

TENNECO INTERMEDIATES DIVISION

TENNECO CHEMICALS, INC.

QUALITY CONTROL

TESTING METHOD

DATE: November, 1973

METHOD NUMBER: 2-9

TITLE: Determination of static heat stability - homopolymer

APPLICATION: Heat stability is determined from milled chips of compounded resin that have been subjected to heat for a period of time and is computed as the ability to withstand discoloration when compared to a standard compounded resin.

APPARATUS:

1. 600 ml polyethylene beaker
2. Torsion balance, 3000 gm capacity
3. Mill, two roll, heated
4. Paper cutter
5. Aluminum foil
6. Metal wedges, all wedges must be cut from the same piece of steel, .040"
7. Oven with rotating shelves at 180°C
8. Surface pyrometer
9. Timer

CHEMICALS:

Sample to be tested
Standard resin
Di-octyl phthalate
Nuoplaz 849
Nuostab V-1399
Stearic acid
Mill cleaning compound

PROCEDURES:

1. Set the mill at 300°F (310°F in Burlington) for all resins except 150, 10-5R and 175 types. Types 175 and 10-5R are

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milled at 275°F and 150 type is milled at 250°F. Check the temperature of the mill with the surface pyrometer.

2. Set the thickness of the mill to give a sheet 40 mils thick; this is done by using the cleaning compound, which is to be used before each run. After the bond is made on the cleaning compound, stop the mill, cut off a piece of the sheet and check the thickness with the mil gauge. If it is not the proper thickness, make necessary adjustment, start the mill and repeat the prior steps until desired thickness is obtained.

Once the desired setting is obtained, turn down the screws (the screws may be marked so as to preclude this step on future testing).

3. When the mill is clean, remove the cleaning compound and stop the mill.
4. To a tared 600 ml polyethylene beaker, add the following ingredients:

100 gm of sample to be tested
51.5 gm of H.S. Wet mix
1.5 gm of *H.S. lubricant mix

*For all resins with a viscosity below 2.00, use the low molecular weight lubricant mix.

5. Mix the ingredients thoroughly, using a metal spatula.
6. Set the timer for five minutes, pour the entire mix on the mill and start the timer and mill at the same time.

Note: In order to get a bond, it is necessary to turn the rollers in as far as possible at the start of the test. When the bond is obtained, turn the rollers back to the 40 mil setting for the duration of this step.

7. Work the stock on the mill rolling it from one end of the rollers to the other.
8. After five minutes of milling, cut the stock on the underside of the rollers, and remove it from the mill.
9. Using a paper cutter, cut approximately twenty 3/4" square chips from the milled sheet while the sheet is still warm.
10. Process some of the standard resin in the same manner.
11. Cover the metal wedges with aluminum foil, place a standard and sample chip on each of the eight metal wedges. Be sure the standard and sample chips are placed in the same position on each of the eight wedges.
12. Place the samples on the rotating shelf in the oven which had previously been set at 180°C.

13. Remove one wedge at a time from the oven according to the following suggested time schedule:

40 minutes
60 minutes
80 minutes
100 minutes
120 minutes
130 minutes
140 minutes
150 minutes

These times may be varied by supervision if it is found that they are not applicable to the product being tested at the time.

PREPARATION OF H.S. MIXES

A. H.S. Lubricant Mix - High Molecular Weight

1. Into a clean, tared container weigh 1000 gm of a regular, low fisheye, 225 resin and add 100 gm of stearic acid.
2. Blend for ten minutes at medium speed in a Hobart mixer.
3. Store in a clean container.

B. H.S. Lubricant Mix - Low Molecular Weight

1. Same as above lubricant mix except 150 resin in place of the 225 resin.

C. H.S. Wet Mix

1. Into a clean, tared one gallon brown bottle, weigh accurately the following:
1800 gm Di-octyl phthalate (DOP)
200 gm Nuoplaz 849
60 gm Nuostab V-1399
3. Shake vigorously and store in a dark place.

CALCULATIONS: A direct comparison is made between the time of failure of the standard versus the sample being tested.

Compare the first chips removed from the oven with the standard and rate as + if it is lighter than the standard, 0; if it is equal and - if it is darker.

Time of failure is when the edges of the chips are completely blackened.

$$\text{H.S. Rating} = \frac{\text{time of failure of sample}}{\text{time of failure of standard}} \times 100$$

REPORT: Report the heat stability to the nearest whole percent.

SAFETY: Gloves should be worn when operating the mill; they are to be cut on the backside to facilitate quick removal if they get caught in the mill. No loose clothing should be worn while operating the mill, i.e., neckties or long sleeves with the cuffs unbuttoned.

REMOVING STOCK FROM THE ROLLING BANK OF THE MILL WHILE IT IS MOVING IS FORBIDDEN: When the material is to be removed, stop the mill and remove the excess material.



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