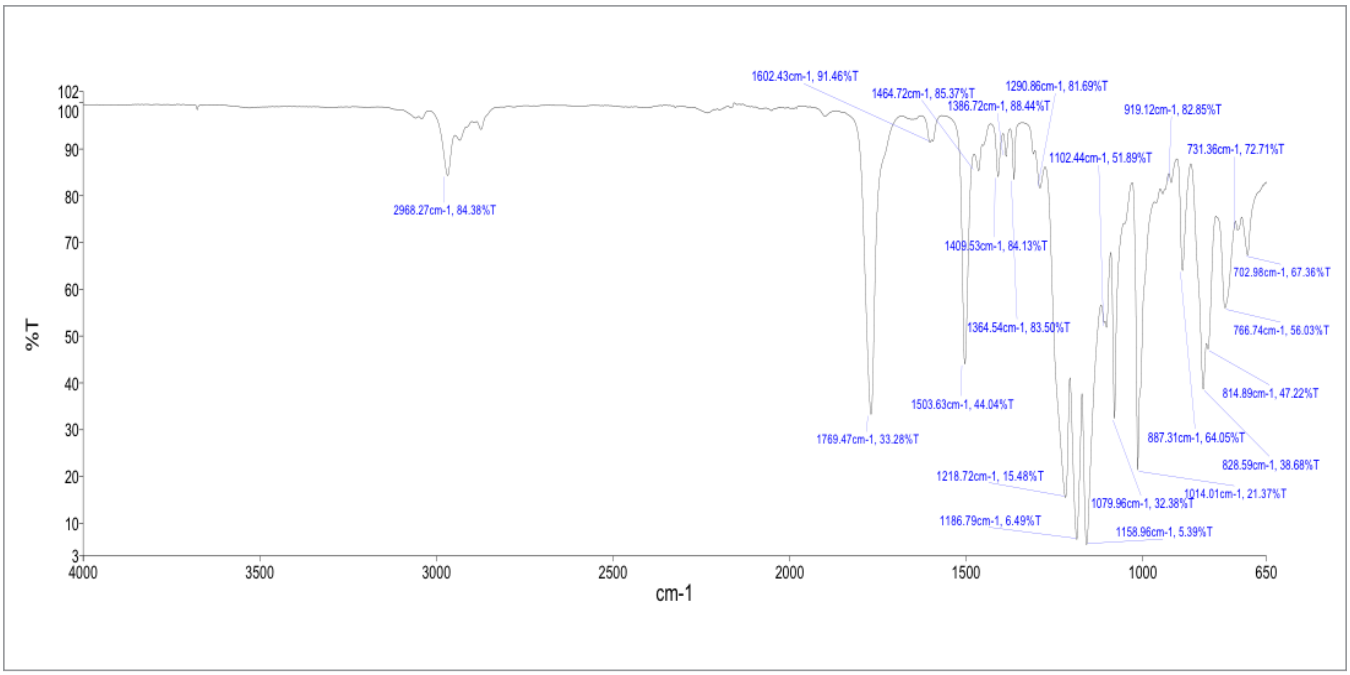
APPLICATION BRIEF

## Pure PC Analysis

# Code 7c i
# Polycarbonate




## FT-IR:



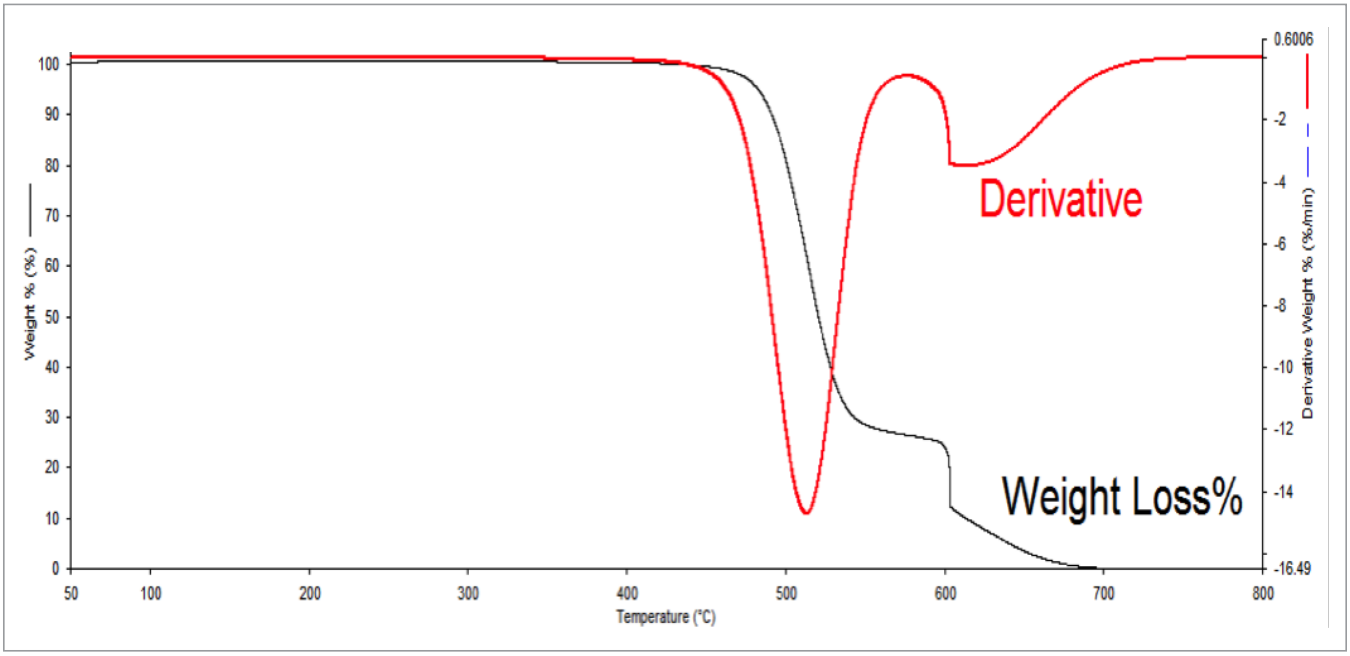

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 2968 | 84 |
| 2 | 1769 | 33 |
| 3 | 1602 | 91 |
| 4 | 1504 | 44 |
| 5 | 1465 | 85 |
| 6 | 1410 | 84 |
| 7 | 1387 | 88 |
| 8 | 1365 | 84 |
| 9 | 1291 | 82 |
| 10 | 1219 | 15 |
| 11 | 1187 | 6 |

| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 12 | 1159 | 5 |
| 13 | 1102 | 52 |
| 14 | 1080 | 32 |
| 15 | 1014 | 21 |
| 16 | 919 | 83 |
| 17 | 887 | 64 |
| 18 | 829 | 39 |
| 19 | 815 | 47 |
| 20 | 767 | 56 |
| 21 | 731 | 73 |
| 22 | 703 | 67 |

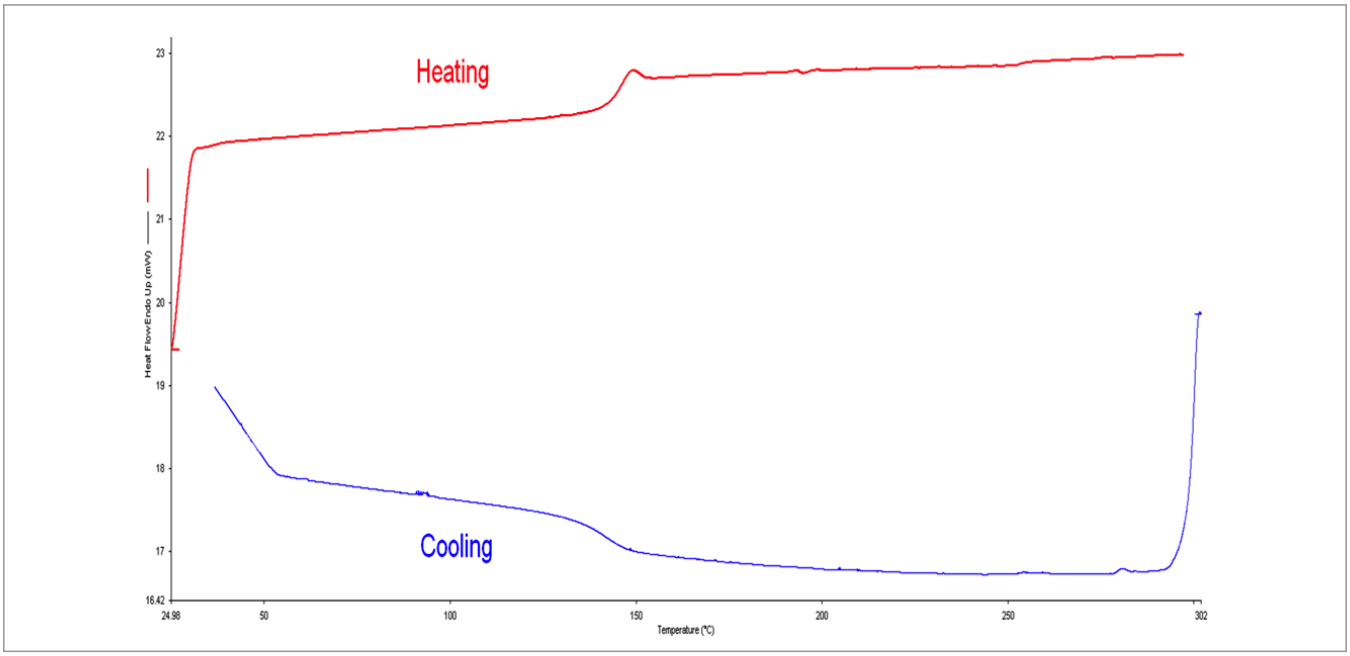
Polycarbonate Applications
- Safety Materials
- Food and Beverage Containers
- Artificial Glass
- Electronic Housing

# Pure PC Analysis

## TGA:




## DSC:

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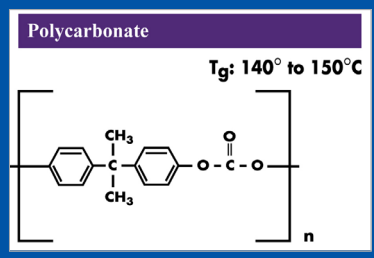
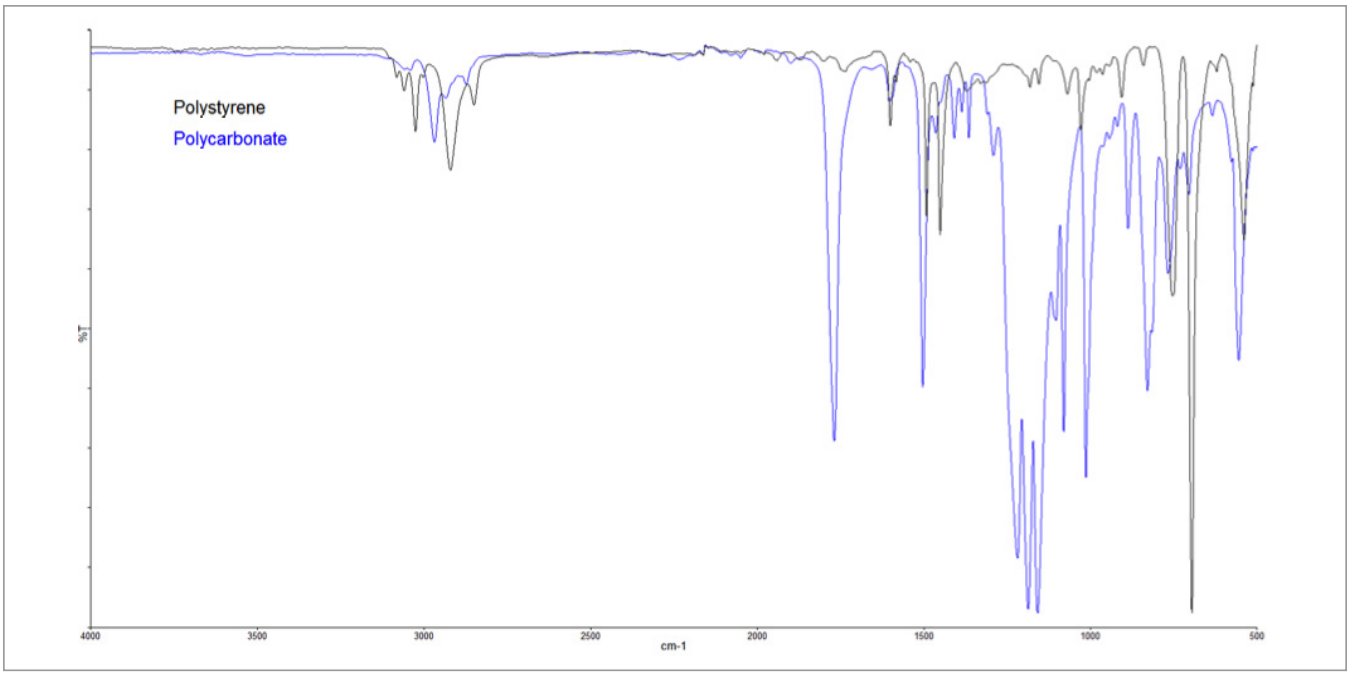
APPLICATION BRIEF

# Code 7c ii Polycarbonate

## Comparing PC to PS by FT-IR




### Method:

Spectrum Two FT-IR
- 4000-450 $cm^{-1}$
- 4 $cm^{-1}$ resolution
- 4 scans

Polycarbonate Applications
- Safety Materials
- Food and Beverage Containers
- Artificial Glass
- Electronic Housing

# Comparing PC to PS by FT-IR

## Discussion:

Polycarbonate (PC) and Polystyrene (PS) are often confused with each other when in rubbish or waste. FT-IR can easily identify the two. The primary bands of interest are the carbonyl at approximately 1700 $cm^{-1}$. By checking for this band, one can quickly differentiate Polycarbonate from other high clarity thermoplastics.

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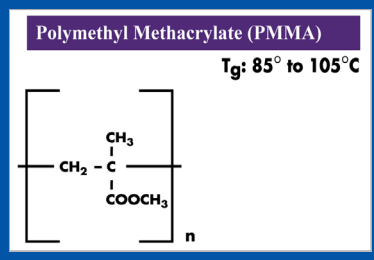
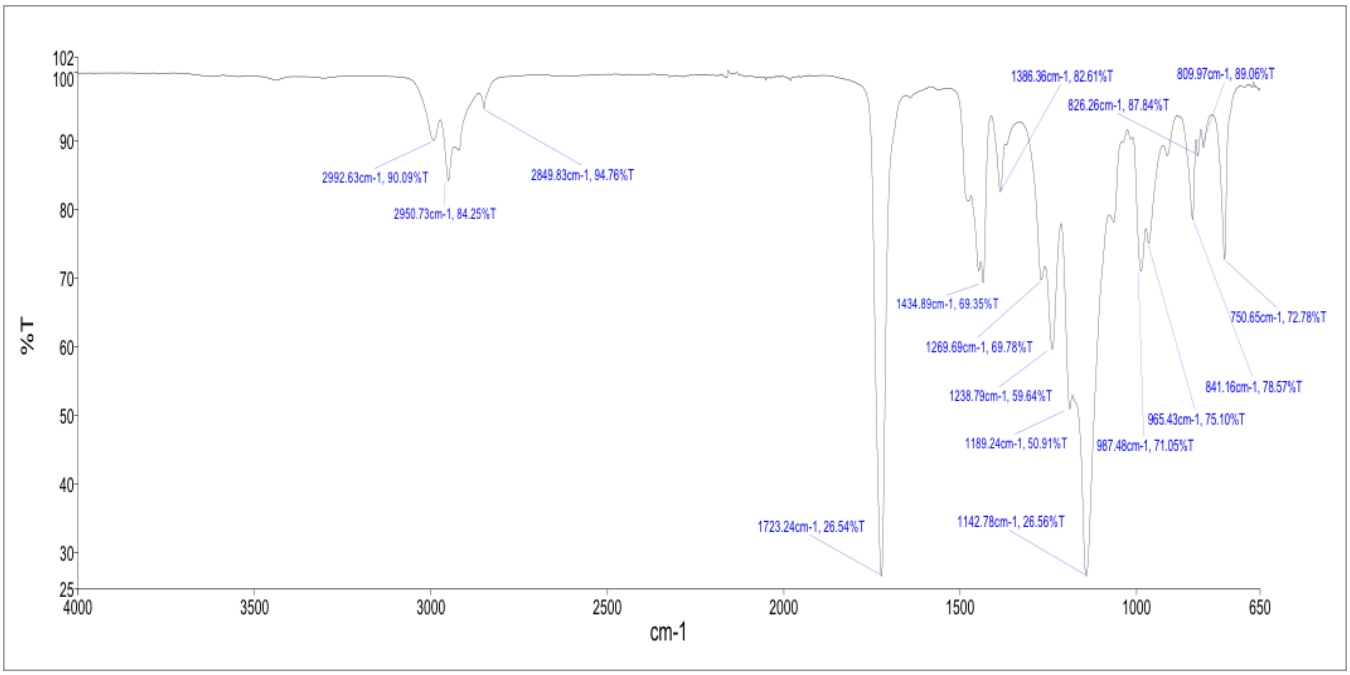
APPLICATION BRIEF

## Pure PMMA Analysis

# Code 7d i
# Poly(Methyl Methacrylate)




## FT-IR:




| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 1 | 2993 | 90 |
| 2 | 2951 | 84 |
| 3 | 2850 | 95 |
| 4 | 1723 | 27 |
| 5 | 1435 | 69 |
| 6 | 1386 | 83 |
| 7 | 1270 | 70 |
| 8 | 1239 | 60 |
| 9 | 1189 | 51 |

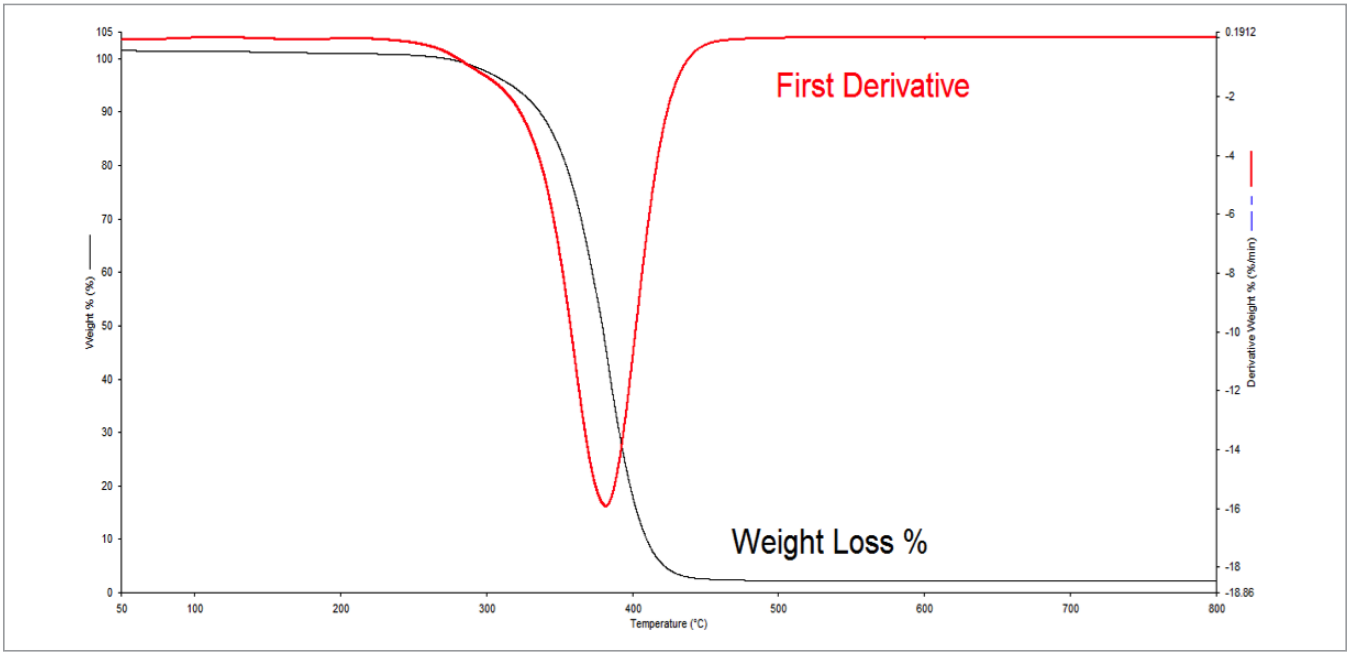
| PEAK NAME | X ($CM^{-1}$) | Y (%T) |
|---|---|---|
| 10 | 1143 | 27 |
| 11 | 987 | 71 |
| 12 | 965 | 75 |
| 13 | 841 | 79 |
| 14 | 826 | 88 |
| 15 | 810 | 89 |
| 16 | 751 | 73 |

Poly(Methyl Methacrylate) (PMMA) Applications

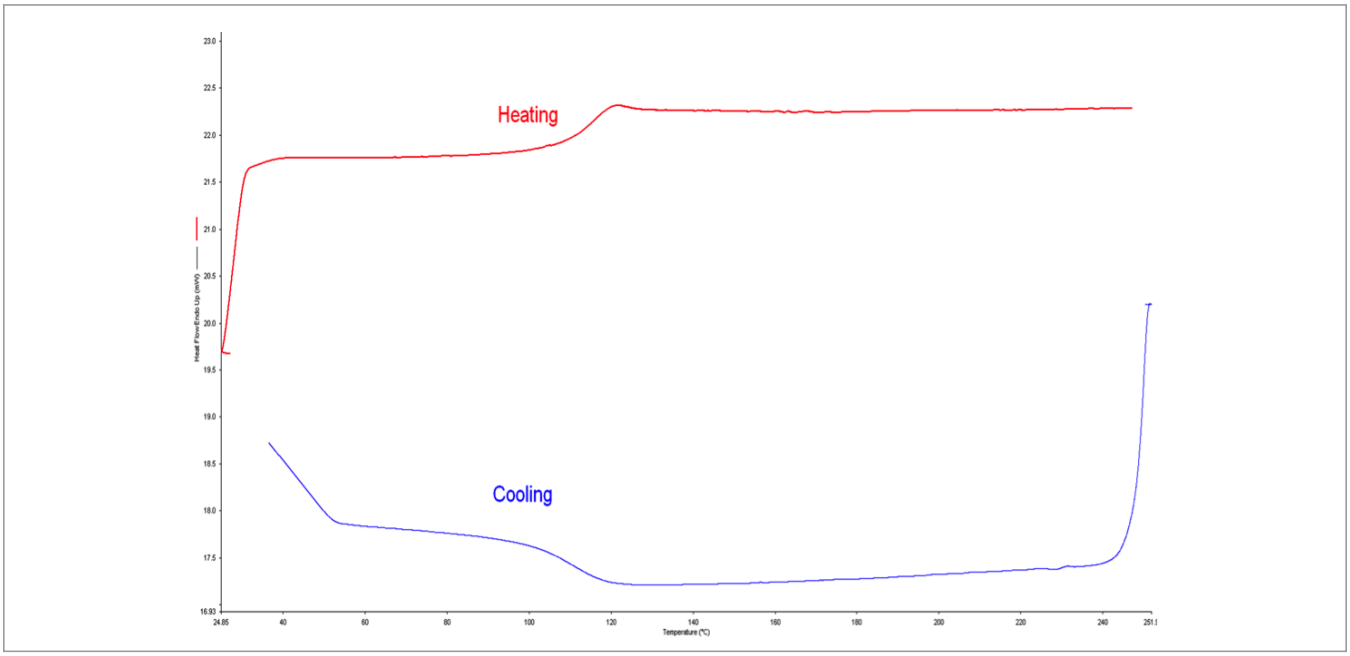
- Artificial Glass
- Electronics
- Automotive
- Construction

# Pure PMMA Analysis

## TGA:




## DSC:

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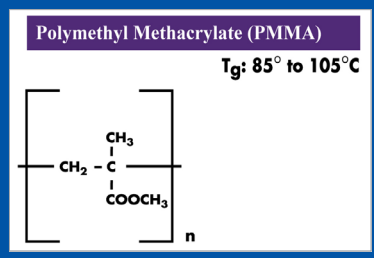
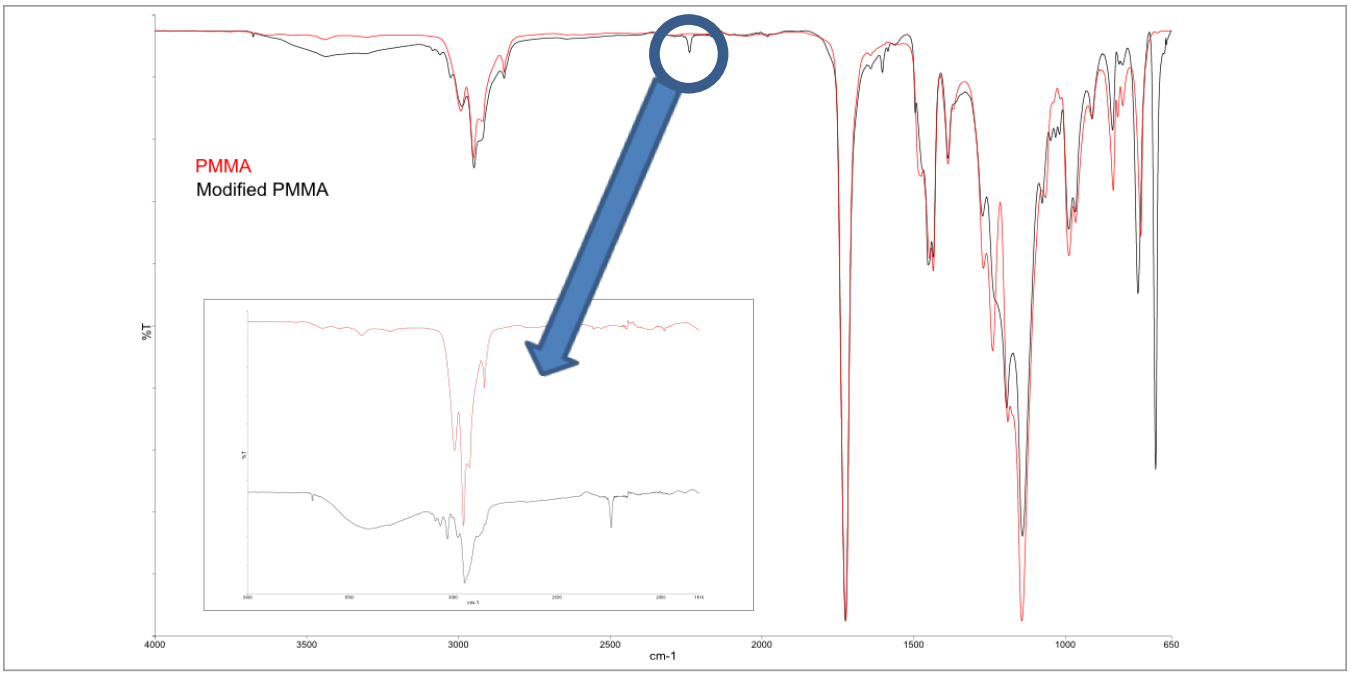
011538_01

APPLICATION BRIEF

# Code 7d ii
# Poly(Methyl Methacrylate)

## Comparing PMMA to MPMMA by FT-IR




**Method:**

Spectrum Two FT-IR
- 4000-450 $cm^{-1}$
- 4 $cm^{-1}$ resolution
- 4 scans

Poly(Methyl Methacrylate) (PMMA) Applications
- Artificial Glass
- Electronics
- Automotive
- Construction

# Comparing PMMA to MPMMA by FT-IR

## Discussion:

FT-IR can be used to look for differences between Polymethyl Methacrylate (PMMA) samples. In this example, impact-resistant PMMA is compared to standard PMMA.

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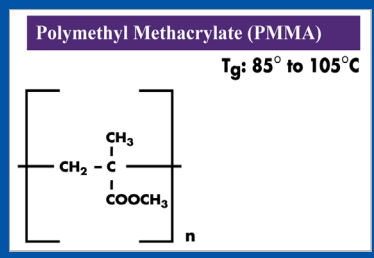
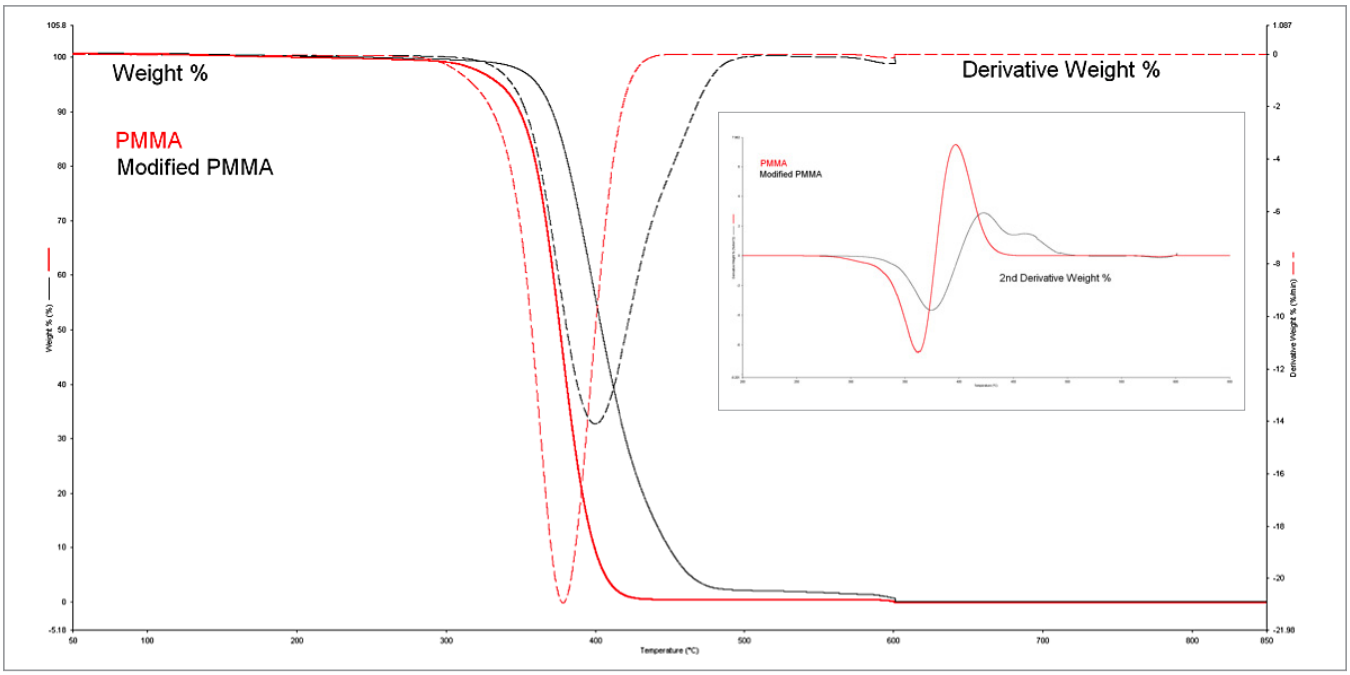
011539_01

APPLICATION BRIEF

# Comparing PMMA to MPMMA by TGA

## Code 7d iii
## Poly(Methyl Methacrylate)




**Method:**

TGA 4000/STA 6000
Nitrogen purge 20 mL $min^{-1}$
1. Heat 30 to 900 °C at 10 °C $min^{-1}$
   a. Swap to air at 600 °C at 30 mL $min^{-1}$

Poly(Methyl Methacrylate) (PMMA) Applications
- Artificial Glass
- Electronics
- Automotive
- Construction

# Comparing PMMA to MPMMA by TGA

## Discussion:

When a polymer system is modified, a certain amount of thermal stability can be added. Utilizing TGA, one can determine the change in thermal stability based on the formulation. The first and second derivatives are helpful in this measurement.

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011540_01

## Recycled Polymer Compendium – User Guide

The information in this document is put forth as a tool to guide users in the analysis of polymer materials. All of the data presented was collected with PerkinElmer instruments on new/clean resins. Remember that actual formulations encountered in the marketplace have been optimized to enhance the properties of the polymer. Some enhancements could be color, structural, cost, UV-protection etc. This guide does not attempt to address all additives and enhancements utilized in the seven codes of polymers. It does, however, provide methods and techniques to guide users through the detection and possible identification of unknown enhancements. Below, the user will find a brief overview of the steps required to reproduce the data within the compendium as well as precautionary information pertaining to the three instrument classes employed in this study.

### Instrument and Sample Guidelines

**1. Fourier Transform Infrared Spectroscopy (FT-IR – Spectrum Two)**

#### Preparing the Instrument

To begin the analysis by FT-IR, several items need to be prepared:

- Verify that the instrument has successfully passed its "Ready Checks," specifically the contamination check.
- If the contamination check fails, clean the diamond ATR crystal using an appropriate solvent and delicate wipe. This author is a proponent of 91% isopapanol and disposable laboratory wipes.
- Allow enough time for the solvent to evaporate then re-run the contamination check.

After the instrument is verified as being ready to operate, no more ready checks are required that day, unless the user's SOP specifies otherwise.

#### Preparing for Analysis

Included in the compendium media device is an FT-IR library file. This library should be added to the list of searchable items. A detailed description of how to set up library files can be obtained within the "Spectrum 10 Tutorials" found in the "Help" Menu.

Within the same folder as the library file, resides a ".SET" file. This file should be imported into the instrument configuration page. See Figures 1 and 2 for import and activation tips.

Now that the instrument is configured, a "background scan" is required. Verify that the pressure arm tip is not near the diamond face before pressing the scan "Background" button (Figure 3).

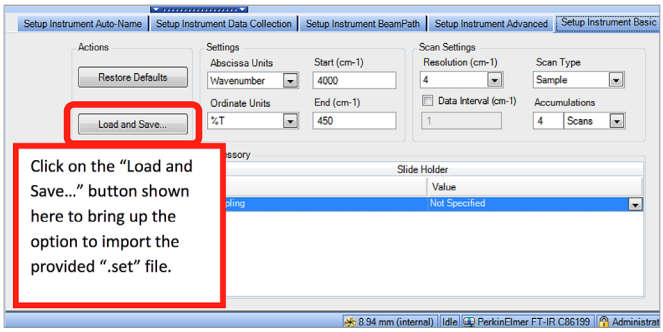


*Figure 1.* "Setup Instrument Basic" tab. This is the area where the methods are configured.

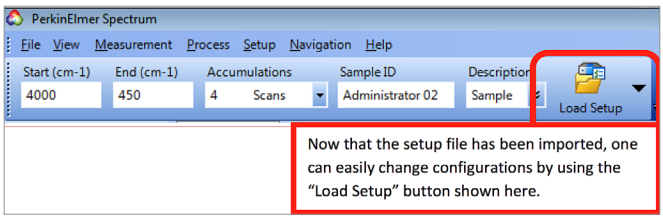


*Figure 2.* Instrument Toolbar. Highlighted is the button which allows for the rapid configuration of the instrument.


*Figure 3.* Identifying the "Scan," "Background" and "Scanalyze" buttons on the instrument control panel.

#### Preparing the Sample

Using a clean sharp knife (razor blade or hobby knife), cut the sample such that it has at least one flat side, but two are preferred so the faces are parallel to each other (Figure 4). Avoid grinding or "dry" sanding the sample as the friction/heat can alter the material's behavior.

Place the newly cut flat face on the diamond and lower the pressure arm down until contact is achieved, but do not tighten past contact.

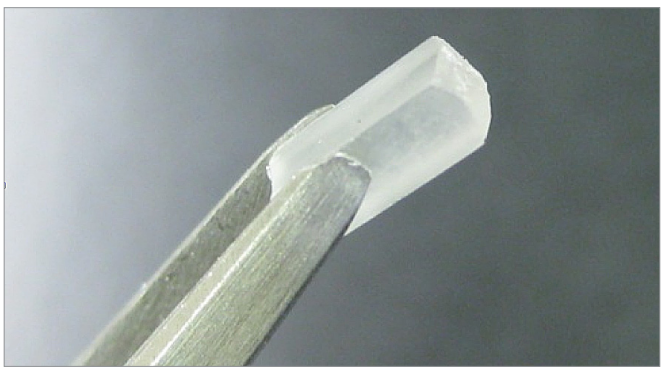

*Figure 4.* Example of a polymer sample cut to have a flat top and bottom, for measurement by both DSC and FT-IR. This sample began as a "chip" (depending on the resin used, the common name used for unprocessed polymer may vary between "flake, chip, pellet, etc.)

### Measurement

Press the "ScanAlyze" button then highlight "Scan and Search" (Figure 3). This action will put the instrument into what is called "Preview Mode". Once the real-time display becomes active, begin applying pressure to the sample. Watching the force meter, continue turning the dial until 90-100 counts is displayed. If peaks are clearly observed, and the largest one is greater than 90%T, then press the "Scan" button (Figure 3) to begin data acquisition and the search. If the sample does not exceed 90%, then remove the force from the sample and reposition, recut, etc., until the sample does meet this requirement. Under no circumstance should additional force be applied beyond 100 counts as this could alter the sample crystal structure.

### Data Analysis

As the data is collected, the search process is already underway. By the time the experiment is complete, the best match will be displayed. In an ideal situation, the best hit will have a score of "1.0", however, this will never occur. Typically, a score greater than 0.90 is considered a reasonable hit (Figure 5). The closer the hit index is to 1, the more confident the analyst can be in the result. If the best hit is less than 0.9, it becomes time to analyze the sample in a more analytical fashion. Within each of the 7 code folders, a data file is provided of the pure polymers. Open the suspected data file and overlay it with the unknown sample and look for differences (Section 7,d, ii in the compendium).

The more common inorganic additives always appear in the same general regions. Over time, the user will be able to spot them rapidly. If the spectra are wildly different, then it may be possible that a polymer blend is present. If a polymer blend or filled material is suspected, then the laboratory should have a set protocol in place for the accepting or rejecting of such polymer lots.

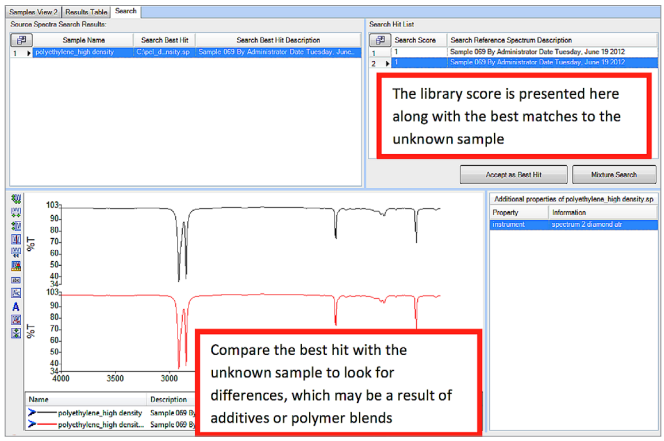


*Figure 5.* Example output from the "ScanAlyze" analysis.

### Cautionary Information

- All data collected in this compendium was obtained utilizing a specific set of instrument parameters. Included in the shipping materials is a Setup file. The extension of this file is ".SET" this includes all of the conditions for the execution of the experiments used in the provided library.
- The measurements were all obtained using a Diamond-attenuated total reflectance accessory (UATR), KBr sample compartment windows and a DTGS detector. Any deviation from this configuration could result in a deviation from the presented data.
- Peak labels are provided for reference only. Peaks can shift due to the pressure applied using the force arm, percent crystallinity, and additives in the polymer. Do not expect your peaks to be in the exact same position as those listed in the compendium.

## 2. Differential Scanning Calorimetry (DSC 4000/DSC 6000)

### Preparing the Instrument

To begin the analysis of polymers by DSC, the instrument must be properly configured and equilibration reached. Activate an inert purge gas such as nitrogen (N2) (Figure 6) with a delivery pressure between 25-30 psi. Proper gas pressures are typically set by a PerkinElmer-authorized service representative at the point of installation. Activate Pyris™ software and verify that the instrument/software connection is achieved. Set the instrument temperature to 50 °C using the "GoTo" temperature button (Figure 6). Activate the cooling device attached to the instrument.

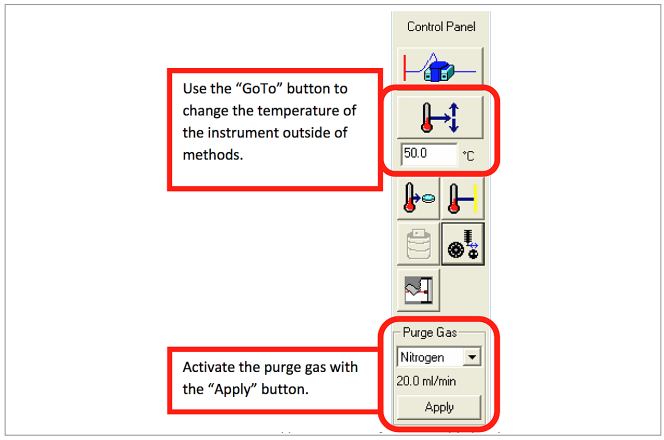


*Figure 6.* Instrument control bar in Pyris software. Highlighted is the "GoTo" temperature and "Apply" purge gas sections.

## Notes for Cooling Devices

If water-cooling is utilized, verify the coolant is at the proper level and no contamination is visible by inspecting the tank. If an intracooler is being used, make sure that the transfer line is not kinked or pinched. Note that once you activate the intracooler, the transfer line cannot be moved. In both cases, the cooling device needs to be powered on for a minimum of 1.0 hour prior to making any measurements. It is not uncommon for some test laboratories to wait for 1.5 hours prior to starting any measurement.

## Notes on Calibration

It is good practice to "check" the instrument calibration on a regular basis and recalibrate as needed. Typically, DSCs are checked and calibrated with the following reference materials: indium (In), zinc (Zn), tin (Sn), and/or gallium (Ga). This policy should be outlined in the laboratory SOP (standard operating procedure).

## Method Editor Configuration

Each of the polymers covered in this compendium have a specific DSC method associated with them. Some of the methods require an intracooler to achieve the best results. Pick the method which best suits the suspected material in its respective folder on the compendium media device. Feel free to alter the temperature ranges so long as the maximum temperature stays below the degradation temperature of the sample. Do not alter the number of steps or the heating rates of the steps between samples, as otherwise, the user will not be able to compare the data generated. If the maximum safe temperature is unknown, then a TGA should be implemented.

With the method editor as the active window (Figure 7) go to File → Open and navigate to the media device to locate the method.

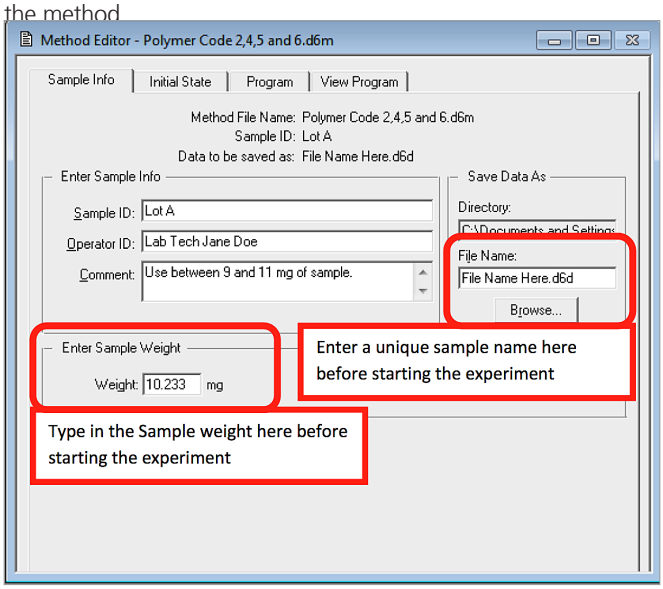


*Figure 7.* Example of fully-populated DSC Method Editor.

## Sample Preparation

Depending on the instrument configuration, either standard aluminum pans or autosampler aluminum pans should be utilized. The sample should be prepared in accordance with the following guidelines:

- Cut the sample such that it will neatly fit inside of the sample pan. The sample should have one flat side that touches the bottom of the pan (Figure 4).
- The sample mass should be between 5-10 mg, and reported to the second decimal place, such as "5.51 mg." The laboratory SOP should identify the exact target sample mass and the desired precision. Once the mass has been properly determined, the sample should be encapsulated with the correct cover and crimping device.
- Using a delicate laboratory wipe, clean any fingerprints or sample which may have accumulated on the sample pan.

## Preparing for the Measurement

Using the "GoTo" temperature button, lower the instrument temperature to a safe level, (less than 30 °C) (Figure 6). Load the sample into the instrument. If an autosampler is equipped, then let the instrument place the sample, otherwise use forceps. Verify that the furnace covers have been returned to their correct locations.

## Executing the Measurement

On the "Sample Information" tab of the Method Editor (Figure 7), enter the mass of the sample to be analyzed. In "Save Location", type in a name which best describes the sample. In the Notes sections, enter in any pertinent information, which will assist future analysts in determining the goal of this project. Figure 7 provides a nice example of how the fields in the "Sample Information" tab should be filled in. The population of these text fields should be outlined in the lab SOP. With a single mouse click, press the "Start" button (Figure 8). Typically, the measurement will take greater than 30 minutes — allow enough time for the experiment to complete. If the measurement is stopped prematurely, the progress to that point will be saved.

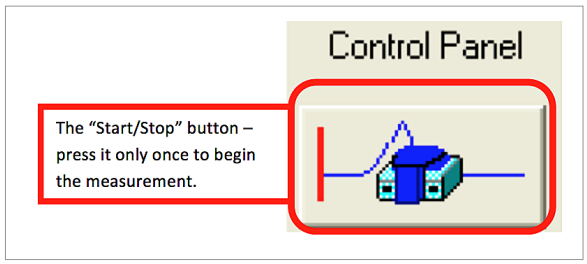


*Figure 8.* DSC "Start/Stop" button.

### Analyzing the Data

Each of the seven codes has data files associated with them in their respective folders. The goal of the analysis will be to answer three idealized questions:

1) Is this the correct polymer?

2) Is it a mixture of polymers?

3) How much of the polymer is present?

These questions can be answered quickly and easily using the following methodology.

To begin, the data files for the unknown sample and a pure material must be prepared for analysis. This process is called "separating the curves." Using the "Curves" menu, select the experiment intervals that will be analyzed. Some DSC files are provided to assist in this analysis. Look to the media device to load these samples.

Typically, the second heating is the curve of interest. Using the "Shift Curve" button found on the toolbar, adjust the segments so that the second heat curves for the unknown material and for the reference polymer overlap each other. This may also require use of the Slope button to align them as closely as possible. Now compare the peaks and glass transitions in these two curves and ask, "Are they close to each other?" If the answer to that question is "no," then look for another polymer and repeat the process.

If the identity can't be confirmed, it is now time to answer the question "could this be a mixture of polymers?" Overlay the polymers which may constitute the observed mixture. An example of this process can be found in section 7, a, ii of this compendium.

"What percent of the unknown can be attributed to the polymer?" This is an estimation to make after determining the identity/ies of the unknown sample. This can be achieved by two methods, the melting enthalpy and/or the delta Cp. The enthalpy of melting the unknown should be less than or equal to that of the pure polymer. Simply divide the enthalpy of the unknown by that of the standard, and multiply by 100 to get the approximate polymer percent in the unknown. An example for this can be found in section 7, b, ii of this compendium.

In the example mentioned above, the suspected glass filler was 33%, however, TGA analysis revealed the % glass to be 29% with about 2.5% moisture (7, b, iv). It is not uncommon for errors in formulation to occur. As can be seen here, the utilization of DSC and TGA has proven that the sample is significantly less filled than what was implied.

If no melting peak is observed for the polymer, the glass transition (Tg) is to be utilized in this measurement. Typically, this number will be less than 1.0 and displayed to the 3rd decimal place. If this is the only means of estimating the percent of polymer present, then great care must be taken to get the sample mass as accurate as possible. The calculation is identical to that of the melting.

### Notes on Quantitation

A great deal of caution should be expressed at this point as this will not be an exact value, only a reasonable approximation. The addition of fillers, additives, and polymer blends can greatly influence this measurement. If exact values are required, then modified DSC, TGA, and/or FT-IR methods are available but are not covered within the guide.

### Cautionary Information

- All data collected in this compendium was obtained utilizing a specific set of instrument parameters. Included in the shipping materials are "method" files — the file extension is ".D6m". Each of the seven codes has its own unique method file.
- The samples were encapsulated in standard aluminum DSC pans.
- Sample masses were between 8.5-10.5 mg.
- All peaks displayed in this compendium are for reference purposes only. Note that peaks can shift not only in temperature but also intensity due to the addition of additives or the blending of polymers. Use the peak positions and areas as a guide to identify the polymer and its approximate concentration.

## 3. Thermogravimetric Analysis (TGA 4000/STA 6000)

### Preparing the Instrument

To begin the analysis of polymers by TGA, the instrument must be properly configured and equilibration reached. Activate the inert purge gas, nitrogen (Figure 9). The delivery pressure for all incoming gases should be between 25-30 psi (this is normally configured by your PerkinElmer-authorized service representative at the point of installation.) Verify that the instrument is powered on. Activate Pyris software and verify that the instrument/software connection is achieved. Set the instrument temperature to 30 °C using the "GoTo" temperature button (Figure 9). Activate the cooling device attached to the instrument.

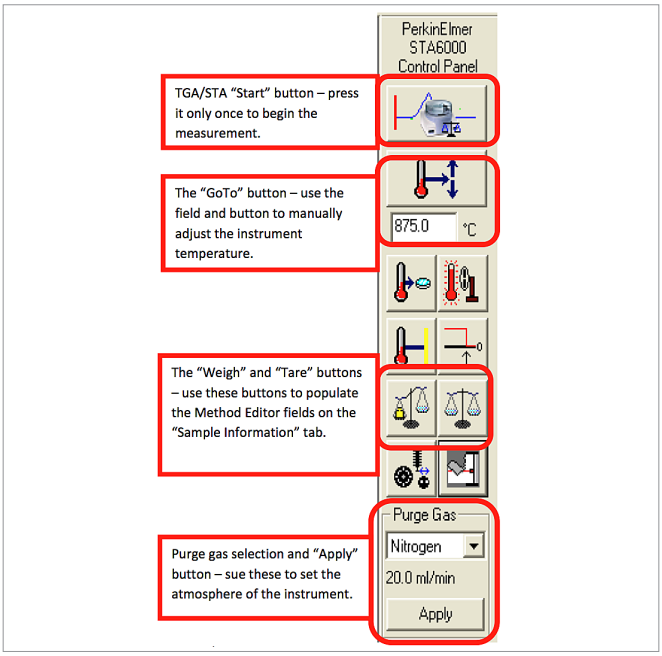


*Figure 9.* TGA/STA control panel.

## Notes for Cooling Devices

Water-cooling is utilized for both the STA 6000 and TGA 4000. Make sure that the coolant level is correct and no contamination is visible by inspecting the tank. The cooling device needs to be powered on for a minimum of 1.0 hour prior to making any measurements. It's not uncommon for laboratories to wait for 1.5 hours prior to starting any measurement.

## Notes on Calibration

It is good practice to "check" the instrument calibration on a regular basis and recalibrate as needed. This policy should be outlined in the laboratory SOP (standard operating procedure).

## Method Editor Configuration

Within the compendium media device, two TGA methods are provided. One method is generic and the other is optimized for carbon black containing polymers. Choose the one that best meets the requirements of the sample. Make the Method Editor the active window, then go to File → Open and navigate to the media device to locate the method (Figure 10).

## Sample Preparation

Preparing samples for a TGA is typically and easy endeavor, however, care must be taken to do so in a repeatable manner. The laboratory should establish a set of protocols for the preparation of samples. The SOP should address the following:

- Is the sample presented as a single piece, chopped, or ground up? Care should be taken when cutting, chopping, or grinding to not introduce foreign material.
- Is the sample placed in a platinum or alumina sample crucible? Platinum is typically the preferred option, but cost tends to push laboratories to alumina. Alumina sample pans will work fine for most polymers, but they do have a limited lifetime.
- Does the sample contain carbon black? If the sample has this additive, then more time may be required to complete the burn off, or oxygen as a purge gas may be required. Feel free to increase the hold times and gas flow rates in the provided methods.
- Does the sample contain inorganic fillers? If the sample contains such additives, then a considerable amount of ash will remain in the TGA pan – make sure to clean the residue out properly.

## Setting Up the Instrument

Make the Method Editor the active window (Figure 10). In "Save Location", enter a name that best suits the unknown sample and populate the remaining fields in accordance with the laboratory SOP.

Using the "GoTo" temperature button (Figure 9), set the instrument temperature to 30 °C. Verify the instrument is stable before tarring or weighing any pans/samples. Place an empty sample crucible on the sensor. If the instrument has an autosampler, allow it to insert and remove the crucible. Tare the sensor, then remove the crucible. Never attempt to put a sample into a pan that is still in the instrument. Place the sample into the holder then return it to the sensor. Allow the instrument to stabilize then record the mass of the sample into the method editor (Figures 9 and 10).

## Executing the Measurement

Verify that all of the appropriate fields have been populated as outlined in the lab SOP (Figure 10). With a single mouse click, press the "Start" button (Figure 9). Typically, the measurement will take greater than 45 minutes – allow enough time for the experiment to complete. If the measurement is stopped prematurely, the progress to that point will be saved.

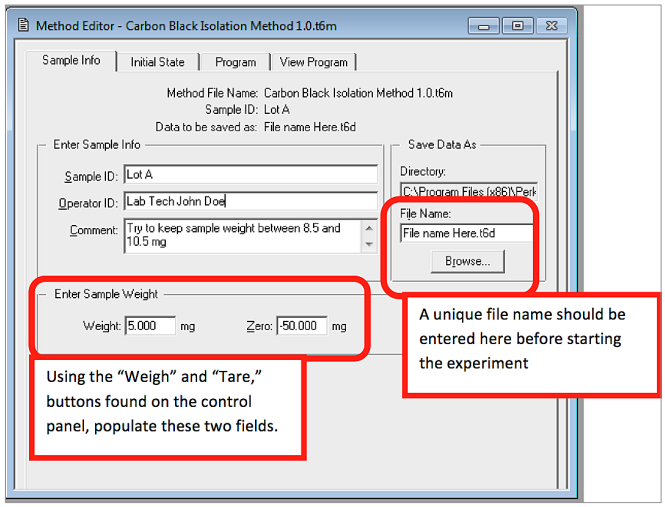


*Figure 10.* Example of fully-populated TGA Method Editor.

## Analyzing the Data

It is this author's preference to view TGA data such that the "X" axis is in Time rather than Temperature when calculating percent weight loss, however, when comparing samples, it's good to overlay them with the "X" axis in temperature. This swap in axis is easily achieved using the "T/t" button on the tool bar. In the spirit of simplicity, all TGA data provided in this compendium is presented with temperature scaling.

In each of the seven code folders, example TGA data files are provided for practice. Many types of each polymer exist. One example would be nylon, which has many forms such as 6, 6/6, 6/12 etc. Should the user require a more robust means of identifying the material, FT-IR or DSC are better choices as additive packages can greatly influence degradation. If the laboratory has a reference material for comparison, add the polymers using the "Add" function in the "File" menu. Utilizing both the Weight % and 1st derivative of the Weight %, verify the identity of the polymer (Figure 11).

Convert the Weight % curves from temperature to time. Remove the 1st derivative curves using the "Remove" button. Note the similarities and differences between pure polymer and the unknown material. Specific attention should be placed on the region after 600 °C as this is the area where carbon black and inorganic fillers are often determined. Using the "Event Mark" and "Step" calculations found in the "Calc" menu, determine moisture, carbon, ash etc. Section 7, b, iv in this compendium shows an excellent example for these calculations.

If an FT-IR is available, it is possible to identify some inorganic fillers such as glass, talc, calcium carbonate, etc. After the TGA has cooled back to a safe temperature, remove the sample crucible and empty the contents on the diamond ATR to collect the spectra (Figure 12). If the identity of the filler is already known, save the FT-IR file so that it can be used for future library searches.

## Cautionary Information

- All data collected in this compendium was obtained utilizing a specific set of instrument parameters. Included in the shipping materials are two "method" files. The extension of the files is either ".t6m" or ".stam". Depending on the polymer system, one method may be more favorable than the other.
- Be consistent in the sample preparation, including particle size, and weights – typically 5-10 mgs of sample is recommended.
- All peaks and steps displayed in this compendium are for reference purposes only. Note that peaks and steps can shift not only in temperature but also intensity due to the addition of additives or the blending of polymers. Use the peak positions of first derivatives and step height as a guide to identify the polymer and its approximate concentrations.

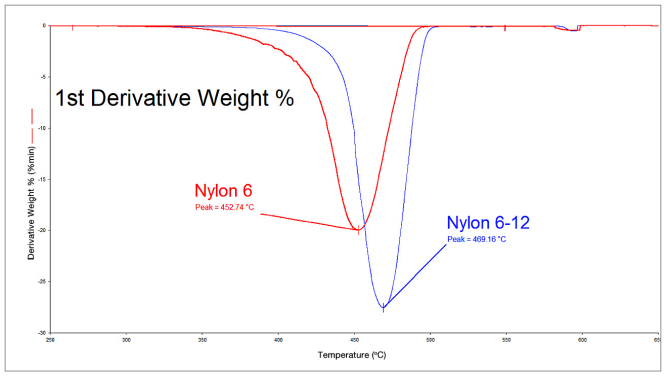


*Figure 11.* Example usage of the 1st derivative plots to determine identity of a polymer. In this case, there is a clear difference between the two forms of nylon.

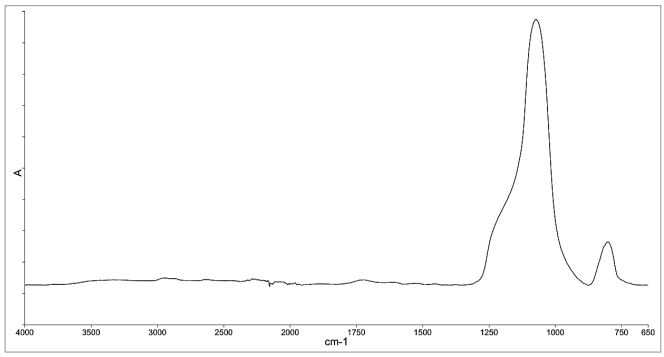


*Figure 12.* FT-IR analysis of the ash remaining in a TGA pan.

## Instrument and Software Training

For a more complete set of instructions on the proper operation of the instruments, contact PerkinElmer for instrument and software training classes which are regularly scheduled throughout the year. Please visit www.perkinelmer.com/training or contact your account manager to learn more about instrument training opportunities.

Learn more about PerkinElmer's analytical solutions at www.perkinelmer.com/polymers

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