



# Characterization of the Quality of Recycled Polymers Using DSC

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## Introduction

The use of polymer recyclates has been increasing over the past decade due to economic, political, environmental and social factors. Recycling refers to the process by which a polymer is reused and reprocessed and can take the form of post-consumer reclaim or the much larger volume post-industrial recycling. Post-consumer recycling is associated with the collection and reuse of discarded plastic items from home use, including plastic food and beverage containers. The post-industrial recycling process refers to polymer 'scrap' from a polymer processing facility, which is sent to a recycling facility where the material is sorted and processed by grinding. The material is then sold as *regrind* to a polymer production plant for incorporation into the virgin polymer feedstock.

For both post-consumer and post-industrial recycling, it is important to identify the type of polymer that is received by the recycling process center for regrind purposes. With the post-consumer market, the containers are clearly marked with the appropriate identification (e.g., PETE, HDPE, PVC, etc.) which makes their recycling more straightforward. For post-industrial recyclates, the situation can become more complicated as the polymer scrap is not necessarily identified. In some cases, a polymer which is specified as being one material may actually be something else altogether or may have an additional, and unspecified, contaminant or other polymer present.

An example where a major problem could potentially occur would be in the use of post-industrial polymer scrap or

Pyris 6 DSC



regrind which inadvertently or accidentally contains polyvinyl chloride (PVC) contaminant. Even a small amount of PVC will give off the highly corrosive hydrogen chloride gas (HCl) during production, which can potentially wreak havoc on a facility which does not normally process PVC.

In dealing with both post-consumer and post-industrial recyclates or regrinds, there is a real need to characterize the scrap and/or regrind in terms of:

- the identification of the main polymer component
- the presence any other secondary contaminants or components
- lot-to-lot variability

Knowing the compositional characteristics of recyclates and regrinds is critical as they can significantly affect both processibility and end product quality. One of the best and most powerful analytical techniques for the analysis of polymeric materials, including recyclates, is differential scanning calorimetry.

## Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) provides a means of characterization issues associated with recyclates and regrinds. The Pyris 6 DSC from PerkinElmer provides an ideal means of characterizing polymeric recyclates and regrinds. The instrument offers the following important features:

Figure 1. DSC results on ABS recyclete resin containing PVC contaminant.

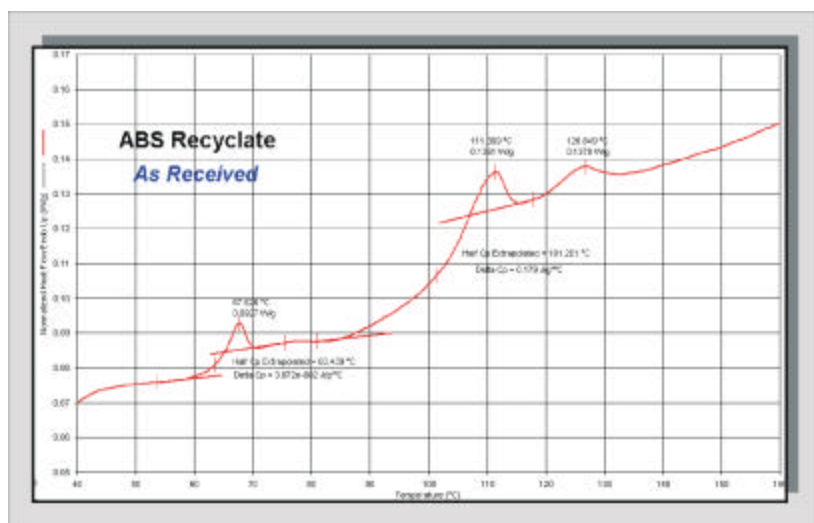
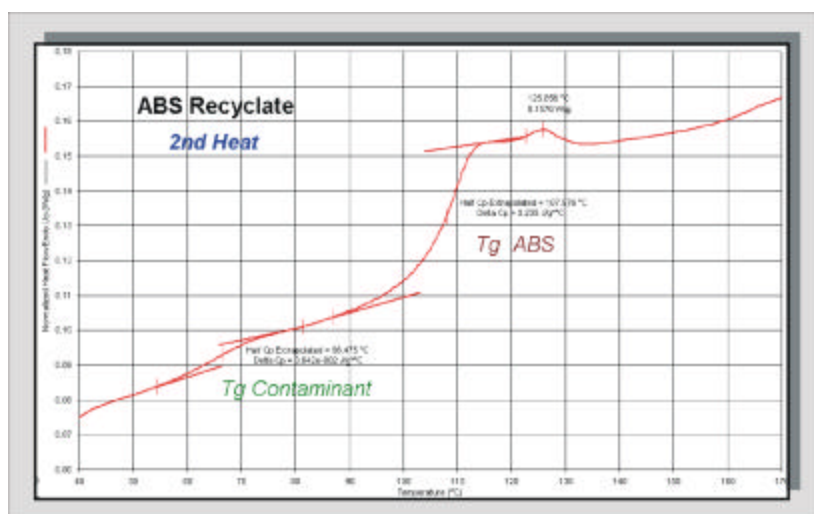


Figure 2. DSC results on ABS recyclete during second heating.



- High performance in terms of sensitivity and resolution
- Low cost
- Outstanding, reliable autosampler for automated loading of up to 45 samples for unattended operation
- Highly stable performance for consistent and reproducible results
- Pyris software (Windows NT-based) for ease of use
- Automated tolerance testing
- Automated built-in gas controller
- Pyris Player for single button, start-to-finish operation

## Results on ABS Recyclate

ABS is widely used for many applications (e.g., PC housings) because of its good performance properties, including impact resistance. For recycling purposes, it is important to know the characteristics of ABS either in post-industrial scrap form or as regrind. The addition of extra component(s) could make the processing of the regrind ABS material difficult or result in less-than-desirable end use properties with the final product.

Displayed in Figure 1 are the DSC results generated on sample of ABS, which contains a PVC contaminant. It is critical to screen many recyclates or regrind polymers for the presence of PVC because of the dangers of off-gassing of hydrogen chloride from the PVC contaminant during processing. The figure shows the DSC heat flow as a function of sample temperature for the ABS resin containing the PVC contaminant at a heating rate of 10 C/min.

These results show that the as-received ABS yields several transitions. The two thermal events at 63 and 101 C are most likely glass transitions with accompanying stress relief and/or enthalpic relaxation effects. ABS should exhibit only one Tg in this temperature interval at about 105 C, which is associated with the softening of the SAN copolymer phase in ABS. It would seem that there is indeed a contaminant in the ABS recyclete, which is manifested as an additional Tg near 63 C.

When the sample is cooled back to ambient and then reheated, the stress relief and enthalpic relaxation effects are eliminated, which 'cleans up' the transitions and improves the interpretation of the data. Displayed in Figure 2 are the DSC results obtained by heating the ABS recyclete sample a second time.



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Figure 3. DSC results on virgin ABS resin.

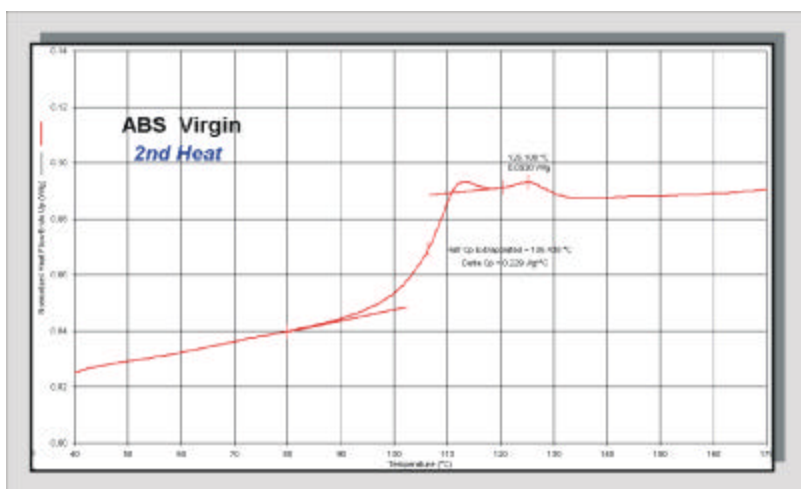
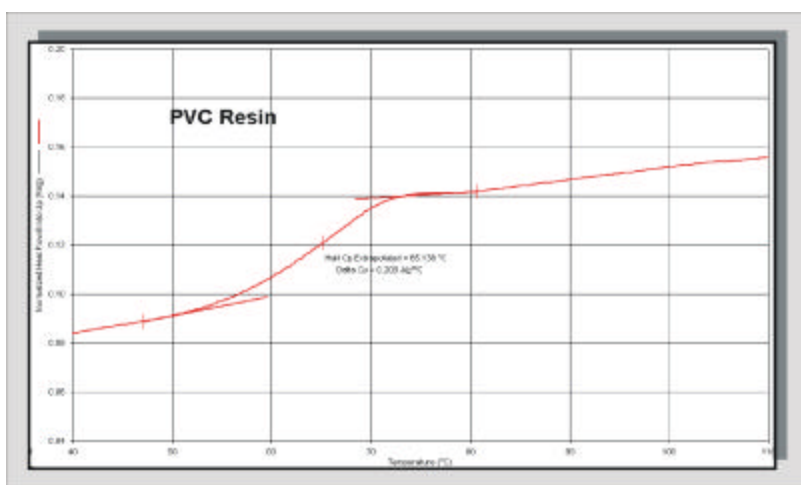


Figure 4. DSC results on PVC resin sample.



During the second heating experiment, the ABS sample still yields two well-defined Tg's at 66 and 107 C, and they are now free of their process-induced, stress relief effects. No further transitions (e.g., melting) were observed when heating the ABS recyclate between 170 and 300 C. A sample of virgin

ABS resin was analyzed using the Pyris 6 DSC under the same conditions to verify that it does not yield a second, low temperature Tg and these results are displayed in Figure 3.

The results obtained on the virgin ABS show no evidence of a Tg near 65 C. The occurrence of the Tg near 65 C on the recyclate

ABS material are definitely due to the presence of a second contaminant component. The temperature of the Tg could be indicative of the presence of a PVC contaminant. A sample of PVC resin was analyzed using the Pyris 6 DSC under the same conditions and these results are displayed in Figure 4.

The PVC resin yields a Tg at 65 C and this is in good agreement with that of the contaminant Tg observed in the ABS recyclate material. It is definitely true that the ABS recyclate contains a contaminant and it seems reasonable that the contaminant could be PVC based on the DSC results. This hypothesis can be verified using a spectroscopy technique such as FTIR.

The information provided on the ABS recyclate by DSC is very informative and demonstrates that the Pyris 6 DSC has the high performance level to detect small levels of contaminants. Processing of the ABS recyclate with the presence of the PVC contaminant would lead to severe problems because of the release of hydrogen chloride gas from the PVC component. The DSC was able to detect the presence of this and thus alert the recycling company to a potential major problem.

### Results on HDPE Recyclate

Another example of the ability of the Pyris 6 DSC to test and characterize polymeric recyclates is with HDPE (high-density polyethylene). In addition to providing useful information such as melting characteristics and percent crystallinities, the DSC also yields information on the presence of other polymer components or contaminants. Displayed in Figure 5 are the DSC results generated on a HDPE recyclate material.

The results reveal the occurrence of two distinct melting transitions at 135 and 153 C. 'Pure' or homogeneous HDPE resin should yield a single melting event and the presence of the smaller transition



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Figure 5. DSC results on HDPE recycle material.

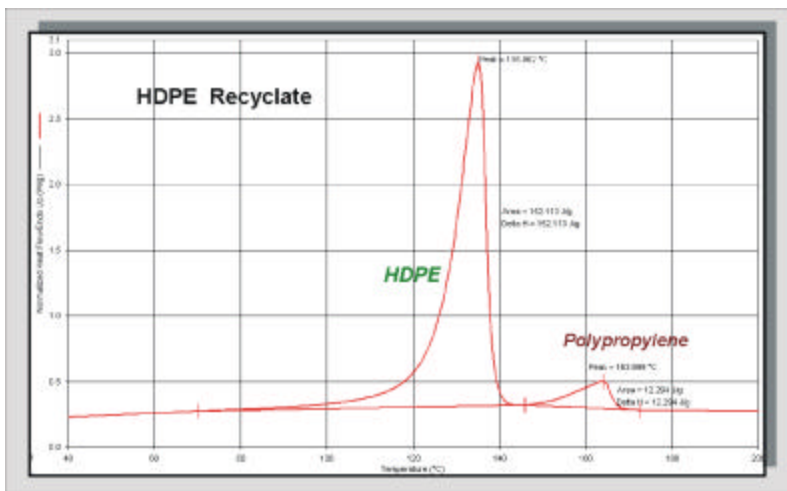
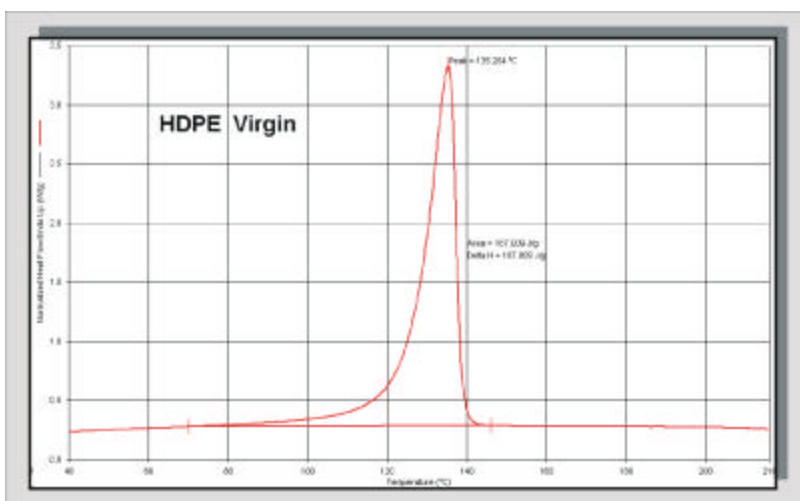


Figure 6. DSC results on virgin HDPE container resin.



at 153 C is indicative of a second component or contaminant.

Displayed in Figure 6 are the DSC results generated on a sample of virgin HDPE resin used for the production of containers for consumer products.

The virgin HDPE polymer yields a single melting transition at 135 C as would be expected for the 'pure' resin. The largest single source of contaminations in recycled HDPE comes from polypropylene since container caps are generally made from

polypropylene and can be accidentally mixed with the HDPE recycled containers or resin. The smaller, higher temperature melting transition observed in the HDPE recyclate DSC scan is most likely due to the presence of polypropylene.

The DSC heats of melting can be used to determine the percent level of polypropylene contaminant in the resin. The heat of total heat of melting is 152.1 + 12.3 J/g or 164.4 J/g for the recyclate material. The estimated level of polypropylene contaminant in the HDPE recyclate is therefore calculated as 12.3 / 164.4 or 7.5%. Not only can the Pyris 6 DSC detect a contaminant, but it can also give an estimation of the percent or level of the contaminant.

## Summary

DSC is a powerful technique for characterizing the thermal properties of polymeric materials, recyclates and regrinds. The presence of polymer 'contaminants' can be detected in both post-consumer and post-industrial recyclates using DSC, and provides useful information to ensure that the recyclate and regrinds will process successfully. It is possible to use the DSC data to estimate the level of contaminants in the resin based on the heats of melting of the components.

The PerkinElmer Pyris 6 DSC system is ideally suited for testing the thermal properties of polymers, recyclates and regrinds based on its ease of use, robust and reliable design, outstanding autosampler and high performance characteristics.

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