Monsanto Chemical Company

Anniston Method No. 14-17-48

SUBJECT: Softening Point of Solid Aroclors

METHOD: Ball and Ring

This method is a modification of the A.S.T.M. standard method of test for softening point of bituminous materials, serial designation: E 28-397. The method differs from the standard method in that the rings are larger than specified. A two ring support is also used in order that two tests may be made simultaneously.

Apparatus

The apparatus consists of the following: (a) Two tapered brass rings, 5/8" inside dia. at bottom, 11/16" inside diameter at top and 1/4" deep: thickness of wall 3/32". (b) Two steel balls, 3/8" diameter weighing 3.45 to 3.55 grams each. (c) A 800 ml. Griffin low form beaker. (d) A ring support for the two rings having a brass plate exactly one inch below the plate supporting the ring. (e) A 300°C. A.S.T.M. low distillation thermometer graduated 1°C.

Preparation of the Sample

The sample shall be melted and stirred thoroughly, avoiding overheating or incorporating air bubbles in the mass and then poured into the ring so as to leave a slight excess on cooling. Since the Aroclors shrink considerably on cooling, the ring should be well filled, nearly to overflowing. A little of the excess should be drawn over the top of the ring at several points so as to prevent the cooled Aroclor from dropping out of the ring. In the same way, the second ring is filled with a standard Aroclor of known softening point. The standard Aroclor should have a softening within at least 10 to 15° of the Aroclor being tested.

The rings while being filled should rest on a clean can lid or on a brass plate which has been amalgamated to prevent the Aroclor from adhering to it. The Aroclor in the rings should be fully cooled and hardened before proceding with the test.

Procedure

Add cool solution (employ water for softening point between 0-80°C, glycerin for between 80-200°C., mineral oil for above 200°C.) to the beaker until the surface of the solution is 2" above the plate holding the rings when the ring support is suspended in the beaker. Place the rings containing the Aroclor to be tested and the standard Aroclor on the ring support. Place a ball on the center of the upper surface of the Aroclor in each ring. Suspend the thermometer so that the bottom of the bulb is level with the bottom of the rings and just midway between the two rings.

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Place beaker and apparatus on a 6 inch round hot plate and heat at such a rate that the temperature is raised 5°C. each minute.

The temperature recorded by the thermometer at the instant the Aroclor touches the bottom plate is reported as the softening point. The heating is continued until both Aroclors have dropped to the bottom plate. No corrections are made for emergent stem.

Notes

1. The standard Aroclor is run along with the sample under test in order to compensate for variations in rate of heating, dilution of glycerin, and thermometer variations.

2. The softening point of the standard sample is determined by making several determinations using fresh glycerin and carefully checking the rise in temperature of the glycerin so that it is as near as possible to 5°C. per minute.

3. The glycerin may be used repeatedly for the tests after removal of the Aroclor.

4. Benzol is used for cleaning the rings and balls.

5. Water can be used instead of glycerin for softening points up to 90°C.

6. For softening points above 100°C, well boiled glycerin should be used. The usual glycerin is not satisfactory above 125 to 130°C, it boils with loss of water while the temperature remains practically constant. It is well to have a supply of high-boiling glycerin on hand to be used only for softening points above 100°C.

7. Air bubbles on the rings and balls during the test should be avoided as far as possible.

8. The rate of rise of temperature of the glycerin shall be uniform for each minute after the first 3 minutes of heating and not averaged over the period of the test.

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