PET Heat History and Discoloration Test

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Introduction – Scope, Significance and Use

This evaluation – using three melt heat histories – is used to qualify PET resins for use as control resins suitable for the Critical Guidance Test procedures.

As a rule, resins and additives that lead to the least discoloration and haze formation with melt heat history are desirable for PET recycling. This evaluation can be useful in developing and selecting resins and additives for commercial use that have low color impact.

When PET is recycled, the resin experiences what are called "melt heat histories". A common situation is:

- Heat history 1: Virgin PET resin is dried with heat and then melted and molded into an article such as a preform used to make a bottle or container.
- Heat history 2: In recycling, washed flake from molded articles is dried and then melted in an extruder to allow for melt filtration and pelletization.
- Heat history 3: Pellets are dried again and melted and molded into a new article.

There are also recycling situations where PET experiences only two histories:

- Heat history 1: The virgin PET resin is dried and melted and molded into an article, such as the preform mentioned above.
- Heat history 2: Washed flake from molded articles is dried and then melted and processed directly into a new article.

This Heat History and Discoloration Evaluation provides a laboratory method that can be used to evaluate the impact of drying and melt heat history on any formation of discoloration and haze in PET resins as well as PET resins containing additives. Additives might include: UV blockers, oxygen scavengers, toners, slip agents, de-nesting additives or reheat additives for example. PET resin is commonly dried with exposure to heat in a desiccant drier prior to melt processing – this desiccant drying step may contribute to color formation in some cases.

While the APR recommends the use of three melt heat histories to best assess a test material, there are situations where only two heat histories are required to identify differences in materials and a third heat history does not add value. Individual investigators can pick the number of melt heat histories depending upon their needs, interests, and specific test requirements.

Test Method Summary

A PET test resin, or resin with additive, is exposed to two or three melt heat histories. Heat history can be generated by, for example:

- Extrusion of pellets to strands, followed by re-pelletization, or
- Injection molding of preforms or plaques followed by granulation

Investigators may use the method most convenient for them.

The final heat history must create 3 mm thick injection molded plaques of amorphous PET that are used to evaluate color and haze values by transmission in a color spectrophotometer.

It is common practice to compare the color of the test material to that of a control PET resin.

Reference documents

PET-P-00 Standard Laboratory Practices

- PET Extrusion Practice, PET-P-06
- Injection Molding 3 mm Plaques in APR Process Practices, PET-P-08
- Plaque Color Measurement, PET-S-XX

Equipment Required

- Injection molding equipment for producing 3 mm thick plaques
- Rotary granulator
- Laboratory extruder with pelletizer may be used to create melt heat histories.
- Resin desiccant drier

Materials Required

- A test resin, or a PET resin mixed with additive of interest.
- An optional PET control resin for comparison. The control resin might be a named PET control resin, or a PET resin that has been qualified earlier as a control resin using this procedure.

Test Method Steps

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1. Record the description of the test material. Examples include:
   - A PET resin made in a reactor.
   - A molded article made with an additive or additive concentrate mixed with PET ahead of injection molding or extrusion.
   - A lubricant applied to PET after sheet extrusion, or a spray applied to PET preforms or bottles after molding.
2. Subject separately, each of the test material, as well as any control material, to melt heat histories. Dry materials in a desiccant drier at 160° C prior to use in an extruder or injection molding unit. This heat history in the drier may contribute to over-all color formation and is a necessary step of the evaluation.
3. The final heat history for each material must be to form 3 mm thick injection molded plaques. Plaques are made using 100% of the control resin in one case, and 100% of the test resin in another.

Test Assessment

Data will reveal the impact of test material on color and haze. Qualitatively, the most desired outcome is that the test material has negligible, if any, impact on color and haze values when compared to the control resin. No specific APR Guidance is available for this test today.

For Control Resin qualification: The resin used for the test, after three drying and melt heat histories, will:

- Have an L value greater than 82
- Have b* less than 4 units
- Haze % less than 9.5

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 16, 2018	Original Document
2	September 3, 2024	Updated hyperlinks to match new website