

POLYMER RECYCLING COMPENDIUM



Introduction

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 most commonly implemented in the analysis of polymers are:

- Differential Scanning Calorimetry (DSC) - 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.
- Thermogravimetric Analysis (TGA) - 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.
- Fourier Transform Infrared Spectroscopy (FTIR) - first-line measurement to assist in the identification of polymers, fillers, and additives.

DSC, TGA, and FTIR are key techniques to ensure polymer identification, and the quality of the materials received and produced.



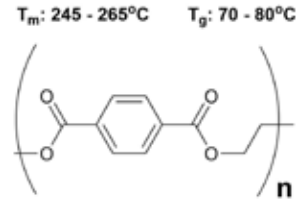
This compendium provides example data and interpretations for the seven codes of recyclable polymers. DSC and TGA raw data files are provided so that the user can easily overlay their data with the expected results for new unprocessed plastics. This allows for a confirmation of identity and assists in the analysis of incoming materials. An FTIR 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.

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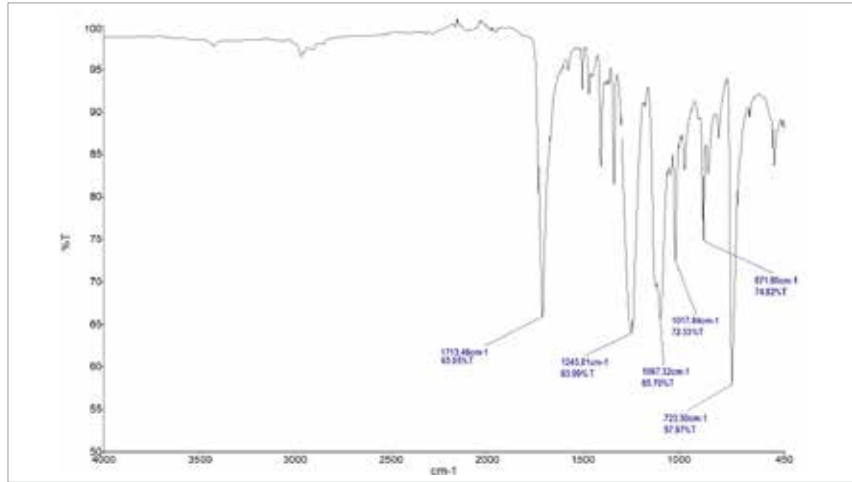
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CODE 1

Poly(ethylene Terephthalate)



FTIR



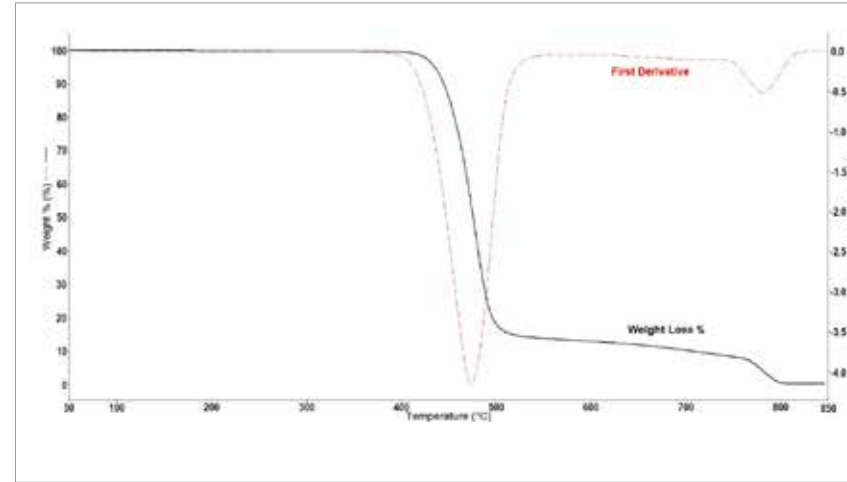
Important FTIR Peak Assignments

Peak Position (cm ⁻¹)	Assignment
1713	C=O Stretch
1245	C-O Stretch
1097	C-O Stretch
723	Aromatic C-H Bend

Polyethylene Terephthalate (PET) Applications:

- Food Packaging
- Fibres/Clothing
- Films
- Electronics

TGA



DSC

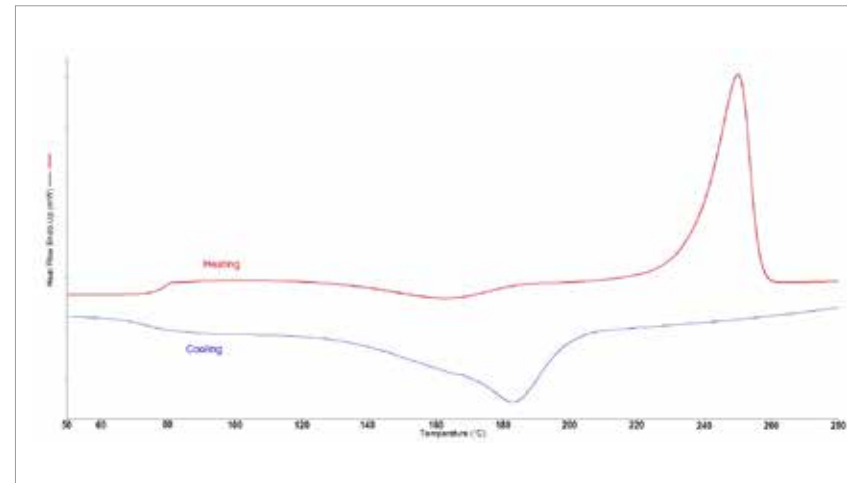


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APPLICATION - Differentiation Between PET and PET-G Using DSC

Introduction

Poly(ethylene terephthalate glycol modified) or PET-G is a derivate of PET which is modified to give more favorable properties for certain applications such as 3D printing. These differences in properties mean that recycling PET-G with PET can lead to an array of processing issues as it makes the polymer less brittle and gives it a higher processing temperature.

Differential Scanning Calorimetry can be implemented to easily differentiate between the two polymers and therefore avoid costly processing errors.

Experimental

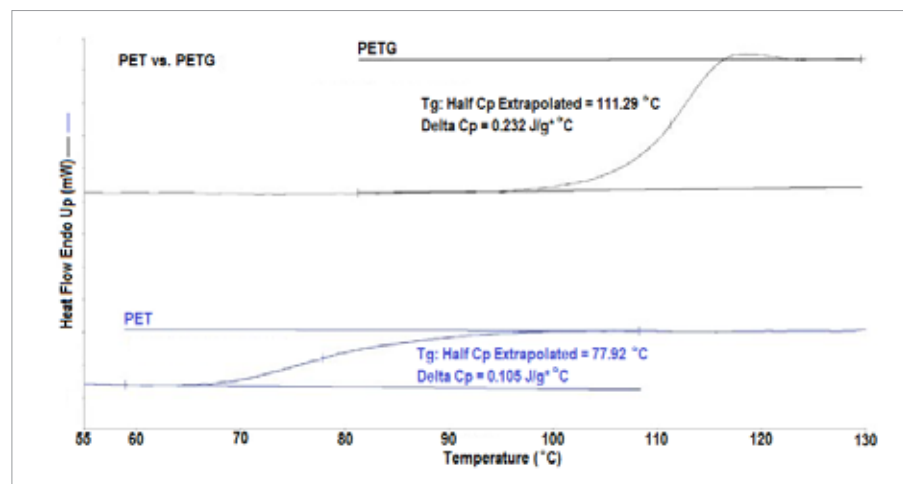
The PerkinElmer DSC 4000 was used to measure samples of PET and PET-G under a nitrogen purge (20 mL min⁻¹) using the following program:

- Heat from 25 °C to 300 °C at 10 °C min⁻¹
- Hold for 2 min at 300 °C
- Cool from 300 °C to 25 °C at 10 °C min⁻¹
- Hold for 2 min at 25 °C
- Heat from 25 °C to 300 °C at 10 °C min⁻¹

Using a heat/cool/heat program such as the one above allows for any previous thermal history to essentially be erased thus allowing for more accurate comparisons between results.

Results and Discussion

The heat flow curves for PET and PET-G are shown below.



In this experiment the glass transition temperature of PET-G is found to be 111 °C whereas the glass transition temperature of PET is found to be 78 °C.

Summary

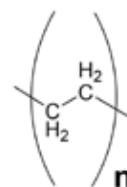
DSC can easily be used to differentiate between two similar polymers by comparison of thermal events such as glass transitions, recrystallizations, and melts.

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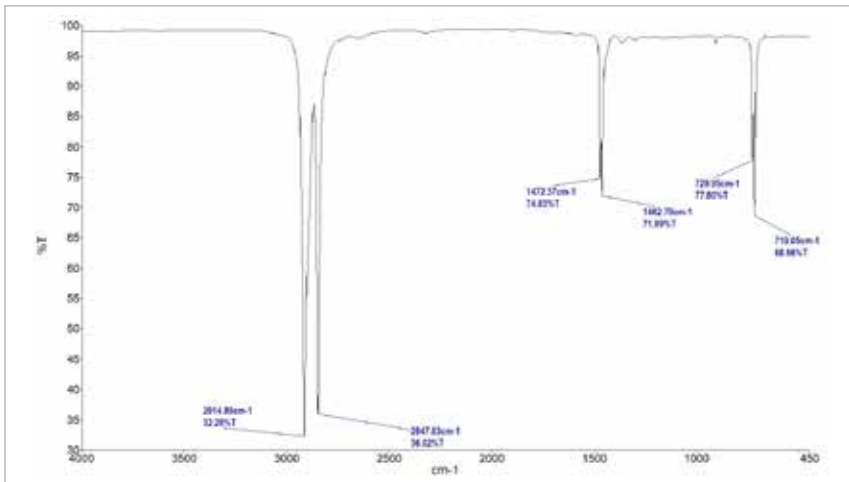
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CODE 2 High Density Polyethylene (HDPE)

T_m : 130 - 140°C T_g : -125°C



FTIR



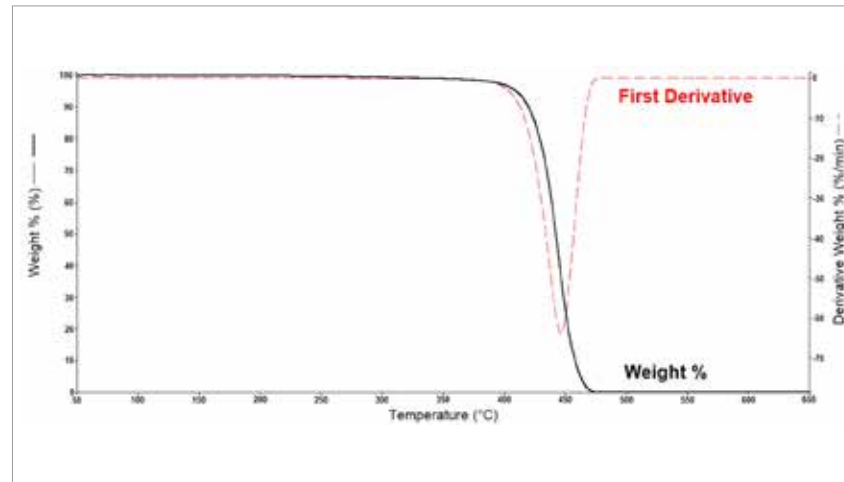
Important FTIR Peak Assignments

Peak Position (cm⁻¹)	Assignment
2915	C-H Stretch
2847	C-H Stretch
1472	CH₂ Bend
1463	CH₂ Bend
730	CH₂ Rock
719	CH₂ Rock

High Density Polyethylene (HDPE) Applications:

- Toys
- Packaging
- Pipes
- Cabling

TGA



DSC

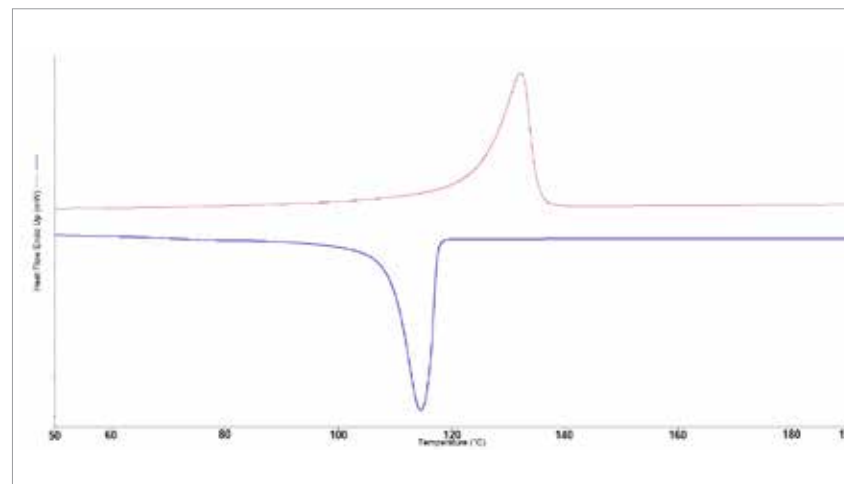


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APPLICATION - Exemplifying the Grade of PE by DSC

Introduction

As well as HDPE, there are various other grades of PE. Polyethylene falls into 2 recycling codes – code 2 (HDPE) and code 4 (LDPE). There is also linear low density polyethylene (LLDPE) and medium density polyethylene (MDPE). These materials can inadvertently be added to the recycling streams for codes 2 and 4. DSC is an effective method for separating the different types of polyethylene.

Experimental

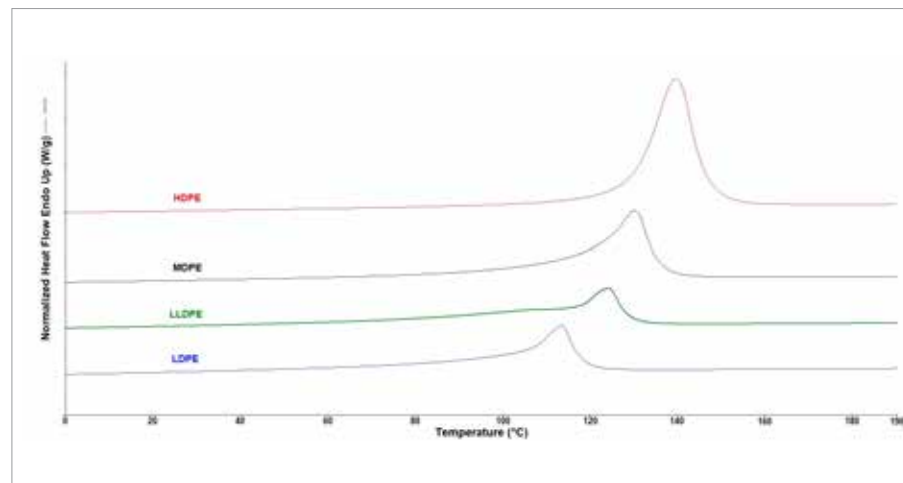
The PerkinElmer DSC 4000 was used to measure samples of polyethylene under a nitrogen purge (20 mL min⁻¹) using the following program:

- Heat from 25 °C to 200 °C at 10 °C min⁻¹
- Hold for 2 min at 200 °C
- Cool from 200 °C to 25 °C at 10 °C min⁻¹
- Hold for 2 min at 25 °C
- Heat from 25 °C to 200 °C at 10 °C min⁻¹

Using a heat/cool/heat program such as the one above allows for any previous thermal history to essentially be erased thus allowing for more accurate comparisons between results.

Results and Discussion

The heat flow curves for various grades of PET are shown below.



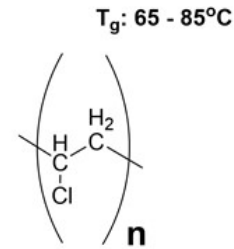
Summary

Despite being chemically similar and therefore difficult to distinguish between using techniques such as infrared spectroscopy, different grades of PE can easily be differentiated using DSC due to differences in melting temperature. This can help avoid materials such as LLDPE and MDPE being added to recycling streams for codes 2 and 4 inadvertently.

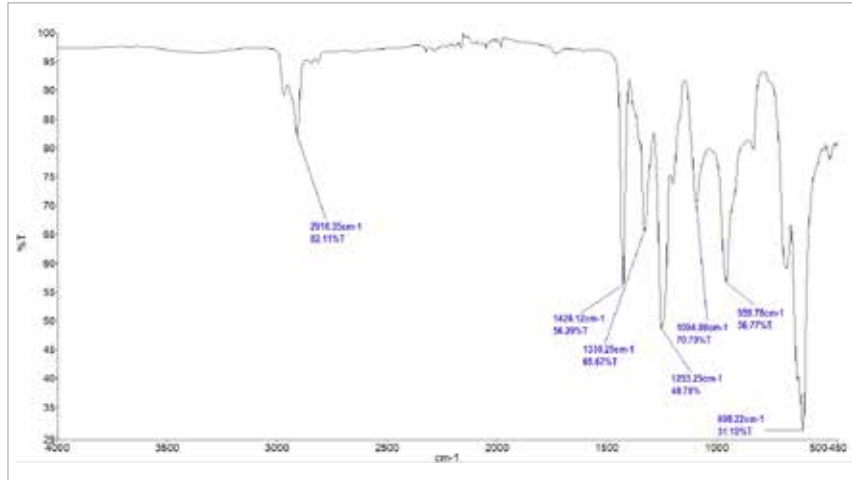
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CODE 3 Poly(Vinyl Chloride)



FTIR

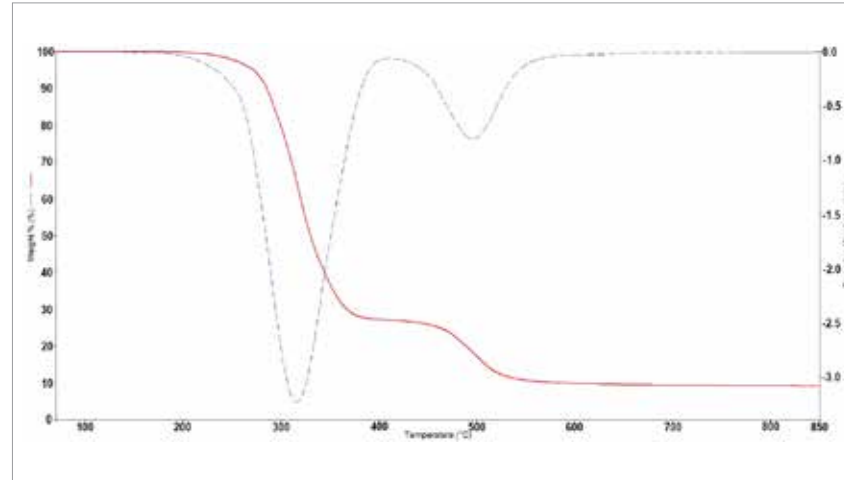


Peak Position (cm ⁻¹)	Assignment
2910	C-H Stretch
1426	CH ₂ Bend
1330	C-H Bend
1253	C-H Bend
1094	C-C Stretch
960	CH ₂ Rock
608	C-Cl Stretch

Applications of PVC:

- Pipes
- Cabling
- Construction Materials
- Medical Tubing and Bags

TGA



DSC

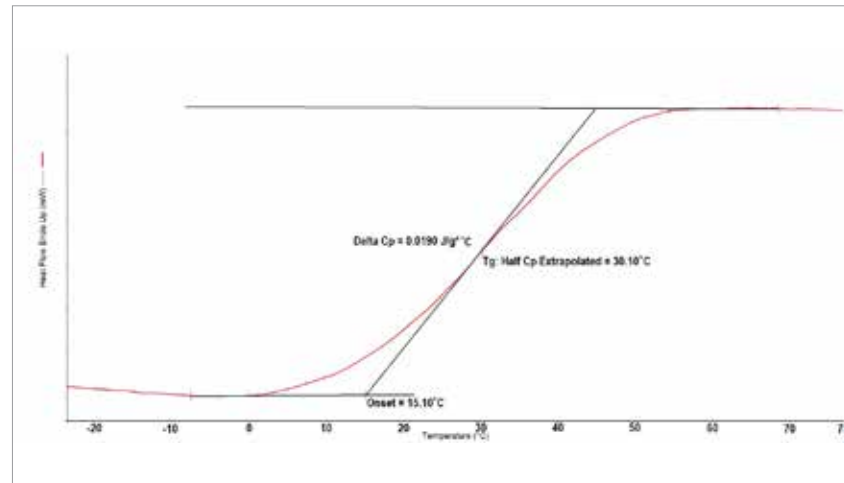


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APPLICATION - Detection of Additives in PVC by TGA

Introduction

Poly(vinyl chloride), or PVC, is frequently used in products such as packaging and piping. However, it often contains additives such as plasticisers to improve properties such as flexibility which make it more favourable as a material in packaging. High sensitivity thermogravimetric analysis can allow for detection of even very low levels of additives.

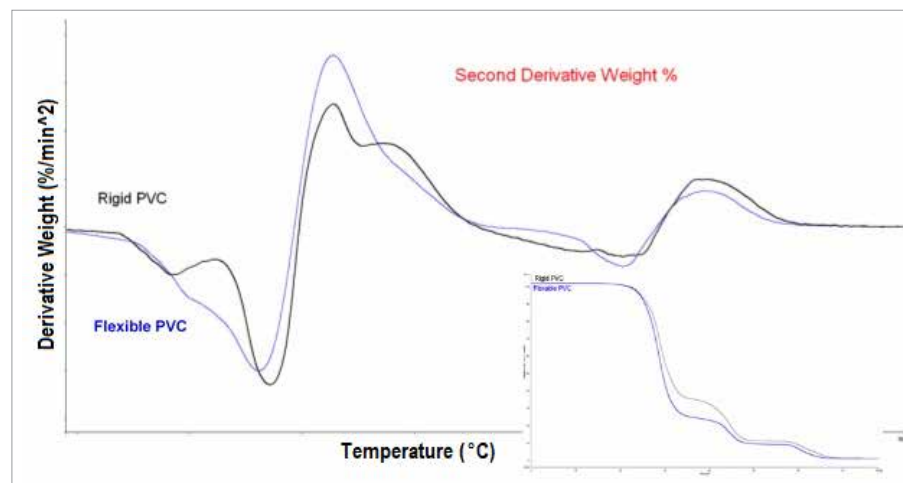
Experimental

The PerkinElmer TGA 8000 was used to measure samples of rigid flexible PVC. The measurement program used was as follows:

- Nitrogen purge at 20 mL min⁻¹
 - ◆ Heat from 30 – 600 °C at 10 °C min⁻¹
- Switch gas to air at 20 mL min⁻¹
 - ◆ Heat from 600 – 900 °C at 10 °C min⁻¹

Results and Discussion

In order to detect very subtle differences in weight loss curves, the second derivative may be used. The data below shows how this can be implemented in practice to show the difference between rigid and flexible PVC.



It can be seen from this data that the second derivative curve makes it possible to identify differences in weight loss which would not be visible from a standard TGA curve.

Summary

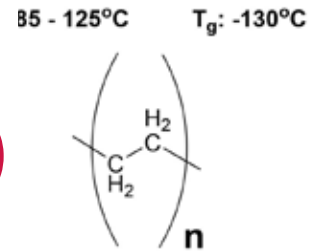
Thermogravimetric analysis, alongside data processing using Pyris™ software, can allow users to detect differences in two samples of PVC, allowing for the correct decisions to be made regarding processing.

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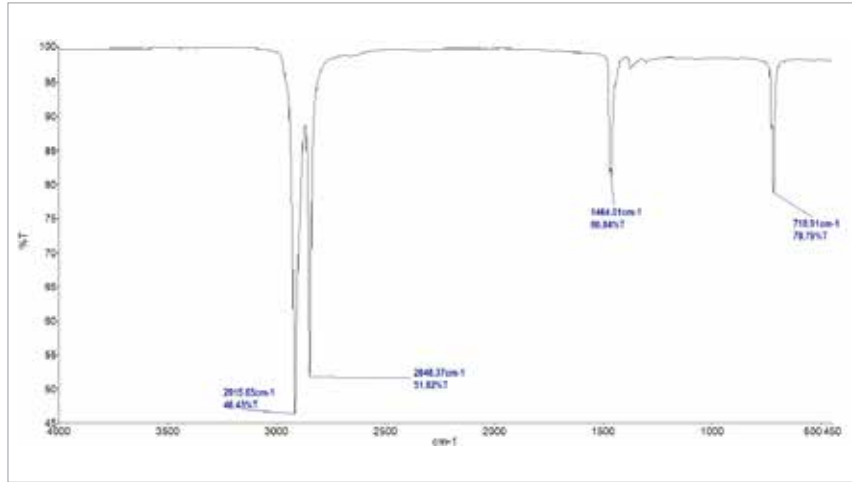
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CODE 4

Low Density Polyethylene (LDPE)



FTIR



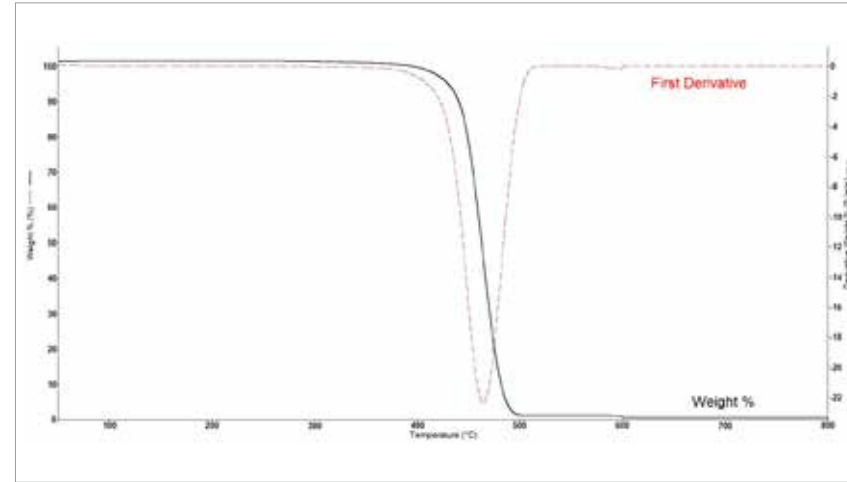
Important FTIR Peak Assignments

Peak Position (cm ⁻¹)	Assignment
2915	C-H Stretch
2848	C-H Stretch
1463	CH ₂ Bend
719	CH ₂ Rock

Low Density Polyethylene (LDPE) Applications:

- Containers
- Food Packaging
- Toys
- Cabling

TGA



DSC

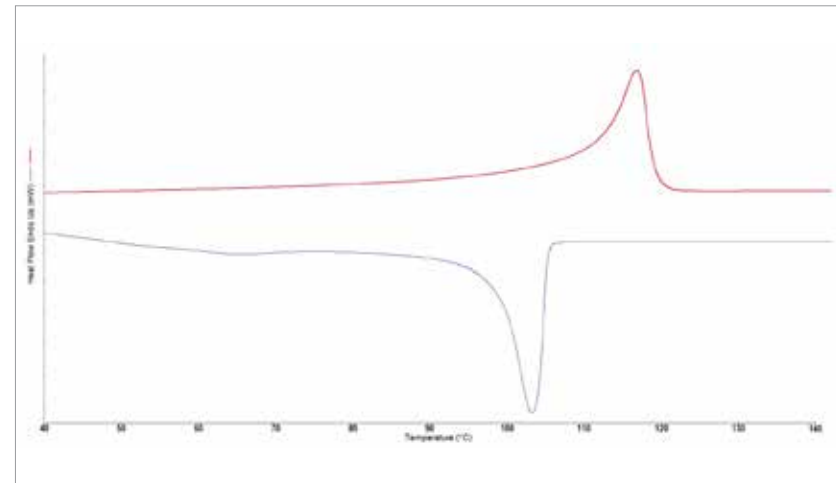
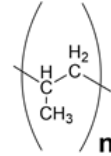


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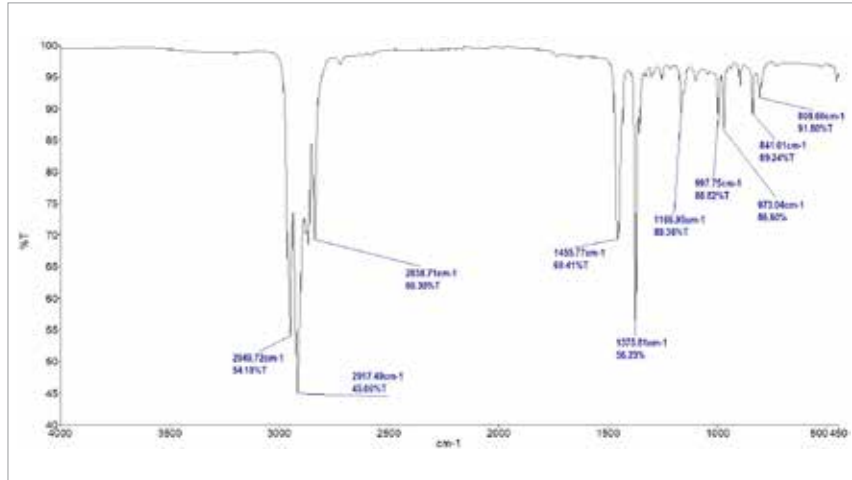
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CODE 5 Polypropylene

T_m : 165 - 175°C T_g : -20 - -5°C



FTIR



Important FTIR Peak Assignments

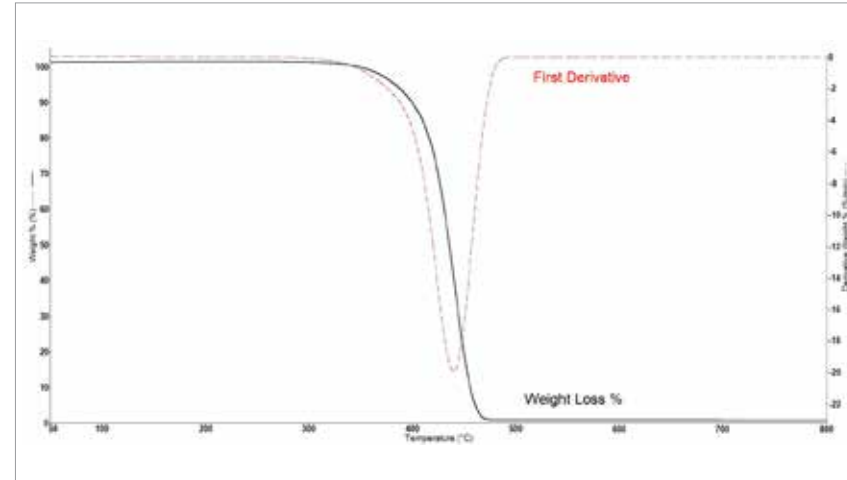
Peak Position (cm⁻¹)	Assignment
2950	C-H Stretch
2917	C-H Stretch
2838	C-H Stretch
1456	CH₂ Bend
1376	CH₃ Bend
1167	C-H Bend*
998	CH₃ Rock*
973	CH₃ Rock*
841	CH₂ Rock*
809	CH₂ Rock*

* includes contributions from other vibrations

Applications of Polypropylene (PP):

- Containers
- Piping
- Fibres/Clothing
- Packaging

TGA



DSC

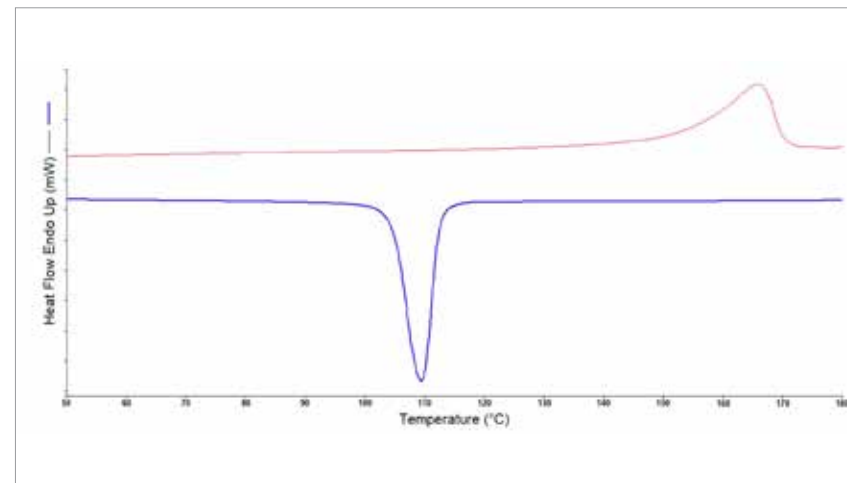


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APPLICATION - Analysis of the Effect of Filler on PP using DSC

Introduction

Polypropylene can be used for a wide variety of applications. This is, in part, allowed by the additives which may be added to alter its mechanical properties. Differential Scanning Calorimetry may be used to detect differences in glass transition temperature caused by additives.

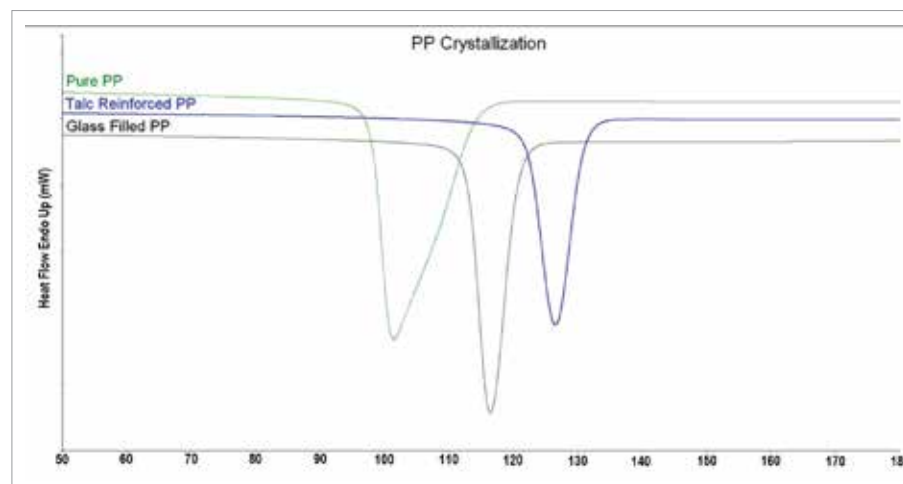
Experimental

Samples of polypropylene with different additives were analyzed using DSC under a nitrogen purge (20 mL min⁻¹) with the following method:

- Heat from 25 °C to 200 °C at 10 °C min⁻¹
- Hold for 2 min at 200 °C
- Cool from 200 °C to 25 °C at 10 °C min⁻¹
- Hold for 2 min at 25 °C
- Heat from 25 °C to 200 °C at 10 °C min⁻¹

Results and Discussion

The crystallization peaks for each variety of polypropylene are shown below.



It can be seen that the crystallization temperature for polypropylene varies depending on additives, indicating a possible difference in mechanical properties and therefore application.

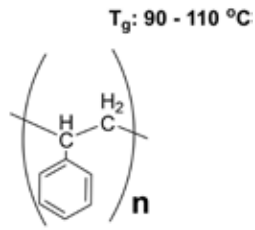
Summary

Differential Scanning Calorimetry allows for analysis of various physical properties of polymers including glass transition, crystallization, and melting. This allows analysts to detect differences between samples due to additives or previous treatment.

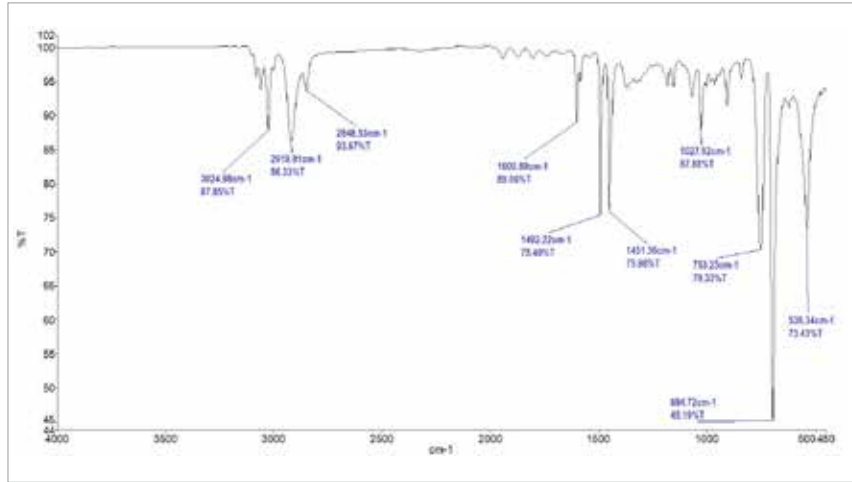
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CODE 6 Polystyrene



FTIR



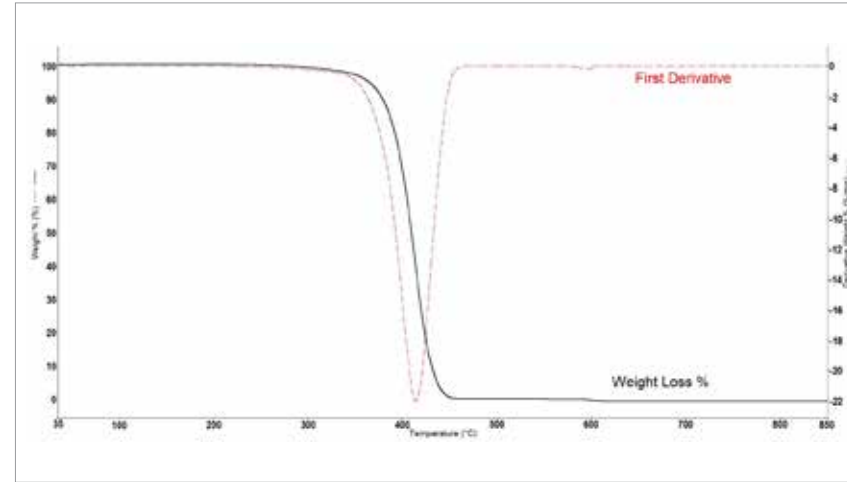
Important FTIR Peak Assignments

Peak Position (cm ⁻¹)	Assignment
3025	Aromatic C-H Stretch
2920	Aliphatic C-H Stretch
2849	Aliphatic C-H Stretch
1601	Aromatic Ring Stretch
1492	Aromatic Ring Stretch
1451	CH ₂ Bend
1028	Aromatic C-H Bend
753	CH ₂ Rock
695	Aromatic C-H Out-of-plane Bend
538	Aromatic Ring Out-of-plane Bend

Polystyrene Applications:

- Food packaging
- Electronics housing
- Insulation
- Disposable dining utensils

TGA



DSC

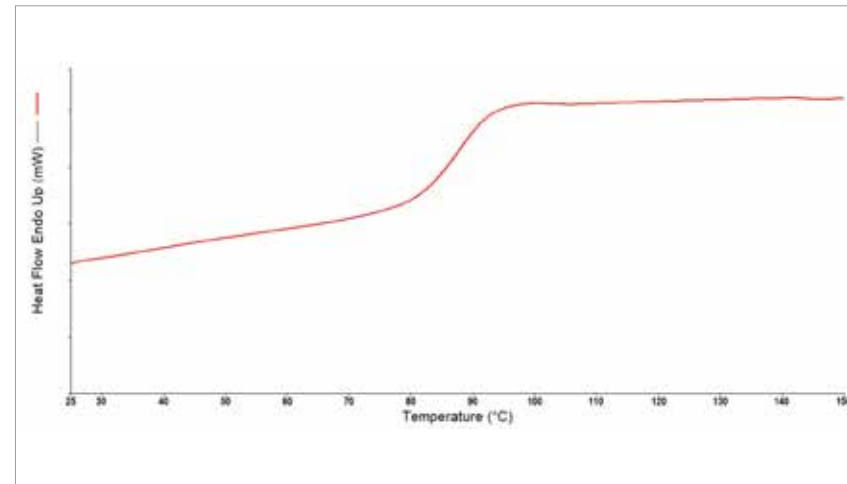


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APPLICATION - Comparing High Impact Polystyrene to General Purpose Polystyrene Using FTIR

Introduction

High Impact Polystyrene (HIPS) is a modified version of polystyrene which is more robust and can withstand harsher conditions when being processed into the final product. HIPS is produced not by the introduction of additives, but by different processing conditions. As a result, it can be difficult to differentiate between PS and HIPS.

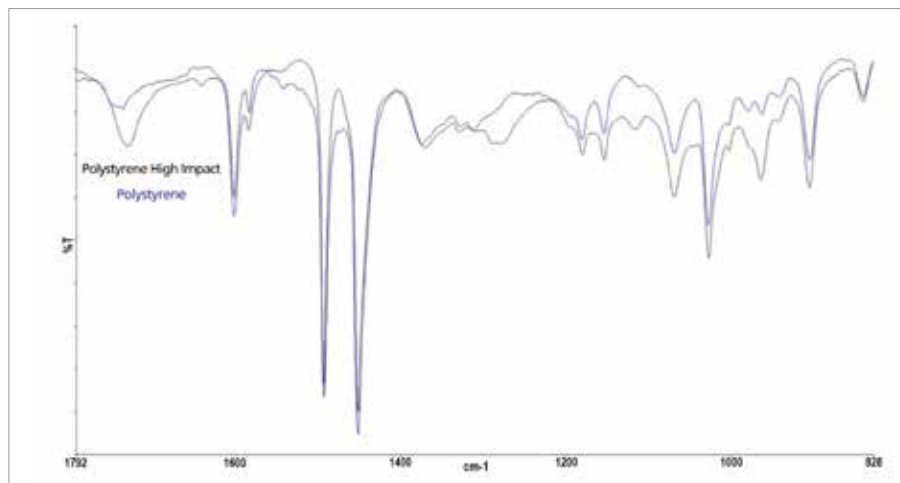
Experimental

Samples of PS and HIPS were measured using a PerkinElmer Spectrum Two™ FTIR spectrometer with an attenuated total reflectance (ATR) accessory using the experimental conditions shown in the table below:

Parameter	Value
Range	4000 – 450 cm ⁻¹
Resolution	4 cm ⁻¹
Number of Scans	4

Results and Discussion

Overlaid spectra of HIPS and PS between approximately 1800 and 800 cm⁻¹ are shown below.



There are clear differences in the fingerprint regions of the IR spectra of these materials. Key differences can be seen around 1650 and 1300 cm⁻¹. To further improve analysis, the PerkinElmer Compare algorithm could be implemented to generate a correlation coefficient between the two spectra hence giving a numeric value to how closely matched they are. This would allow for accurate determination of the identity of the sample.

Summary

Infrared spectroscopy can provide a method whereby polystyrene and high impact polystyrene can quickly and easily be differentiated.

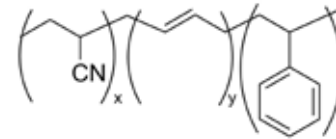
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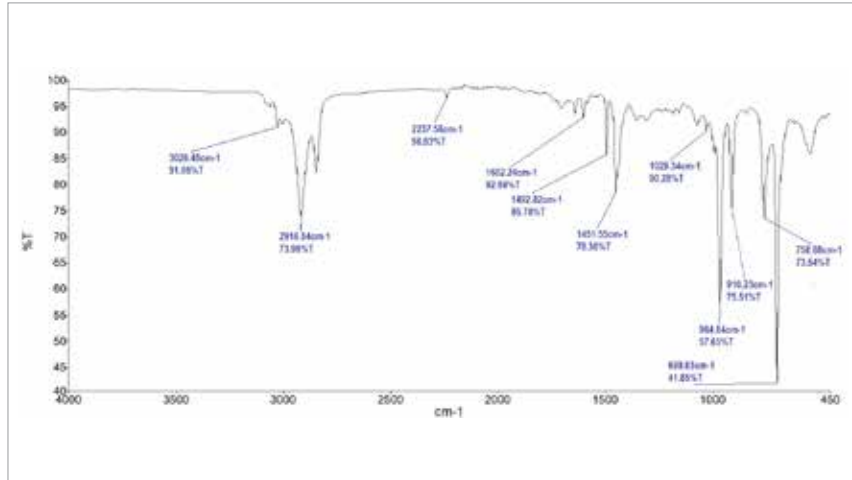
CODE 7a

Acrylonitrile Butadiene Styrene (ABS)

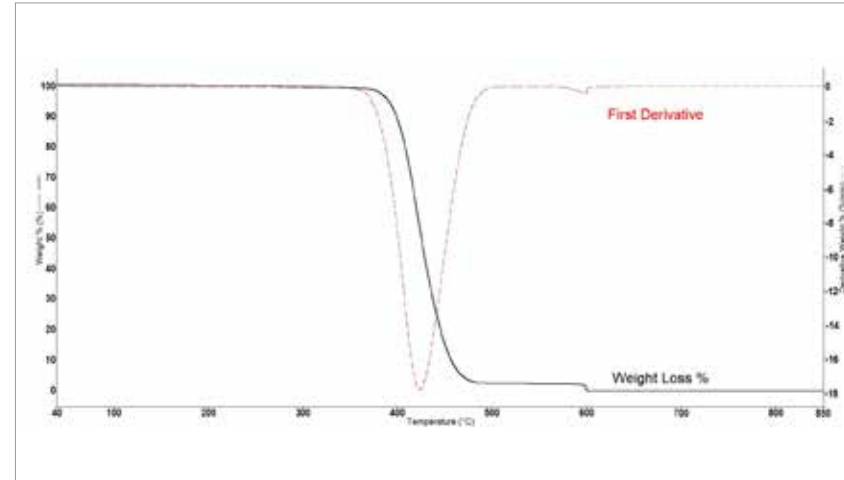
$T_g = 110 - 125\text{ }^\circ\text{C}$



FTIR



TGA



Important FTIR Peak Assignments

Peak Position (cm ⁻¹)	Assignment
3028	Aromatic C-H Stretch
2925	Aliphatic C-H Stretch
2237	C≡N Stretch
1602	Aromatic Ring Stretch
1493	Aromatic Ring Stretch
1452	CH ₂ Bend
1028	Aromatic C-H Bend
966	=C-H Bend
911	C=C Bend
759	Aromatic Ring out-of-plane Bend*
698	Aromatic C-H out-of-plane Bend*

Applications of ABS:

- Automotive
- Protective Equipment
- Toys
- Electronics Housing

* includes contributions from other vibrations

DSC

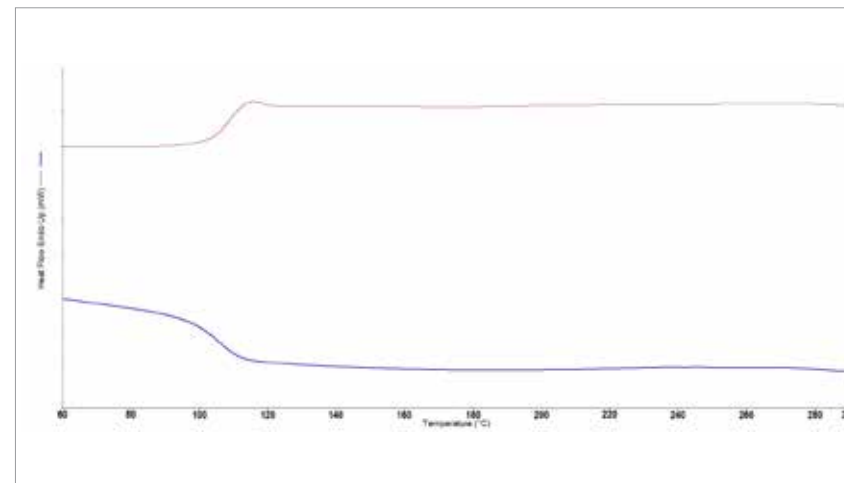


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APPLICATION - Detecting ABS-Nylon Formulations by DSC

Introduction

ABS and Nylon blends are commonplace in applications such as injection moulding. However, it is important to recognize that the addition of Nylon to a recycling process only containing ABS could have consequences for subsequent processing due to the high melting temperature of Nylon (approx. 240 °C). It is therefore important to have a method whereby ABS samples containing nylon can be identified quickly and accurately. Differential Scanning Calorimetry (DSC) provides an ideal solution to this issue.

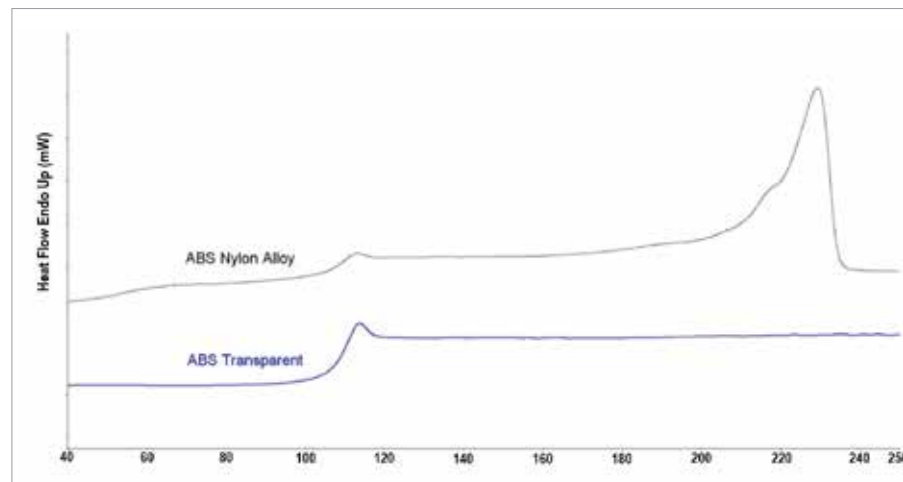
Experimental

Samples of pure ABS and an ABS/Nylon blend were analyzed using DSC under a nitrogen purge (20 mL min⁻¹) with the following method:

- Heat from 40 °C to 250 °C at 10 °C min⁻¹
- Hold for 2 min at 250 °C
- Cool from 250 °C to 40 °C at 10 °C min⁻¹
- Hold for 2 min at 40 °C
- Heat from 40 °C to 250 °C at 10 °C min⁻¹

Results and Discussion

The DSC curves for both samples are shown below:



The presence of Nylon can be easily detected by the presence of a melt peak around 240 °C.

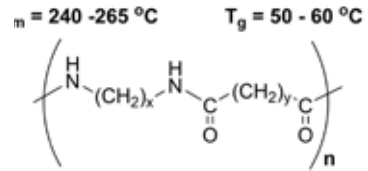
Summary

There are many different ABS formulations and therefore it is useful to have a technique which can easily differentiate between them. DSC provides a robust method by which these differences can be detected.

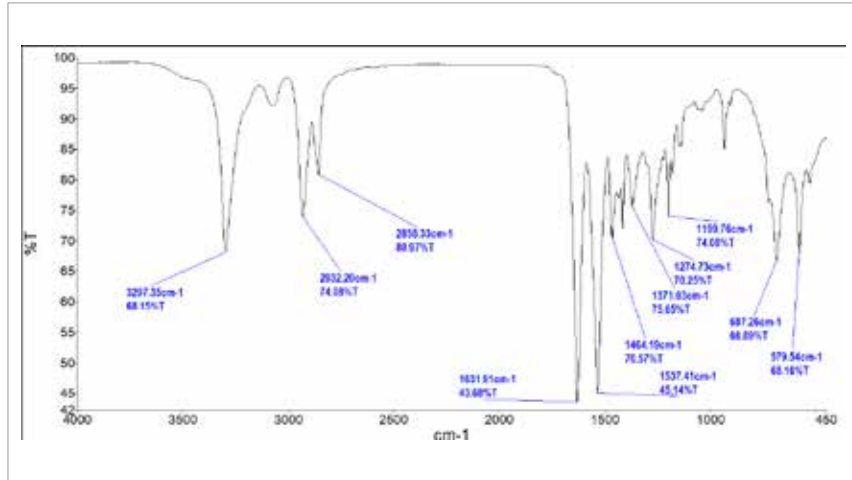
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CODE 7b Nylon



FTIR



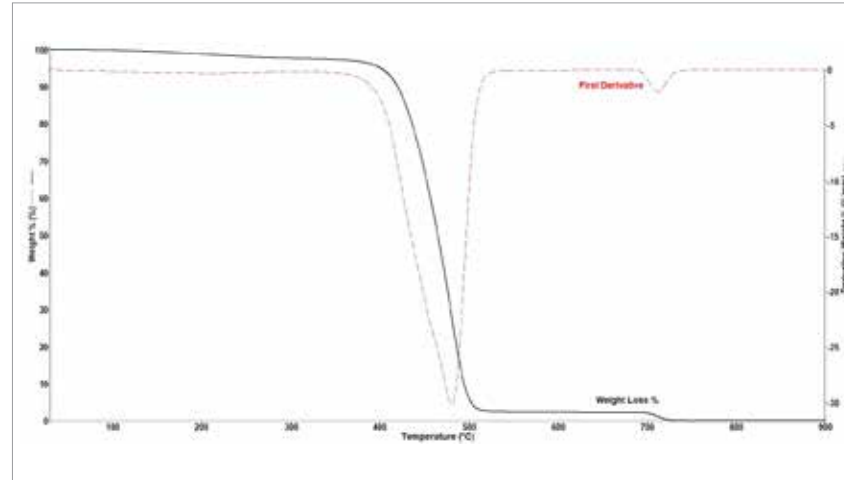
Important FTIR Peak Assignments

Peak Position (cm ⁻¹)	Assignment
3297	N-H Stretch
2932	C-H Stretch
2858	C-H Stretch
1632	C=O Stretch
1537	N-H Bend
1464	CH ₂ Bend
1371	CH ₂ Bend
1274	C-N Bend
1199	CH ₂ Bend

Applications of Nylon:

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

TGA



DSC

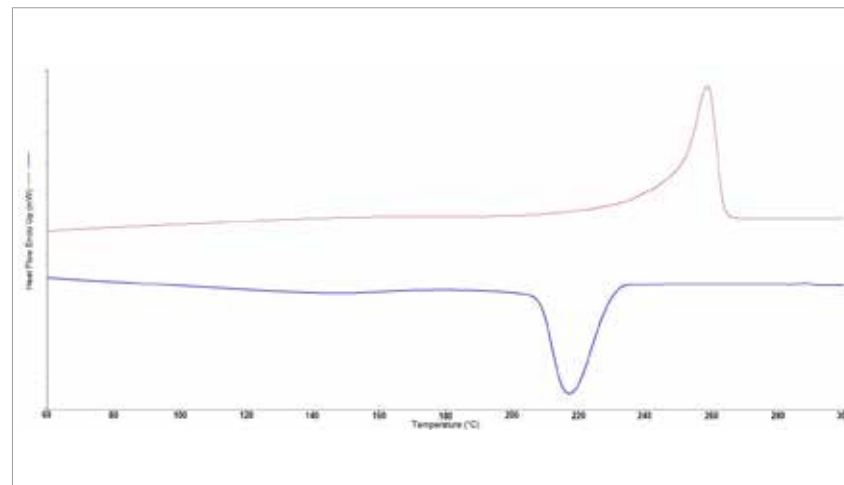


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APPLICATION - Filled Nylon Comparison using IR, DSC and TGA

Introduction

Addition of additives to thermoplastics such as nylon is common to improve stability or enhance mechanical properties. Nylon frequently contains a glass filler for this purpose. It is therefore important to have a method by which nylon materials containing different additives can be detected. This can be done by IR, DSC or TGA.

Experimental

Samples of pure and glass-filled nylon were analyzed using all three techniques. The data collection parameters for each technique are shown below.

FTIR

Parameter	Value
Range	4000 – 450 cm^{-1}
Resolution	4 cm^{-1}
Number of Scans	4

TGA

- Nitrogen purge at 20 mL min^{-1}
 - ◆ Heat from 30 – 600 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$
- Switch gas to air at 20 mL min^{-1}
 - ◆ Heat from 600 – 900 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$

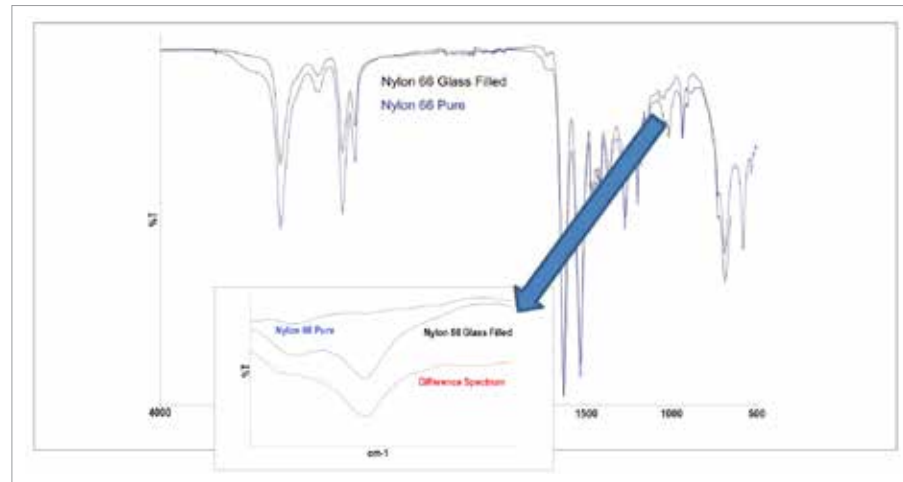
DSC

Nitrogen purge at 20 mL min^{-1}

- Heat from 40 $^{\circ}\text{C}$ to 290 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$
- Hold for 2.0 min at 290 $^{\circ}\text{C}$
- Cool from 290 $^{\circ}\text{C}$ to 40 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$
- Hold for 2.0 min at 40 $^{\circ}\text{C}$
- Heat from 40 $^{\circ}\text{C}$ to 290 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$

Results and Discussion

FTIR



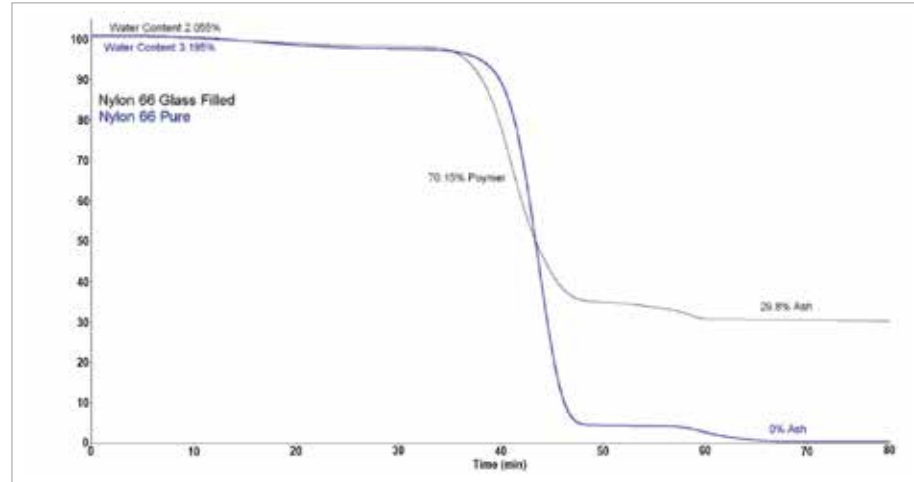
It can be seen from the figure above that there is a peak associated with the inorganic filler at approximately 1019 cm^{-1} . It is possible to isolate these peaks by using PerkinElmer's spectral subtraction algorithms.

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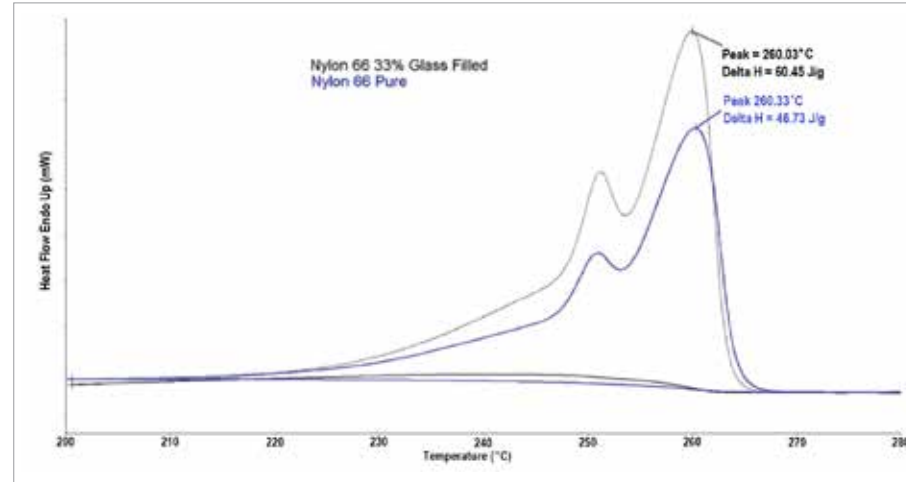
APPLICATION - Filled Nylon Comparison using IR, DSC and TGA *cont...*

TGA



The TGA curve for pure nylon shows that, after being heated in air, there is no material left which corresponds to a lack of inorganic filler. On the other hand, the glass filled nylon TGA curve shows an ash content of 29.8% due to the glass filler which will not decompose on heating under air.

DSC



Using DSC we can demonstrate that the presence of inorganic glass filler increases the ΔH of melting for nylon. This can be extended further into a calibration curve by plotting concentration against ΔH allowing for prediction of quantities of filler in unknown samples.

Summary

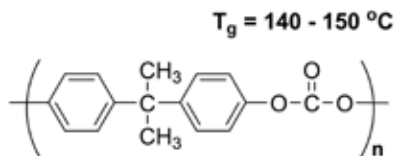
There are three materials characterization techniques, FTIR, DSC and TGA, which can be used for detection of glass filler in nylon. This allows users to avoid adding materials with different physical properties into the same recycling streams.

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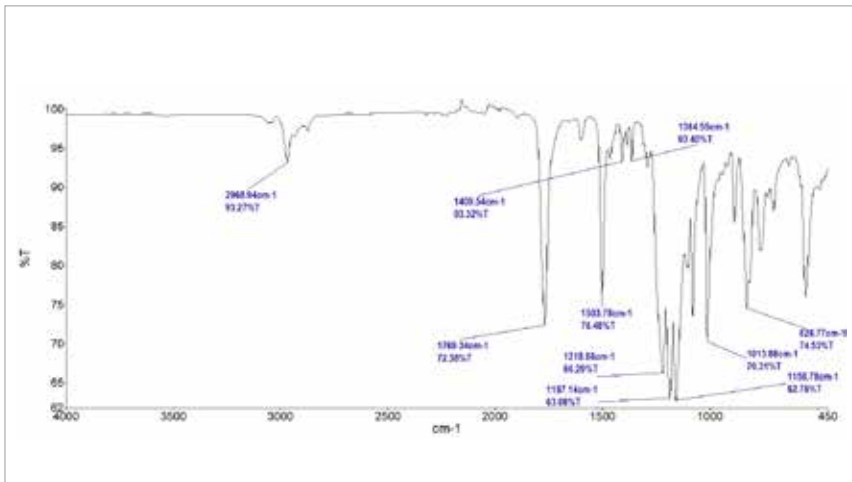
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CODE 7c

Polycarbonate



FTIR



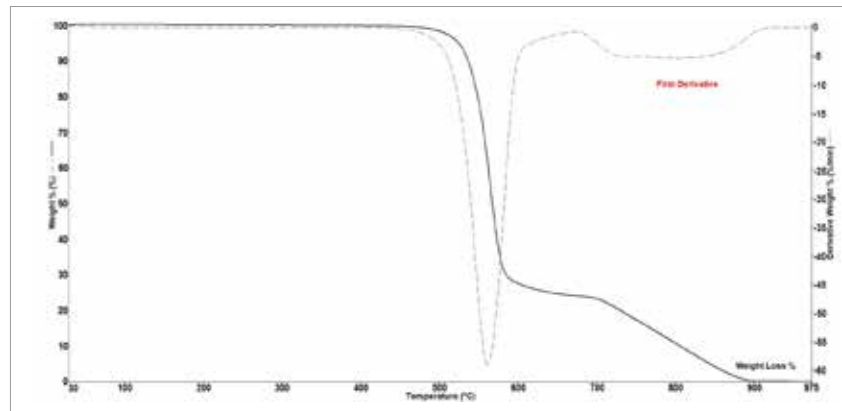
Important FTIR Peak Assignments

Peak Position (cm ⁻¹)	Assignment
2968	C-H Stretch
1769	C=O Stretch
1503	Aromatic Ring Stretch
1409	Aromatic Ring Stretch
1364	CH3 Bend
1219	C-H Bend
1187	C-O Stretch
1159	C-O Stretch
1014	Aromatic C-H in-plane bend
829	Aromatic C-H out-of-plane bend

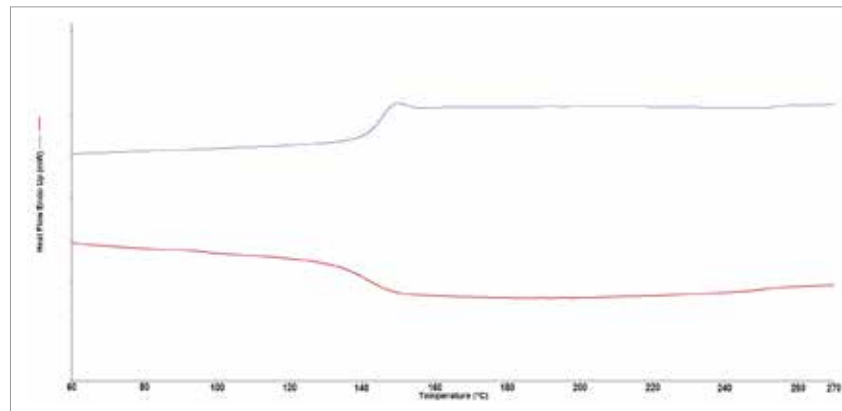
Applications of Polycarbonate:

- Safety applications
- Food and beverage containers
- Artificial glass
- Electronic housing

TGA



DSC (High Temp, 60 to 270 °C)



DSC (Low Temp, -100 to -50 °C)

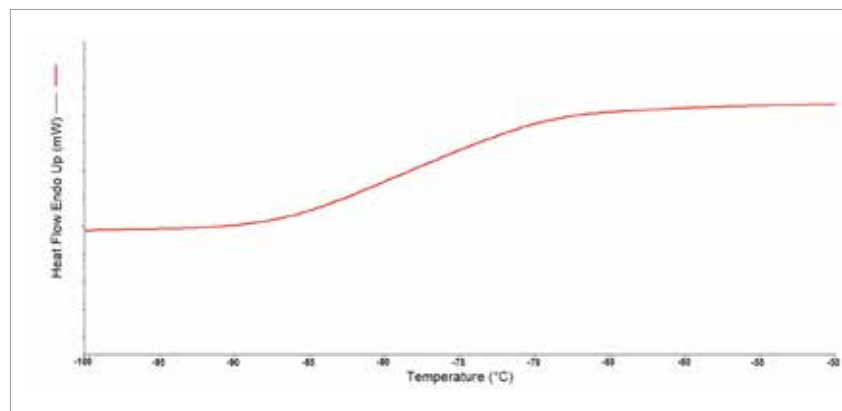


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APPLICATION - Comparing Polycarbonate to Polystyrene Using FTIR

Introduction

Due to visible similarities, polycarbonate (PC) and polystyrene (PS) are often confused with each other when in rubbish and waste. Fortunately, FTIR spectroscopy can quickly and easily identify both these materials.

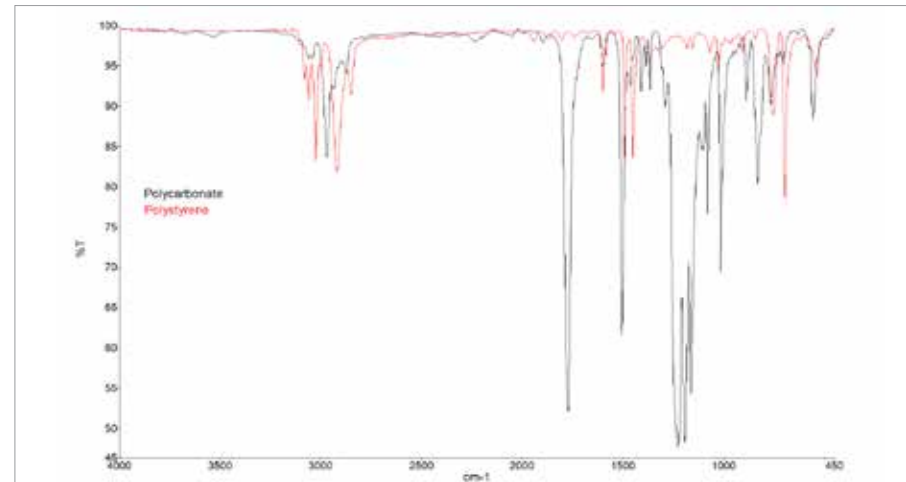
Experimental

Samples of polycarbonate and polystyrene were measured using a PerkinElmer Spectrum Two™ FTIR spectrometer and data collection parameters shown in the table below.

Parameter	Value
Range	4000 – 450 cm ⁻¹
Resolution	4 cm ⁻¹
Number of Scans	4

Results and Discussion

The overlaid spectra of PC and PS are shown below.



The most obviously distinguishing feature is the strong peak present at 1769 cm⁻¹ which corresponds to the C=O stretch in polycarbonate, a functional group which is not present in polystyrene.

Summary

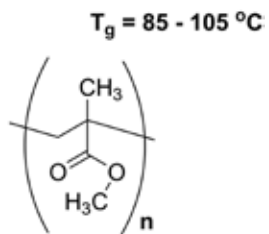
FTIR spectroscopy provides a fast, simple method for differentiating between two similar materials, allowing the user to avoid the costly error of adding the wrong material into a recycling stream.

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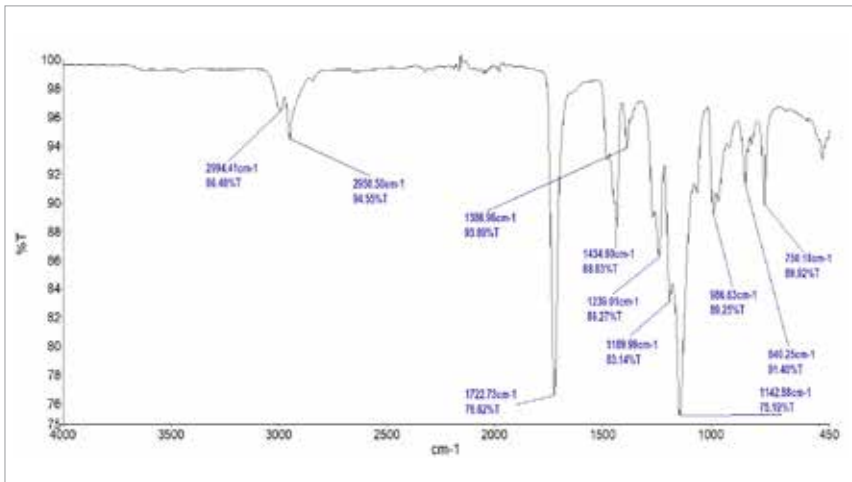
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CODE 7d

Poly(Methyl Methacrylate)



FTIR



Important FTIR Peak Assignments

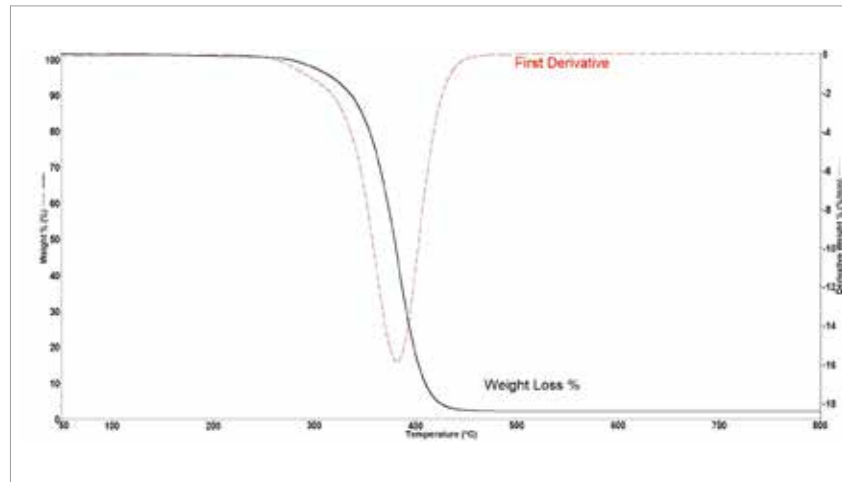
Peak Position (cm ⁻¹)	Assignment
2992	C-H Stretch
2951	C-H Stretch
1723	C=O Stretch
1435	CH ₂ Bend
1386	CH ₃ Bend
1239	C-O Stretch
1189	CH ₃ Rock
1143	C-O Stretch
987	CH ₃ Rock
965	C-H Bend
841	CH ₂ Rock*
751	CH ₂ Rock*

* includes contributions from other vibrations

Applications of PMMA:

- Artificial Glass
- Electronics
- Automotive
- Construction

TGA



DSC

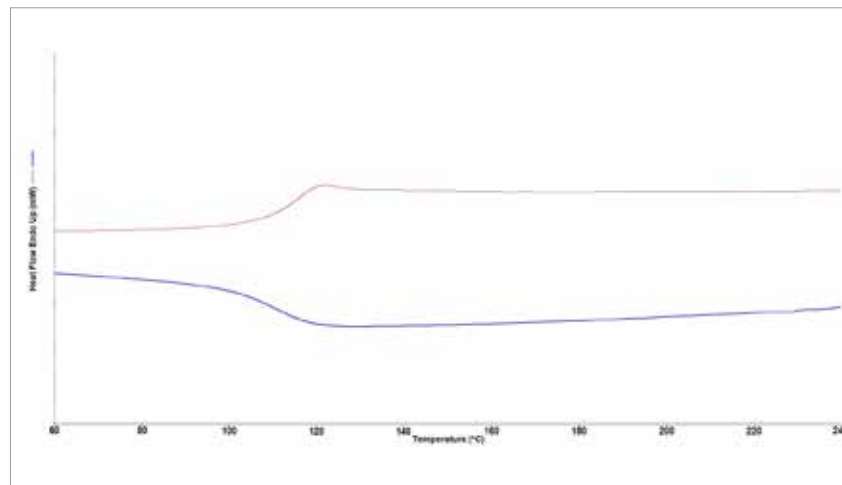


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APPLICATION - Comparing PMMA to Modified PMMA using FTIR Spectroscopy and TGA

Introduction

Poly(methyl methacrylate) is usually fairly brittle without any modification. It is therefore common practice to alter this material to give it a higher impact and scratch resistance. FTIR spectroscopy and TGA can be used to differentiate between modified and non-modified PMMA.

Experimental

FTIR

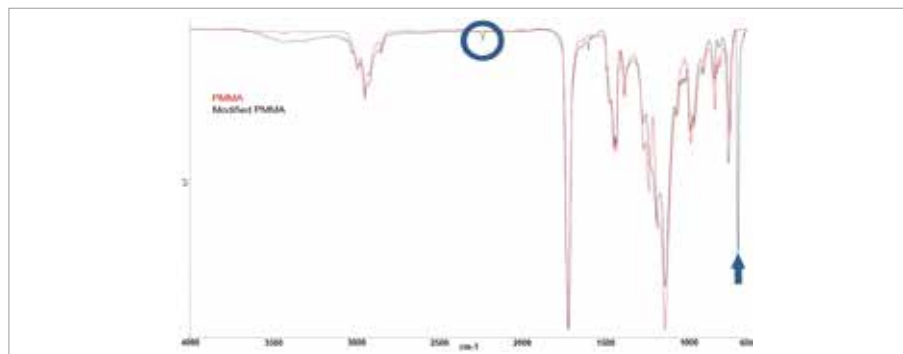
Parameter	Value
Range	4000 – 450 cm^{-1}
Resolution	4 cm^{-1}
Number of Scans	4

TGA

- Nitrogen purge at 20 mL min^{-1}
 - ◆ Heat from 30 – 600 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$
- Switch gas to air at 20 mL min^{-1}
 - ◆ Heat from 600 – 900 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$

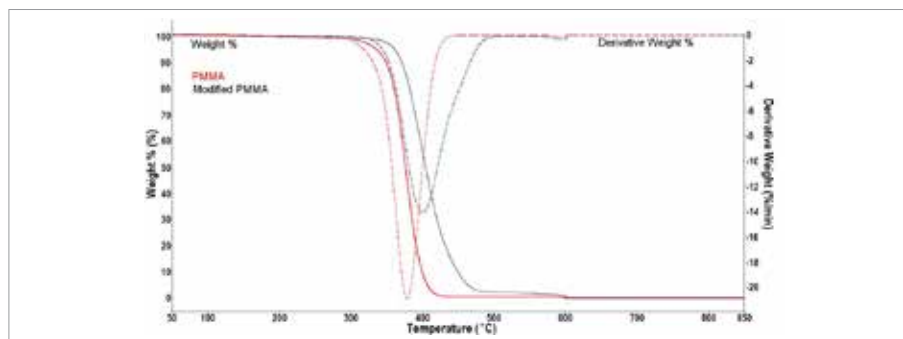
Results and Discussion

The overlaid IR spectra of PMMA and modified PMMA are shown below.



The spectra above show peaks in modified PMMA at around 2200 cm^{-1} and 700 cm^{-1} . This allows for easy differentiation between the two materials.

The TGA curves for PMMA and modified PMMA are shown below.



It can be seen from these results that the maximum weight loss in modified PMMA occurs at a slightly higher temperature than that of non-modified PMMA.

Summary

Both TGA and FTIR provide users with fast and reliable methods for differentiating between modified and non-modified PMMA.

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User Guide

Introduction

The information and data included in this compendium is intended to guide users in the analysis of polymer materials. All data was collected using clean, pure resins and PerkinElmer instrumentation. It is important to remember that real-world samples may have been altered to improve various properties and thus may not present data exactly as seen in this document. The scope of this compendium is not intended to cover every possible additive found in the different classes of polymers, but rather to give the user the tools to carry out meaningful analysis which will be important for structural elucidation and identification of recycled materials.

In this section of the guide, users will find an overview of the steps required to reproduce the data in this compendium, alongside some useful information regarding sample preparation and data collection for each technique.

Fourier-Transform Infrared Spectroscopy

Preparing the Instrument

To begin analysis by FTIR, several items need to be prepared:

- Verify that the instrument has passed all "Ready Checks", specifically contamination checks (if a check regarding the instrument hardware fails, contact technical support).
- If the contamination check fails, clean the diamond ATR crystal with the appropriate solvent and disposable wipe (isopropanol is the most common for this purpose).
- Allow some time for any residual solvent to evaporate then re-run the contamination check.

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

Preparing for Analysis

Included in the PerkinElmer polymer recycling pack is an FTIR library file. This library should be added to the list of searchable libraries in Spectrum 10. Instructions on how to do this may be found in help section of the software.

In the same folder as the library file is a '.SET' file. This can be imported onto the instrument configuration page. See Figures 1 and 2 for visual guidance on import and activation.

Once the instrument is configured, a background scan is required. Verify that the ATR shoe on the pressure arm is not near the ATR crystal before pressing the 'Background' button (Figure 3).

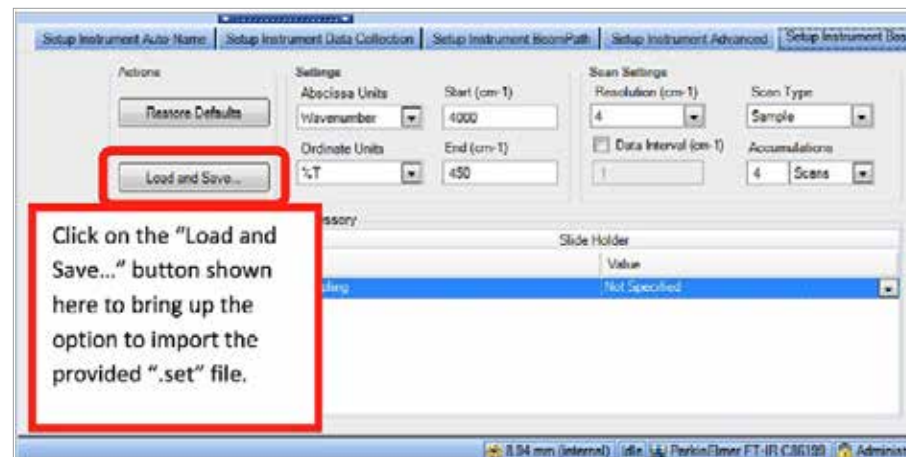


Figure 1. "Setup Instrument Basic" tab. This is the area where the most important method parameters are configured.

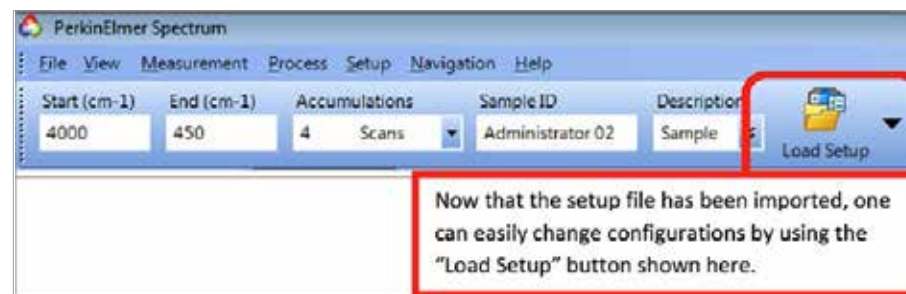


Figure 2. Instrument Toolbar. Highlighted is the button which allows methods to be imported.

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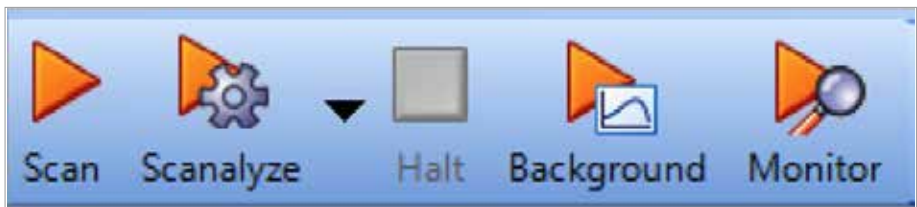


Figure 3. Identifying the "Scan", "Background" and "Scanalyze" buttons on the instrument control panel.

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 such that the flat faces are parallel to each other (Figure 4). Avoid grinding or 'dry' sanding the sample as the friction/heat may 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 pass contact at this stage.



Figure 4. Example of a polymer sample cut to have a flat top and bottom, for measurement by both DSC and FTIR.

Measurement

Press the "Scanalyze" button then highlight "Scan and Search". This action will put the instrument into what is called "Preview Mode". Once the real-time display becomes active, being applying pressure to the sample by twisting the dial on the ATR pressure arm clockwise. 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 to begin data acquisition and the library search. If the largest peak does not exceed 90%T, then remove the force from the sample and reposition, recut etc., until the sample does meet this requirement. Force above 100 units should not be applied as this could alter the sample crystal structure.

Data Analysis

When using the 'Scanalyze' function, the search process begins while the data is collected. By the time the experiment is complete, the best match will be displayed. In an ideal situation, the best hit would have a score of 1.000, however, this will never occur. Typically, a score greater than 0.900 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.

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.

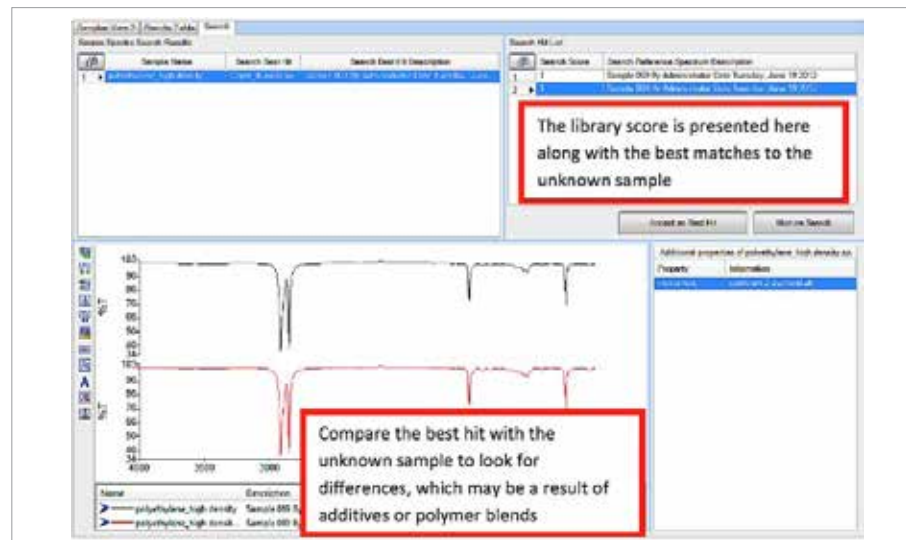


Figure 5. Example output from the 'Scanalyze' analysis.

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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 universal attenuated total reflectance accessory (UATR) with diamond crystal, KBr sample compartment windows and a DTGS detector. Any deviation from this configuration could result in a deviation from the presented data.
- Peak labels and assignments 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.

Differential Scanning Calorimetry

To begin the analysis of polymers by DSC, the instrument must be correctly configured, and equilibrium must be reached. An inert gas (such as N₂ or He) should be used to purge the system and furnace with a delivery pressure of 30-40 psi. Correct 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 has been established. Set the temperature to 50°C using the 'Go To' temperature button (Figure 6). Activate the cooling device attached to the instrument.

Notes for Cooling Devices

If water-cooling is utilized, verify the coolant is at the correct 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 the intracooler is activated, the transfer line cannot be moved. In either case, the cooling device should be powered on for a minimum of one hour prior to making any measurements.

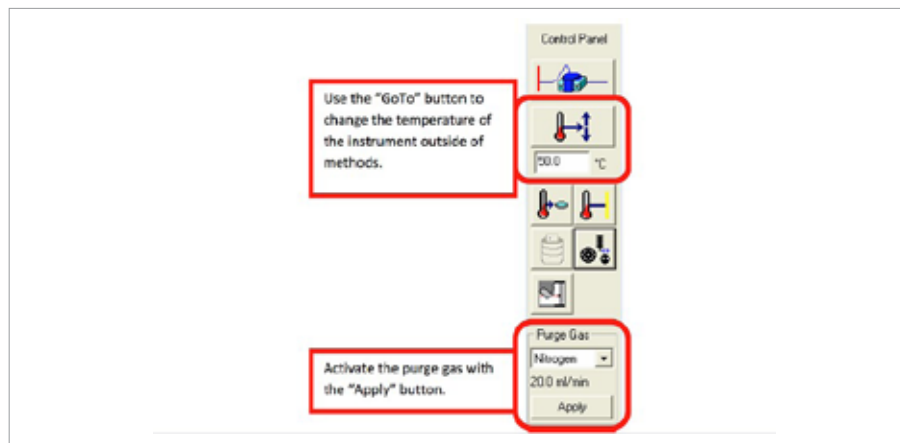


Figure 6. Instrument control bar in Pyris software. Highlighted is the "Go To" temperature and "Apply" purge gas sections.

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, tin, lead and/or zinc. This policy should be outlined in the laboratory SOP.

Method Editor Configuration

Each of the polymers have a specific DSC method associated with them. For the best results, an intracooler should be used. Pick the method which best suits the suspected material in its respective folder. Feel free to alter the temperature ranges so long as the maximum temperature stays below the degradation temperature of the material being measured. Do not alter the number of steps or heating rates used between samples. Data collected using different heating rates can not be compared. If the maximum safe temperature is unknown, a TGA experiment should be performed to check the degradation temperature of the material.

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

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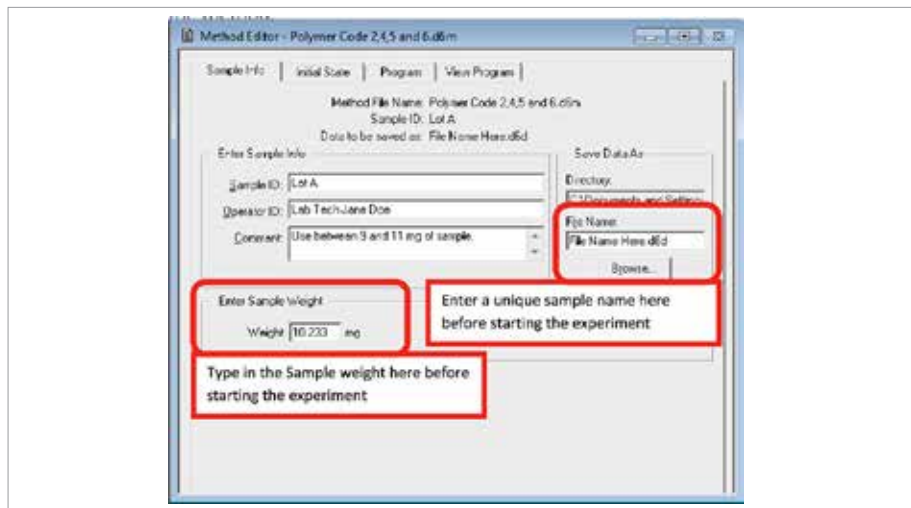


Figure 7. Example of editing the sample information in the DSC method editor.

Sample Preparation

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

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

Preparing for Measurement

Using the 'Go To' temperature button, lower the instrument temperature to a safe level (< 30 °C). Load the sample into the instrument. If an autosampler is equipped, then allow the instrument to place the sample, otherwise use forceps. Verify that the furnace covers have been returned to their correct location.

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 any other important information. Figure 7 provides a good example of how the fields in this section should be filled in. The population of these text fields should be outlined by the laboratory SOP. To start the run, press the "Start/Stop" button (Figure 8). Typically, measurements will take in excess of 30 minutes to complete – allow enough time for the experiment to complete. If the measurement is stopped prematurely, data collected up until that point will be saved.

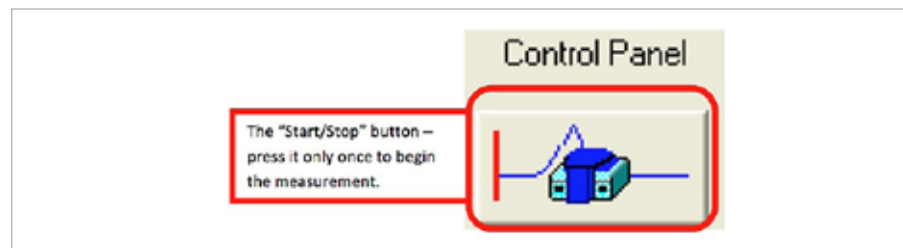


Figure 8. DSC "Start/Stop" button.

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 the pure material used for comparison must be prepared for analysis. Using the "Curves" menu, select the experiment intervals that will be analyzed. Some DSC files are provided to assist in this analysis.

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Typically, the second heat 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 the use of the “Slope” button to align them as closely as possible. Now compare the various peaks and glass transitions found in these two curves. Are they close to one another? If the answer is “no”, then look for another polymer and repeat the process.

If the polymer cannot be identified using the DSC data provided, then the sample may either be a polymer not present in this compendium or a mixture of polymers. If the former is the case, measuring an IR spectrum of the sample and searching this against a commercial library may assist in identification. If the latter is true, overlaying DSC curves of the possible constituent polymers may be the way forward.

If no melting peak is observed in the polymer (e.g. in polystyrene), the glass transition (T_g) can be utilized in this measurement. Typically this number will be less than one and displayed to three decimal places. 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.

Notes on Quantitation

A great deal of caution should be expressed at this point as the aforementioned method will not provide an exact value but rather a reasonable approximation. The addition of fillers, additives, and polymer blends can greatly influence this measurement. If more accurate values are required, then TGA will likely provide the best method for obtaining this information.

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 transitions displayed in this compendium are for reference purposes only. Note that the 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.

Thermogravimetric Analysis

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 25-40 psi (this is normally configured by the PerkinElmer service representative at the point of installation). Verify that the instrument is powered on. Activate Pyris software and verify that instrument-software connection is achieved. Set the instrument temperature to 30 °C using the “Go To” temperature button (Figure 9). Activate the cooling device attached to the instrument.

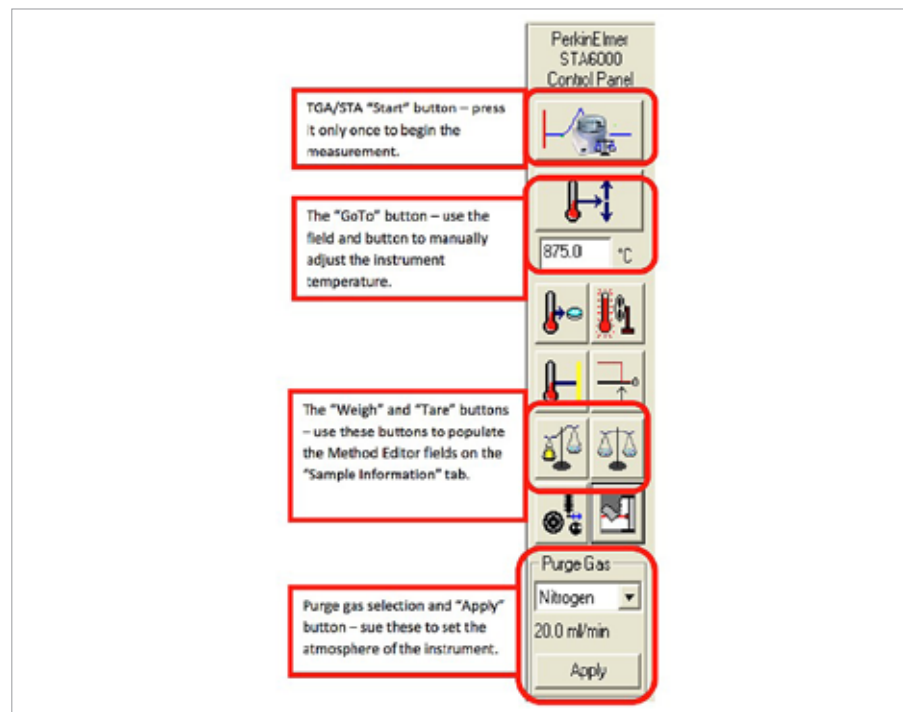


Figure 9. TGA/STA control panel.

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Notes for Cooling Devices

Water-cooling is utilized for both the STA6000 and TGA4000. Ensure 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 one hour prior to making any measurements. It is not uncommon for laboratories to wait 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.

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 a relatively easy endeavour, 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 usually the preferred option, but cost tends to push laboratories towards alumina. Alumina sample pans will work well for most polymers but 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 old 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 “Go To” 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 pan on the balance. If the instrument has an autosampler, allow it to insert/remove the crucible. Never attempt to put a sample into a pan that is still in the instrument. Place the sample into the crucible then return it to the instrument. 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 laboratory SOP (Figure 10) and click the “Start” button (Figure 9). Typically, the measurement will take longer than 45 minutes. Allow enough time for the experiment to complete. If the measurement is stopped prematurely, the data collected up until that point will be saved.

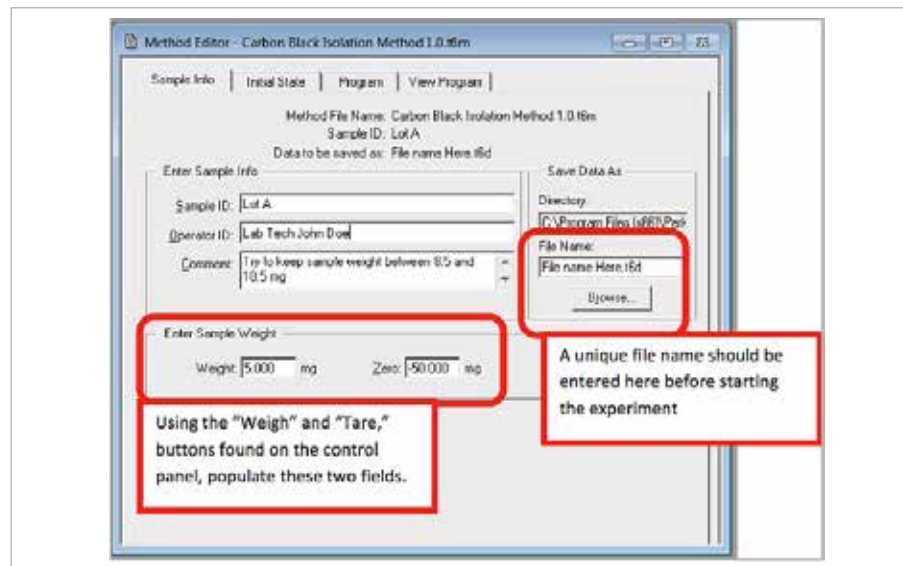


Figure 10. Example of fully-populated TGA Method Editor.

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Analyzing the Data

TGA data may be viewed with either time or temperature on the x-axis (which may be changed using the “T/t” button on the toolbar) and weight or weight % on the y-axis (which may be changed by in the “Display” menu).

In each of the seven code folders, example TGA data files are provided for comparison and 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 a material, DSC or FTIR should be utilized as additives can greatly influence degradation. If the laboratory has a reference material for comparison, add the data using the “Add” function in the “File” menu. Utilizing both the weight and the 1st derivative of the weight, the identity of the material may be verified.

If an FTIR 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 onto the ATR crystal to collect the spectrum (Figure 12). If the identity of the filler is already known, save the spectrum 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.

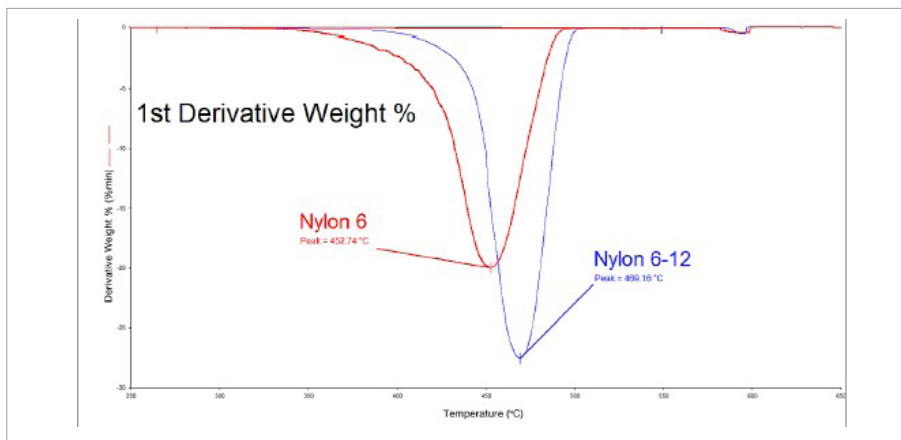


Figure 11. Example of usage of the 1st derivative plots to determine the identity of a polymer. In this case, there is a clear difference between the two forms of nylon.

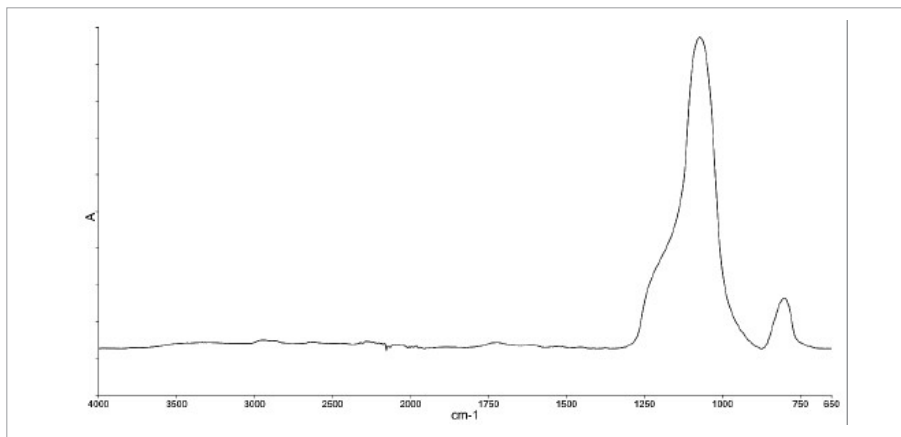


Figure 12. FTIR analysis of the ash remaining in a TGA pan.

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.

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