CONFIDENTIAL

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Determination of the Gel Count in Low Molecular Weight Polyvinyl Chloride for Rigid Applications using a Brabender Extruder

APPLICATION: This method uses the Brabender extruder for the determination of the gel level in low molecular weight resin (10R) for rigid application. Extruded sample strips are compared to standards to provide a numerical gel grading level.

APPARATUS:

1. Brabender Plasticorder, Model PL-V300 (3 hp. drive) equipped with: 3/4" extruder, 25":1" length/diameter; fitted with 5:1 compression ratio screw and 4" x 0.020" die head; feed hopper, water cooled; temperature control unit; pressure gauge 0-6000 psig
2. Take up rolls
3. Triple beam balance ± 0.1g readout, or equivalent
4. Blender, Ronson "Cook'n Stir"
5. Kodak timer
6. 20 mesh Tyler screen
7. Gauge, thickness ± 0.1 mil readout
8. Industrial vacuum cleaner
9. Cylinder, cleaning (similar to a graduated cylinder of 1000 ml capacity, however, fabricated out of 316 S.S.) 2" diameter, 27" high.
10. Vessel, cleaning (large enough to hold the separated die head and die head adaptor)
11. Quart glass jar or quart fiber container
12. Brass spatula
13. Cleaning rod fitted with a brass brush and gun cleaning patches 4" diameter or gauze 2" wide
14. Vibrator fitted to the feed hopper

15. Beakers, 100 ml, 600 ml
16. Brown wrapping paper
17. Asbestos gloves
18. Grease gun

REAGENTS:

1. Irvinil 640 (Standard PVC)
2. Thermolite 31 stabilizer
3. Acryloid K-120N
4. Nuoplaz 849
5. Primol 205
6. Compounded PVC, Clear
7. Tetrahydrofuran, Reagent Grade
8. Silicone grease, Lubricating Grade

SAMPLE PREPARATION:

1. Set the blender temperature control to 200°F and allow the blender to warm up.
2. Into a previously tared 600 ml beaker weigh 400 gm of the sample polyvinyl chloride and 8 gm of Acryloid K-120N.
3. Into a previously tared 100 ml beaker weigh 12 gm of Thermolite 31, 4 gm of Nuoplaz 849, 2 gm of Primol 205.
4. Transfer the contents of the 600 ml beaker into the blender set the function selector to "blend" and start blending at speed setting of 3, after 5 minutes of blending, pour the contents of the 100 ml beaker slowly into the blender.
5. After additional 5 minutes of blending, stop the blender and spread the dry blend on a piece of wrapping paper. CLEAN THE BLENDER THOROUGHLY.
6. Allow the dry blend to cool to room temperature. Screen to 20 mesh and transfer to a quart jar or container.
7. Prepare all samples following steps 1. through 6. as well as a standard using Irvinil 640 instead of the 400 gm of the sample Polyvinyl Chloride.

PROCEDURE:

1. Install the die head and extruder barrel pressure gauge.
2. Turn on the cooling water for the feed hopper.
3. On the temperature control unit, turn on the main power switch and the switches

for zone 1 through 4. Set the temperature gauges to the following maximum settings:
Zone 1: 345°F, Zone 2: 350°F, Zone 3: 360°F, Zone 4: 370°F.
Adjust the variable transformers to a current of 1 ampere for Zone 1 to 3 and for Zone 4 to 3 amperes. Let equilibrate for 25 minutes after maximum temperature has been reached. (Note 1)

4. Set the plasticorder to a 1:5 sensitivity and 5 x scale. Set the damper minus 4 notches.
5. Start the extruder and adjust the screw speed to 30 rpm, turn on the chart drive. Check the zero, adjust the zero suppression to 2000 m/gm. The full scale range is 2000 - 7000 m/gm of torque.
6. Place the take up rolls in front of the extruder.
7. Start the hopper vibrator.
8. Fill the hopper 1/3 with the compounded PVC.
9. Start the take up rolls and thread the ribbon through the rolls. Adjust the speed of the take up roll so that the thickness of the ribbon is about 5 mil ± 0.5 mil. Repeat step 8, after the hopper is almost empty.
10. After the hopper is empty again fill the hopper with the dry blended Irvinil 640 (standard). Run until a level torque line is reached and the ribbon is clear from the compounded PVC. After 5 minutes cut out a ribbon sample.
11. When the hopper is empty vacuum the remainder of the dry blend from the hopper, dislodging any sticking polymer with a gentle blow from a forced air nozzle.
12. Fill the hopper about 1/3 with the sample dry blend.
13. Run for 5 minutes and cut out a ribbon sample.
14. Repeat steps 12. and 13. for all the dry blended samples and rerun the Irvinil 640 at the end of the sample runs.

15. Run the extruder empty. Turn off the heat (turn the variable transformers to 0 setting) and the chart drive. Set zero suppression to maximum on the plasticorder.
16. Remove the die head from the adaptor. Separate the die halves and remove the remaining polymer. (Note 2)
17. After the die has cooled, to hand warm, place the die in a vessel filled with tetrahydrofuran for the final cleaning step. (Stubborn traces of polymer are removed with a brass spatula). (Note 3)
18. Remove the adaptor from the barrel by removing the adaptor collar and carefully remove the polymer plug.
19. Fill the hopper with compounded PVC to about 1/2 (the barrel temperature should be now about 300°F). Purge the barrel of the cleaning PVC. Remove the pressure gauge and regrease if needed. Reinstall the die adaptor. Turn off the cooling water. (Note 4).
20. Grade the sample ribbon by comparison with standard ribbons.

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Issued by: /s/ H. B. Carr
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Manager - Quality Control

Attachment

Note 1: A slow warm up will prevent the barrel from crazing or cracking because of overheating. This is because heaters can introduce more heat than steel is able to conduct. Therefore, the life of the barrel and screw will be increased.

Note 2: The remaining polymer is best removed by blowing compressed air between the die and polymer while pulling on the polymer with a pair of pliers. Do not allow any tools but brass tools to come into direct contact with the die metal.

Note 3: When the die is being reassembled heat the loosely assembled die in an oven to about 200°F and tighten the screws of the die while the die is hot.

Note 4: After every 4th run remove the screw from the barrel for cleaning the following way:

1. Push the screw out from the rear of the barrel (hopper).
2. Grasp the screw at the die end of the barrel and pull out from the barrel. Clean the barrel with the cleaning rod by wetting the patches or gauze with THF. Repeat until barrel is clean.
3. Insert the hand warm screw into the cleaning cylinder filled with THF. Let sit overnight. Wipe off the swollen polymer. Remove the stubborn polymer traces with the brass spatula. Reinstall the screw and the die adaptor.