PET IV Build Rate Evaluation

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Introduction – Scope, Significance and Use

This document presents a method to evaluate the rate and extent of IV (intrinsic viscosity) build for crystallized PET pellets during solid state polymerization conditions.

The Critical Guidance Protocol for PET Resins and Additives, PET-CG-01 includes an evaluation of IV build rate to confirm that new resins or additives do not interfere with this important PET recycling process step. The evaluation compares the of IV build rate of an innovation compared to a control PET Resin. The evaluation also confirms that the innovation sample can be solid stated to at least a 0.90 dl/g value required for some recycled PET applications.

Reference Documents

PET-P-07 – Solid State Polymerization of PET
ASTM D4603
ASTM D1238 Method B

Test Method Summary

This test method evaluates the IV build rate of crystallized PET pellets. The solid stating rate of a control resin is measured over eight hours' time, and then the solid stating rate of a test material is evaluated under the same conditions.

The test does not call for a specific solid stating process; individual laboratories generally have their own custom equipment for conducting solid stating evaluations of PET.

In a solid stating evaluation is critical to control:

- The starting moisture content of samples.
- The temperature.

- The moisture content in the atmosphere surrounding the PET pellets.
- The pellet size of the PET.

These requirements can be achieved by:

- Carefully producing the PET pellets to have similar size.
- Screening pellets used for the evaluation to remove those that are over/under sized.
- Conditioning the samples before evaluation to allow similar moisture content.
- Solid stating the pellets in a vacuum rotary drier, or
- Solid stating the pellets in a thermostatted container under vacuum, or
- Solid stating in a thermostatted container swept with dry nitrogen.

Equipment Required

Any equipment and apparatus suitable for conducting solid state polymerization with a temperature of at least 205° C is acceptable. Some common example approaches include:

1. The rotary vacuum solid stating reactor described in the PET Practices, PET-P-06
2. A vacuum container with:
   - Vacuum oven, or other vacuum container, capable of reaching temperature of minimum 205°C and controlling at that temperature.
   - Vacuum pump capable of -736.6 mmHg within 2-5 minutes
3. Alternatively, a thermostatted container to hold PET resin that can be continuously swept with dry nitrogen may be used.

Materials Required

- Pellets of crystallized control
- Pellets of crystallized test blend(s)

Test Method Steps

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1. Measure and record the IV values for all resins and blends involved in the evaluation.
    a. The solution IV method described in ASTM D4603 can be used for any sample.
    b. ASTM method 1238-13 may be used to measure the melt viscosity of a PET sample, and then the melt viscosity can be correlated to the solution IV value using a calibration curve.
2. Confirm the pellet size of control and test resins are similar. Screening the pellets to remove over-sized and under-sized pellets is recommended.
3. Condition all samples under the same storage conditions prior to solid stating so that all samples in the evaluation have similar starting point moisture level.
4. Conduct solid stating of each resin in the evaluation for a period of 8 hours. The time of exposure to solid stating conditions begins when the resin sample reaches 190° C. A minimum temperature of 205° C should be used. 215 ° C will generally provide faster IV build rates and so provide more satisfactory results. Any of the solid stating approaches listed below for illustration are acceptable:
    a. The use of a rotary vacuum unit is described in PET-P-07 describing a practice for Solid State Polymerization of PET pellets.
    b. Illustrative vacuum oven method and a nitrogen sweep method are outlined below.
        1) Pellet samples can be contained in a clean aluminum dish.
        2) Raise temperature of vacuum oven to 210°C +/- 5°C
        3) Once oven has reached required temperature pull a vacuum.
        4) Break vacuum with purging nitrogen.
        5) Open door and quickly place required samples in the oven.
        6) Pull vacuum immediately. (Samples will take approximately 5-10 minutes to come to temperature depending on oven size.)
        7) After 8 hours of time, remove samples for IV evaluation.
    c. Illustrative N2 Flow SSP Reactor Method
        1) Load PET into the SSP container and seal closed.
        2) Begin the nitrogen gas flow at 5-10 scf/h with a target of 7.5scf/h
        3) Begin heating the unit to reach a pellet temperature of 210°C +/- 5°C
        4) Once the pellet reaches a temperature of 190°C the time for sample testing can be started.
        5) After 8 hours of time, remove samples for IV evaluation.

5. Evaluate the IV of all samples after an 8 hour solid stating time increment using either ASTM D4603 or ASTM D1238 Method B
6. Providing the control and the test resins have achieved a minimum value of 0.90 dl/g, there is no need for additional solid stating exposure.
7. If any test resin blend has not achieved 0.90 dl/g value within 8 hours, that resin sample may be solid stated for an additional time increment of up to 7 hours, for a total exposure time of 15 hours to determine whether the resin can achieve a 0.90 dl/g value.

IV Build Rate Evaluation

1. Calculate the secant IV build rate for the control resin in units of dl/g·hr. that is simply … (the ending IV value – the starting IV value)/8 hours
2. Calculate the secant IV build rate for the test resin over 8 hours.
3. For the sake of calculation, normalize the starting IV of the test resin blend equal to that of the control. Calculate the expected IV of the test blend after 8 hours of solid stating time.

A preferred result meets these two criteria:

- There is no greater than 0.040 IV units difference for the value for the control resin minus the calculated IV of the test resin at 8 hours.
- The test blend achieves a minimum 0.90 IV within a total of 15 hours solid stating time.

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 16, 2018	