



SECTION 4 TESTING

Proper interpretation of laboratory test results can greatly aid in determining the traits of an asphalt emulsion. As advances have been made in asphalt emulsion technology, corresponding advances in emulsion testing have evolved. Some of these tests are designed to measure performance qualities. Others deal with composition, consistency, and stability of the material. Laboratory tests are normally performed for these purposes:

- To provide data for specification requirements
- To control the quality and uniformity of the product during manufacturing and use
- To predict and control the handling, storage, and field performance properties of the material

A review of emulsion specifications used across the United States reveals a wide variety of requirements. Many are directly related to the emulsions produced by specific manufacturers. Because it is impractical to discuss the multitude of requirements and test methods, this chapter deals primarily with the methods in ASTM D 244 and AASHTO T 59. However, a few non-ASTM tests that are often used, especially with emulsions that have been modified with polymers, have also been included. ASTM is considering the adoption of some of these tests.

There are a number of new tests, mostly for asphalt cements, that are in the developmental stage. For instance, SHRP came up with new test procedures, testing apparatus, and specifications that are part of the Superpave binder specification. Many of these tests are currently available and could probably be applied to asphalt emulsion residues. The tests are designed to better measure the various properties of asphalt cements, emulsions, emulsion residues, and modified systems. They will be included in this manual as they become incorporated in asphalt emulsion specifications.

This chapter will first consider the tests that apply to asphalt emulsions. It then describes the tests run on the asphalt emulsion residue after the water has been removed by distillation or evaporation.

4.1 Asphalt Emulsion Tests

Proper sample handling is important to achieve valid test results. Asphalt emulsions are made hot, some are stored hot, and some are transported and applied hot. Hot samples collected in the field are often delivered to the laboratory at ambient temperatures. Asphalt emulsion samples with viscosity requirements at 50°C (122°F) should be heated to $50 \pm 3^\circ\text{C}$ ($122 \pm 5^\circ\text{F}$) in a 70°C (160°F) water bath or oven. Samples should be stirred, not shaken, to insure homogeneity.

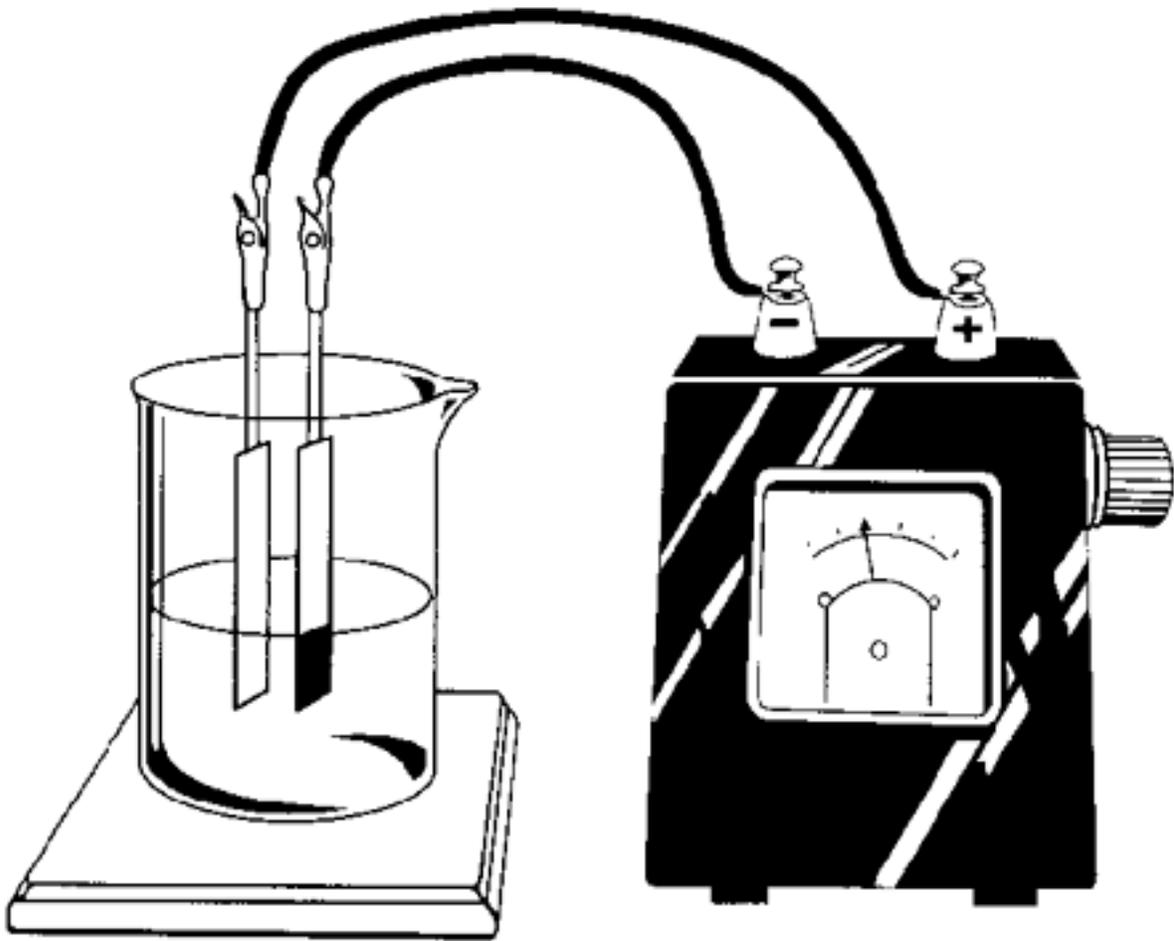


Figure 4-1 Particle Charge Test

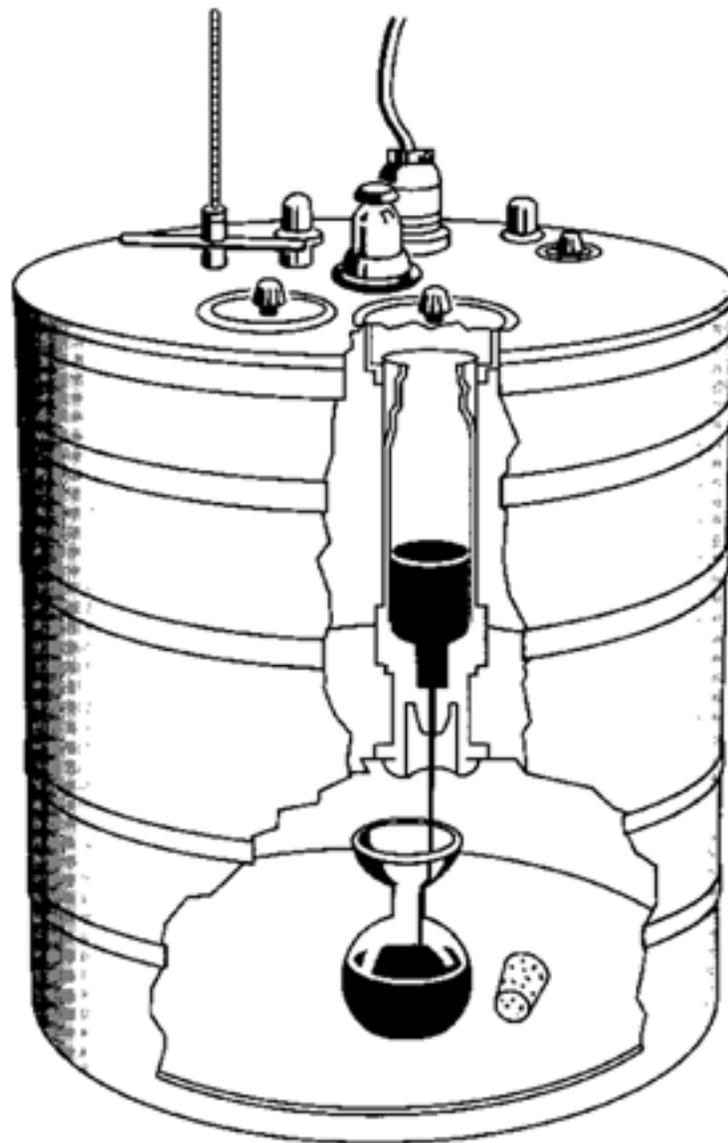


Figure 4-2 Saybolt Furol Viscosity Test

4.1.1 Particle Charge Test

The particle charge test is used to identify cationic emulsions. It is performed by immersing a positive electrode (anode) and a negative electrode (cathode) into an emulsion sample and connecting them to a controlled direct-current electrical source ([Figure 4-2 Saybolt Furol Viscosity Test](#)). At the end of the test period, the electrodes are observed to determine if the cathode has an appreciable layer of asphalt deposited on it. If so, the emulsion is cationic.

4.1.2 Emulsion Viscosity

Viscosity is defined as a fluid's resistance to flow. For asphalt emulsions, the Saybolt Furol viscosity test ([Figure 4](#)) is used as a measure of viscosity. Results are reported

in Saybolt Furol seconds. Depending on the type of emulsion, one of two testing temperatures are used, 25°C and 50°C (122°F and 122°F).



4.1.3 Demulsibility

The demulsibility test indicates the relative rate at which the colloidal asphalt globules in the rapidsetting asphalt emulsion will break when spread in thin films on soil or aggregate. Calcium chloride causes the minute asphalt globules present in the anionic asphalt emulsion to coalesce. A solution of calcium chloride and water is thoroughly mixed with the RS emulsion and the mixture is poured over a sieve to determine how much the asphalt globules coalesce. Specifications prescribe the concentration of the solution and the minimum amount of asphalt to be retained on the sieve (typically 60 percent). Rapid setting emulsions are expected to break almost immediately upon contact with aggregate, such as when chip sealing.

A similar test is run on cationic rapid-setting emulsions. However, rather than calcium chloride solution, a solution of dioctyl sodium sulfosuccinate is used.

4.1.4 Identification Test for Cationic Rapid-Setting Emulsion

This is a recent ASTM D 244 test that has taken the place of the classification test. Like the classification test, it involves the coating of silica sand. In this new test, the sand is first washed with hydrochloric acid and isopropyl alcohol, but unlike the classification test, no Portland cement is used. The emulsion is mixed with the sand for two minutes. At the end of the mixing period, an excess of uncoated area compared to the coated area is considered positive identification of a cationic rapid setting emulsion.

4.1.5 Identification of Cationic Slow-Set Emulsions

This is also a relatively new ASTM D 244 test that is used if the result of the particle charge test is inconclusive. A weighed amount of washed and dried silica sand is mixed with a weighed amount of CSS asphalt emulsion and mixed until the aggregate is completely coated. The amount of emulsion in the mix should be 5% by total weight of sand. The mix is cured for 24 hours and then placed in a beaker of boiling distilled water. After 10 minutes, the sample is placed on a level surface and the coating is observed. If the coating is in excess of 50% of the total mix, it is considered a positive test for a cationic slow setting emulsion.

4.1.6 Settlement and Storage Stability Tests

These tests indicate the emulsion's stability in storage. They detect the tendency of asphalt globules to settle over a period of time. A prescribed volume of emulsion is allowed to stand in a graduated cylinder for a specified time (five days for the settlement test and 24 hours for the storage stability test). Samples are then taken from the top and bottom of the cylinder. Each sample is placed in a beaker and weighed and heated to evaporate the water. The residue is then weighed. The weights obtained are used to find the difference, if any, between the asphalt residue content in the upper and lower portions of the cylinder. This provides a measure of settlement. Many agencies will accept the 24-hour test, while others require the five-day test.



4.1.7 Cement Mixing

The cement mixing test does the same for slow setting asphalt emulsions as the demulsibility test does for rapid setting grades. In the field, the SS grades are often mixed with fine materials and dusty aggregates. In the cement mixing test, a sample of asphalt emulsion is mixed with finely ground Portland cement and the mixture is washed over a 1.40 mm (No. 14) sieve. Specifications limit the amount of material that can be retained on the sieve. The result of this test indicates the ability of a slowsetting emulsified asphalt to mix with a high surface area material without breaking.

4.1.8 Sieve Test

The sieve test is another measurement of quality and stability of the emulsion. The retention of an excessive amount of asphalt particles on a sieve indicates that problems may occur in the handling and application of the material. In the sieve test, a representative sample of asphalt emulsion is poured through an 850- μm (No. 20) sieve. For anionic emulsions, the sieve and retained asphalt are rinsed with a mild sodium oleate solution and then with distilled water. For cationic emulsions, distilled water only is used for rinsing. After rinsing, the sieve and asphalt are dried in an oven and the amount of retained asphalt determined by weighing.

4.1.9 Coating Ability and Water Resistance

This test has three purposes. It determines the ability of an asphalt emulsion to:

1. Coat the aggregate thoroughly
2. Withstand mixing action while remaining as a film on the aggregates
3. Resist the washing action of water after the completion of mixing.

The test is primarily used to identify mediumsetting asphalt emulsions suitable for mixing with coarse-graded calcareous aggregates. This test is not adaptable to rapid-setting or slow-setting asphalt emulsions.

The reference aggregate is coated with calcium carbonate dust and then mixed with the asphalt emulsion. About one-half of the mixture is then placed on absorbent paper for a visual inspection of the surface area of aggregate coated by the asphalt emulsion. The remainder of the mixture is sprayed with water and rinsed until the rinse water runs clear. This material is then placed on absorbent paper and inspected for coating. The test is then repeated and for this second run, the aggregate is coated with water before the emulsion is added, mixed and then visually inspected for good, fair or poor coating ability.

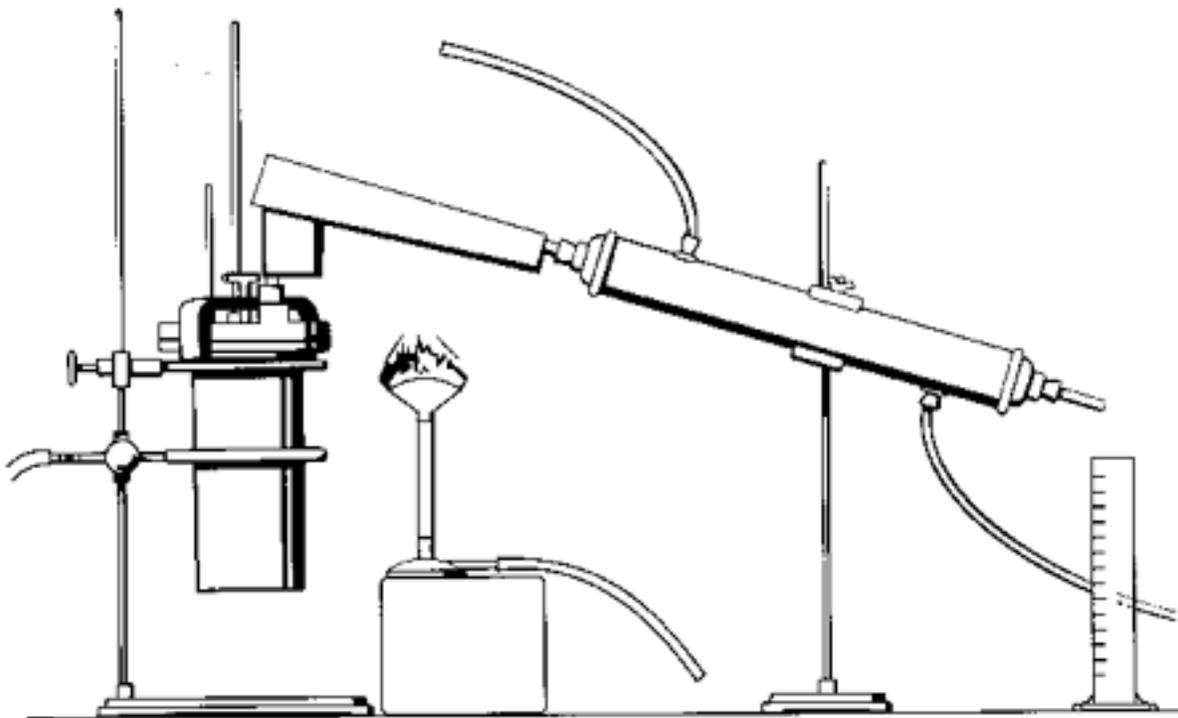


Figure 4-3 Distillation Test for Asphalt Emulsion

4.1.10 Field Coating Test

Field coating tests are used at the project site to determine:

- The ability of an asphalt emulsion to coat the job aggregate
- The ability of the emulsion to withstand mixing
- The water resistance of the emulsion-coated aggregate

Measured amounts of the job aggregate and job emulsion are hand mixed. The ability of the aggregate to remain coated during a five-minute cycle is observed. The resistance of the coating to wash-off is determined by filling a container of the coated aggregate with water and emptying it five times. The coating of the aggregate is visually rated as good, fair, or poor. A rating of good means that the aggregate is fully coated (except for pinholes and sharp edges). A rating of fair indicates an excess of coated over uncoated aggregate area. A rating of poor indicates an excess of uncoated over coated aggregate area.

4.1.11 Unit Weight of Asphalt Emulsion

The unit weight (kg/liter or lb/gallon) is computed by finding the weight of an asphalt emulsion in a standard measure of known volume. Results are reported to the nearest 0.01 kg/liter at 25°C (lb/gal at 77°F).



4.1.12 Residue and Oil Distillate by Distillation

Distillation is used to separate the water from the asphalt. If the material contains oil, it will be separated along with the water. The relative proportions of asphalt cement, water, and oil in the emulsion can be measured after the distillation has finished. Additional tests may be run on the asphalt cement residue to determine the physical properties of the end-use asphalt.

The distillation test procedure for asphalt emulsion uses an aluminum alloy still and ring burners (**Figure 4-3 Distillation Test for Asphalt Emulsion**). Normally, the distillation is run at a temperature of 260°C (500°F) for fifteen minutes. Since the emulsion seldom approaches this temperature in the field, it should be noted that some residue properties could be altered, such as elastic properties given by polymer modification. Some agencies have changed the temperature and time at which the distillation test is run for these special products.

4.1.13 Residue by Evaporation

The oven evaporation test is carried out in an oven at a temperature of 163°C (325°F) for three hours. This test may be used in lieu of the distillation test, but the procedure usually yields lower penetration and ductility results than the distillation test. The evaporation test may not be used if a float test is to be run on the residue.

4.2 Examination of Residue

The same desirable characteristics in the base asphalt cement should show up in the residual asphalt after emulsification and coalescence. In some cases, the properties are actually improved. The most common tests run on the residue include specific gravity, solubility in trichloroethylene, penetration, ductility and the float test. These tests are described in detail in ASTM D 70, D 2042, D 5, D 113 and D 139 (AASHTO T 228, T 44, T 49, T 51 and T 50) respectively.

Specific gravity of the residue is usually not specified by user agencies. However, the information is often useful for making volume corrections at various temperatures.

The solubility test is a measure of the bituminous portion of the asphalt residue. The portion that is soluble in trichloroethylene represents the actual asphalt binder and the insoluble portion represents inorganic contaminants. Solubility is determined by dissolving the asphalt cement in the solvent and separating the soluble and insoluble portions by filtering.

The penetration test is a measure of the hardness of the asphalt residue at the specified test temperature. This test measures the depth of penetration of a standard needle under a load of 100 g for five (5) seconds at a temperature of 25°C (77°F). However, other temperatures and loads are sometimes used when additional information is desired.

The ductility of asphalt is its ability to be extended or pulled into a narrow thread. This test is run by molding a briquette of asphalt cement under standard conditions and dimensions. The asphalt briquette is then brought to a specified test temperature in a water bath. It is pulled at a specified rate of speed until the thread connecting the two

ends breaks. The elongation at which the thread of material breaks is designated as ductility.



The float test is performed on the residue from distillation of high float asphalt emulsions. The test is a measure of the resistance to flow at an elevated temperature. In the test, a test plug is formed by cooling the asphalt residue in a brass collar (Figure 4-4 Float Test). The collar is then screwed into the bottom of an aluminum float and placed into a testing bath of water heated to 60°C (140°F). The time required for the hot water to break through the plug is measured in seconds.

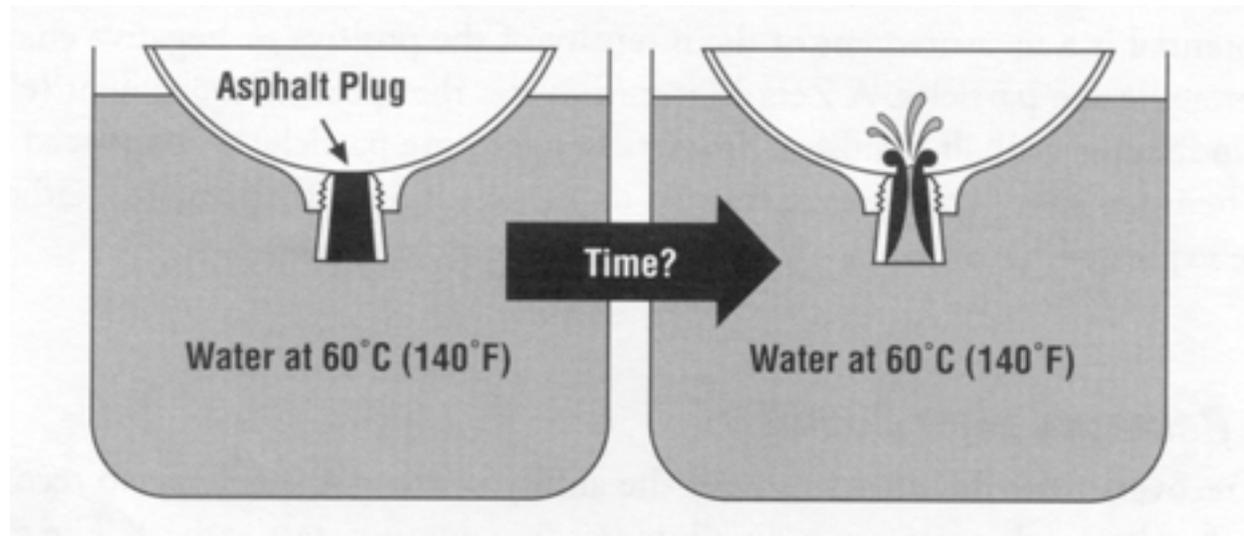


Figure 4-4 Float Test

4.3 Additional Emulsion and Residue Tests

In recent years other tests that are not included in the ASTM or AASHTO standards have been developed to measure unique properties of asphalt emulsions and their residues. Many of these methods were designed to test materials that have been modified by polymers or other modifiers. Some of these tests are discussed below and ASTM and/or AASHTO are considering a few for adoption. The first three tests given are run on the emulsion and the others are run on the emulsion residue.

4.3.1 Breaking Index

The breaking index test is designed to measure the quickness of break of very rapid setting asphalt emulsions used for chip seal. Specified silica sand is added with stirring to 100 grams of the emulsion under controlled conditions of rate and temperature. The weight of the sand in grams needed to cause the system to break (turn black and sticky) is considered the breaking index.



4.3.2 Vialit Test

The Vialit procedure has been found quite useful in the design of surface treatments (chip seals) using an RS or CRS asphalt emulsion. The asphalt emulsion is poured at its design application rate onto a metal plate and allowed to flow to a uniform film thickness. Aggregate is applied to the binder-covered plate and embedded by rolling. After it is cured, treated with water and dried again, the plate is turned upside down. A 500 gram steel ball is dropped from a specified height three times on the reverse side of the plate and the weight loss of the aggregate is measured. There are various ways of reporting the results. This test is particularly helpful using the actual job aggregate under the field humidity and temperature conditions.

4.3.3 Zeta Potential

The Zeta Potential is a measurement of the intensity of the positive or negative charge of asphalt emulsion and/or aggregate particles. A Zeta Meter measures the speed of movement (electrophoretic mobility) of individual asphalt emulsion droplets or aggregate particles when placed in an aqueous medium. The intensity of charge each droplet or particle inherently possesses, either positive or negative, can be expressed in millivolts (1/1000 V) as the Zeta Potential.

4.3.4 Elastic Recovery after Ductility

The elastic recovery after ductility measures the ability of the test specimen to recover to its original length after being elongated in a ductilometer (see ductility test earlier). The sample is pulled to a specified length at a specified temperature. The thread is then cut in the middle. After a specified time, the thread is measured again to see how closely it returns to its original position. The percentage of recovery is reported.

4.3.5 Force Ductility

The force ductility is also run in a ductilometer (see ductility test earlier). Unlike the regular ductility test, the actual force that is necessary to cause this elongation is measured. A special force ductility adapter is necessary for this method and a strip chart recorder or computer data acquisition set-up is also required. The measured results may be reported in several ways. One of the most common is the ratio obtained from the first and second peaks yielded by the graph.

4.3.6 Ring and Ball Softening Point

The ring and ball softening point test (ASTM D 36/AASHTO T 53) was developed not by the road paving industry but rather by the roofing and waterproofing industry. It is another method of measuring a rheological property of an asphalt or residue at elevated temperatures and is used for specification purposes by some agencies. Two disks of the sample in brass rings are heated at a controlled rate in a liquid bath. Each disc supports a steel ball. The softening point is the temperature at which the discs of asphalt soften enough to allow the balls to drop.

4.3.7 Tensile Strength (Stress)

Similar to the force ductility test, the tensile strength is a measure of the force required to stretch the sample. Rather than the horizontal ductilometer, however, this test uses an upright tensile pull machine. A strip chart recorder or computer plots the stress (force) against the strain (elongation). The result is usually reported as the stress in kg/cm^2 at a specified elongation (strain).



4.3.8 Torsional Recovery

Torsional recovery is also used to measure the elastic properties of a modified asphalt residue. This test measures the recovery of a sample, and is conducted in a three-ounce tin or penetration cup. A shaft and arm assembly is immersed in the molten sample and upon cooling is twisted to a 180-degree arc from the starting point. After a specified time the recovery mark is read and data reported as a percent recovery. There are a number of variations of this test.

4.3.9 Toughness and Tenacity

The toughness and tenacity test uses a tensile pull machine similar to that used in the tensile strength test. The force necessary to pull the sample is measured. However, the method of reporting the results differs for this test. The area under the stress/strain curve generated by this test is measured in two different ways and is reported as toughness and tenacity. Great care must be taken with the interpretation of the data generated by this test. Major variations in test results between two or more laboratories are common because of the difficulty of reading the area under the curve.