Debbie,
Good afternoon, please save this to the SDMS file for Capital City Plume.
Thank you,
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----- Forwarded by Scott Miller/R4/USEPA/US on 01/18/2012 12:12 PM -----

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To:	Scott Miller/R4/USEPA/US@EPA, James E Landmeyer <jlandmey@usgs.gov>
Date:	01/18/2012 12:04 PM
Subject:	Fw: Solubility Test

Resending ... per Scott's request.
Athena

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Date:	09/27/2011 10:25 AM
Subject:	Solubility Test

Scott and Jim,

http://nersp.nerdc.ufl.edu/~tia/Bituminous-Materials.pdf

See section on Solubility Test.

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BITUMINOUS MATERIALS

Mang Tia, Professor of Civil and Coastal Engineering, University of Florida

Introduction

The term *bituminous materials* is generally used to denote substances in which bitumen is present or from which it can be derived [Goetz and Wood, 1960]. *Bitumen* is defined as an amorphous, black or dark-colored, (solid, semi-solid, or viscous) cementitious substance, composed principally of high molecular weight hydrocarbons, and soluble in carbon disulfide. For civil engineering applications, bituminous materials include primarily *aphalts* and *tars*. Asphalts may occur in nature (natural asphalts) or may be obtained from petroleum processing (petroleum asphalts). Tars do not occur in nature and are obtained as condensates in the processing of coal, petroleum, oil-shale, wood or other organic materials. *Pitch* is formed when a tar is partially distilled so that the volatile constituents have evaporated off from it. *Bituminous mixtures* are generally used to denote the combinations of bituminous materials (as binders), aggregates and additives.

This chapter presents the basic principles and practices of the usage of bituminous materials in pavement construction. In recent years, the use of tars in highway construction has been very limited due to the concern with the possible emission of hazardous flumes when tars are heated. Thus, this chapter deals primarily with asphalts.

Types of Bituminous Materials Used in Pavement Construction

Asphalt cement is an asphalt which has been specially refined as to quality and consistency for direct use in the construction of asphalt pavements. An asphalt cement has to be heated to an appropriate high temperature in order to be fluid enough to be mixed and placed.

Cutback asphalt is a liquid asphalt which is a blend of asphalt and petroleum solvents (such as gasoline and kerosine). A cutback asphalt can be mixed and placed with little or no application of heat. After a cutback asphalt is applied and exposed to the atmosphere, the solvent will gradually evaporate, leaving the asphalt cement to perform its function as a binder.

Emulsified asphalt (or *asphalt emulsion*) is an emulsion of asphalt cement and water that contains a small amount of emulsifying agent. In a normal emulsified asphalt, the asphalt cement is in the form of minute globules in suspension in water. An emulsified asphalt can be mixed and applied without any application of heat. After an asphalt emulsion is applied, sufficient time is required for the emulsion to break and the water to evaporate to leave the asphalt cement to perform its function as a binder. In an *inverted emulsified asphalt*, minute globules of water are in suspension in a liquid asphalt, which is usually a cutback asphalt. Inverted asphalt emulsions are seldom used in pavement applications.

Conventional Tests on Asphalt Cements and Their Significance

In this section, the purpose and significance of the commonly-used tests on asphalt cements are described. Readers may refer to the appropriate standard test methods for detailed description of the test procedures.

Penetration Test

The penetration test is one of the oldest and most commonly-used tests on asphalt cements or residues from distillation of asphalt cutbacks or emulsions. The standardized procedure for this test can be found in ASTM D5 [ASTM, 2001]. It is an empirical test which measures the consistency (hardness) of an asphalt at a specified test condition. In the standard test condition, a standard needle of a total load of 100 g is applied to the surface of an asphalt sample at a temperature of 25 °C for 5 seconds. The amount of penetration of the needle at the

end of 5 seconds is measured in units of 0.1 mm (or penetration unit). A softer asphalt will have a higher penetration, while a harder asphalt will have a lower penetration. Other test conditions which have been used include (1) 0 °C, 200 g, 60 sec., and (2) 46 °C, 50 g, 5 sec.

The penetration test can be used to designate grades of asphalt cement, and to measure changes in hardness due to age hardening or changes in temperature.

Flash Point Test

The flash point test determines the temperature to which an asphalt can be safely heated in the presence of an open flame. The test is performed by heating an asphalt sample in an open cup at a specified rate and determining the temperature at which a small flame passing over the surface of the cup will cause the vapors from the asphalt sample temporarily to ignite or flash. The commonly-used flash point test methods include (1) the Cleveland Open Cup (ASTM D92) and (2) Tag Open Cup (ASTM D1310). The Cleveland Open-Cup method is used on asphalt cements or asphalts with relatively higher flash points, while the Tag Open-Cup method is used on cutback asphalts or asphalts with flash points of less than 79 °C.

Minimum flash point requirements are included in the specifications for asphalt cements for safety reasons. Flash point tests can also be used to detect contaminating materials such as gasoline or kerosine in an asphalt cement. Contamination of an asphalt cement by such materials can be indicated by a substantial drop in flash point. When the flash point test is used to detect contaminating materials, the Pensky-Martens Closed Tester method (ASTM D93), which tends to give more indicative results, is normally used. In recent years, the flash point test results have been related to the hardening potential of asphalt. An asphalt with a high flash point is more likely to have a lower hardening potential in the field.

Solubility Test

Asphalt consists primarily of bitumens, which are high-molecular-weight hydrocarbons

soluble in carbon disulfide. The bitumen content of a bituminous material is measured by means of its solubility in carbon disulfide. In the standard test for bitumen content (ASTM D4), a small sample of about 2 g of the asphalt is dissolved in 100 ml of carbon disulfide and the solution is filtered through a filtering mat in a filtering crucible. The material retained on the filter is then dried and weighed, and used to calculate the bitumen content as a percentage of the weight of the original asphalt.

Due to the extreme flammability of carbon disulfide, solubility in trichloroethylene, rather than solubility in carbon disulfide, is usually used in asphalt cement specifications. The standard solubility test using trichloroethylene is designated as ASTM D 2042.

The solubility test is used to detect contamination in asphalt cement. Specifications for asphalt cements normally require a minimum solubility in trichloroethylene of 99.0 percent.

Unfortunately, trichloroethylene has been identified as a carcinogen and contributing to the depletion of the earth's ozone layer. The use of trichloroethylene will most likely be banned in the near future. There is a need to use a less hazardous and non-chlorinated solvent for this purpose. Results of several investigations have indicated that the solvent n-Propyl Bromide appears to be a feasible alternative to trichloroethylene for use in this application [Collins-Garcia et al, 2000].

Ductility Test

The ductility test (ASTM D113) measures the distance a standard asphalt sample will stretch without breaking under a standard testing condition (5 cm/min at 25 °C). It is generally considered that an asphalt with a very low ductility will have poor adhesive properties and thus poor performance in service. Specifications for asphalt cements normally contain requirements for minimum ductility.

Viscosity Tests

The viscosity test measures the viscosity of an asphalt. Both the viscosity test and the penetration test measure the consistency of an asphalt at some specified temperatures and are used to designate grades of asphalts. The advantage of using the viscosity test as compared with the penetration test is that the viscosity test measures a fundamental physical property rather than an empirical value.

Viscosity is defined as the ratio between the applied shear stress and induced shear rate of a fluid. The relationship between shear stress, shear rate and viscosity can be expressed as:

$$\text{Shear Rate = Shear Stress / Viscosity} \tag{1}$$

When shear rate is expressed in units of 1/sec. and shear stress in units of Pascal, viscosity will be in units of Pascal-seconds. One Pascal-second is equal to 10 Poises. The lower the viscosity of an asphalt, the faster the asphalt will flow under the same stress.

For a **Newtonian fluid**, the relationship between shear stress and shear rate is linear, and thus the viscosity is constant at different shear rates or shear stress. However, for a non-Newtonian fluid, the relationship between shear stress and shear rate is not linear, and thus the apparent viscosity will change as the shear rate or shear stress changes. Asphalts tend to behave as slightly non-Newtonian fluids, especially at lower temperatures. When different methods are used to measure the viscosity of an asphalt, the test results might be significantly different, since the different methods might be measuring the viscosity at different shear rates. It is thus very important to indicate the test method used when viscosity results are presented.

The most commonly-used viscosity test on asphalt cements is the Absolute Viscosity Test by Vacuum Capillary Viscometer (ASTM D2171). The standard test temperature is 60 °C. The absolute viscosity test measures the viscosity in units of Poise. The viscosity at 60 °C represents the viscosity of the asphalt at the maximum temperature a pavement is likely to experience in most parts of the United States.

When the viscosity of an asphalt at a higher temperature (such as 135 °C) is to be

determined, the most commonly-used test is the Kinematic Viscosity Test (ASTM D2170),

which measures the kinematic viscosity in units of Stokes or centi-Stokes. Kinematic viscosity

is defined as:

$$\text{Kinematic Viscosity} = \text{Viscosity} / \text{Density} \tag{2}$$

When viscosity is in units of Poise and density in units of g/cm^3, the kinematic viscosity

will be in units of Stokes. To convert from kinematic viscosity (in units of Stokes) to absolute

viscosity (in units of Poises), one simply multiplies the number of Stokes by the density in units

of g/cm^3. However, due to the fact that an asphalt might be non-Newtonian and that the

kinematic viscosity test and the absolute viscosity test are run at different shear rates, conversion

by this method will not produce accurate results and can only serve as a rough estimation. The

standard temperature for the kinematic test on asphalt cement is 135 °C. The viscosity at 135 °C

approximately represents the viscosity of the asphalt during mixing and placement of a hot mix.

Thin Film Oven and Rolling Thin Film Oven Tests

When an asphalt cement is used in the production of asphalt concrete, it has to be heated

to an elevated temperature and mixed with a heated aggregate. The hot asphalt mixture is then

hauled to the job site, placed and compacted. By the time the compacted asphalt concrete cools

down to the normal pavement temperature, significant hardening of the asphalt binder has

already taken place. The properties of the asphalt in service are significantly different from

those of the original asphalt.

Since the performance of the asphalt concrete in service depends on the properties of the

hardened asphalt binder in service rather than the properties of the original asphalt, the properties

of the hardened asphalt in service need to be determined and controlled.

The Thin Film Oven Test (TFOT) procedure (ASTM D1754) was developed to simulate

the effects of heating in a hot-mix plant operation on an asphalt cement. In the standard TFOT

procedure, the asphalt cement sample is poured into a flat-bottomed pan to a depth of about 1/8

inch (3.2 mm). The pan with the asphalt sample in it is then placed on a rotating shelf in an oven

and kept at a temperature of 163 °C for five hours. The properties of the asphalt before and after

the TFOT procedure are measured to determine the change in properties that might be expected

after a hot-mix plant operation.

The Rolling Thin Film Oven Test (RTFOT) procedure (ASTM D2872) was developed

for the same purpose as the TFOT and designed to produce essentially the same effect as the

TFOT procedure on asphalt cement. The advantages of the RTFOT over the TFOT are that (1) a

larger number of samples can be tested at the same time, and (2) less time is required to perform

the test. In the standard RTFOT procedure, the asphalt cement sample is placed in a

specially-designed bottle, which is then placed on its side on a rotating shelf, in an oven kept at

163 °C, and rolled continuously for 85 minutes. Once during each rotation, the opening of the

bottle passes an air jet, which provides fresh air to the asphalt in the bottle for increased

oxidation rate.

While the RTFOT and TFOT have generally worked well on pure asphalts, problems

were encountered when modified asphalts were used. Asphalts modified with crumb rubber and

SBR tended to spill out from the RTFOT bottles during the RTFOT process. When TFOT was

used on these modified binders, a thin skin tended to form on the surface of the modified asphalt,

which reduced the homogeneity and the aging of the samples.

A feasible alternative to the RTFOT and TFOT for use on modified asphalts appears to

be the modified rotavapor aging procedure [Sirin et al, 1998]. The rotavapor apparatus, which

was originally used for recovery of asphalt from solution (ASTM D5404), was modified to work

as an aging device for asphalt.   The binder to be aged is placed in a rotating flask, which is

immersed in a temperature-controlled oil bath. An air pump is used to provide a controlled air flow to the flask. Different aging effects can be produced by using different combinations of process temperature, process duration and sample size. Using a process temperature of 163 °C, process duration of 165 minutes and a sample size of 200 g has been found to produce aging severity similar to that of the RTFOT.

Ring & Ball Softening Point Test

The ring and ball softening point test (ASTM D36) measures the temperature at which an asphalt reaches a certain softness. When an asphalt is at its softening point temperature, it has approximately a penetration of 800 or an absolute viscosity of 13,000 poises. This conversion is only approximate and can vary from one asphalt to another, due to the non-Newtonian nature of asphalts and the different shear rates used by these different methods.

The softening point temperature can be used along with the penetration to determine the temperature susceptibility of an asphalt. Temperature susceptibility of an asphalt is often expressed as:

$$M = [\log(p_2) - \log(p_1)]/(t_2 - t_1) \tag{3}$$

where M = temperature susceptibility

t_1, t_2 = temperatures in °C

p_1 = penetration at t1

p_2 = penetration at t2

Since an asphalt has approximately a penetration of 800 at the softening point temperature, the softening point temperature can be used along with the penetration at 25 °C to determine the temperature susceptibility as:

$$M = [\log(\text{pen at 25 °C}) - \log(800)]/(25 - \text{S.P. Temp.}) \tag{4}$$

The M computed in this manner can then be used to compute a Penetration Index (PI) as

follows:

$$PI = (20 - 500 M) / (1 + 50 M) \tag{5}$$

The Penetration Index is an indicator of the temperature susceptibility of the asphalt. A high PI indicates low temperature susceptibility. Normal asphalt cements have a PI between -2 and +2. Asphalt cements with a PI of more than +2 are of low temperature susceptibility, while those with a PI of less than -2 are of excessively high temperature susceptibility.

Conventional Methods of Grading and Specifications of Asphalt Cements

There are three conventional methods of grading asphalt cements. These three methods are (1) grading by penetration at 25 °C, (2) grading by absolute viscosity at 60 °C, and (3) grading by absolute viscosity of aged asphalt residue after the rolling thin film oven test (RTFOT) procedure. These three methods of grading and the associated ASTM specifications of asphalt cements are presented and discussed in this section.

The method of grading of asphalt cements by standard penetration at 25 °C is the first systematic method developed and is still used by a few highway agencies in the world. The standard grades by this method include 40/50, 60/70, 85/100, 120/150 and 200/300 asphalts, which have penetrations of 40 to 50, 60 to 70, 85 to 100, 120 to 150, and 200 to 300, respectively. The Asphalt Institute recommends the use of a 120/150 or 85/100 pen. asphalt in the asphalt concrete for cold climatic condition with a mean annual temperature of 7 °C or lower. For warm climatic condition with a mean air temperature between 7 and 24 °C, a 85/100 or 60/70 pen. asphalt is recommended. For hot climatic condition with a mean annual air temperature of 24 °C or greater, the use of a 40/50 or 60/70 pen. asphalt is recommended [Asphalt Institute, 1991].

ASTM D946 [ASTM, 2001] provides a specification for penetration-graded asphalt

cements. Table 1 shows the specification for 60/70 and 85/100 pen asphalts as examples.

According to this specification, the only requirement on the consistency of the asphalt cements is

the penetration at 25 °C. There is no requirement on the consistency at either a higher or lower

temperature, and thus no requirement on the temperature susceptibility of the asphalt cements.

Two asphalts may be of the same penetration grade and yet have substantially different

viscosities at 60 °C. This problem is illustrated in Figure 1. Thus, it is clear that specifying the

penetration grade alone will not ensure that the asphalt used will have the appropriate viscosities

at the expected service temperatures. Other requirements in the specification are (1) minimum

flash point temperature, (2) minimum ductility at 25 °C, (3) minimum solubility in

trichloroethylene, and (4) penetration and ductility at 25 °C of the asphalt after aging by the

TFOT procedure.

Since penetration is an empirical test, grading by penetration was thought to be

unscientific. Considerable efforts were made in the 1960s to grade asphalts using fundamental

units. Early attempts were made to grade asphalts by viscosity at 25 °C and 20 °C. However,

problems were encountered in measuring viscosity at such low temperatures. With some

reluctance, the temperature for grading asphalt by viscosity was moved to 60 °C, which

represents approximately the highest temperature pavements may experience in most parts of the

United States. When an asphalt is graded by this system, it is designated as AC followed by a

number which represents its absolute viscosity at 60 °C in units of 100 poises. For example, an

AC-20 would have an absolute viscosity of around 2,000 poises at 60 °C. An AC-20 roughly

corresponds to a 60/70 pen. asphalt. However, due to the possible effects of different

temperature susceptibility and non-Newtonian behavior, the conversion from a viscosity grade to

a penetration grade may be different for different asphalts. Figure 2 shows the effects of

different temperature susceptibility on the viscosity variation of two asphalts which have the

same viscosity grade. In an effort to control this variation, the requirements for a minimum penetration at 25 °C and a minimum viscosity at 135 °C were added to the specification.

ASTM D3381 [ASTM, 2001] provides two different specifications for asphalt cements graded by absolute viscosity of the original asphalt at 60 °C. Table.2 show the requirements for AC-10 and AC-20 grade asphalts in the two specifications as examples. The main difference between these two specifications is that one of them requires a lower temperature susceptibility than the other. Limits on temperature susceptibility are specified through a minimum required penetration at 25 °C and a minimum required kinematic viscosity at 135 °C. The other requirements are similar to those in the specification of penetration-graded asphalts. The other requirements are (1) minimum flash point temperature, (2) minimum ductility at 25 °C, (3) minimum solubility in trichloroethylene, and (4) required properties of the asphalt after aging by the TFOT procedure (by means of maximum viscosity at 60 °C and ductility at 25 °C).

The third asphalt grading system is to grade asphalts according to their viscosity when placed on the road (after aging due to the heating and mixing process). This grading system has been adopted by several western states in the U.S. Grading is to be based on the absolute viscosity at 60 °C of the asphalt residue after the Rolling Thin Film Oven Test (RTFOT) procedure, which simulates the effects of the hot-mix plant operation. An asphalt graded by this system is designated as AR followed by a number which represents the viscosity of the aged residue at 60 °C in units of poises. For example, an AR-6000 would have an aged residue with an absolute viscosity of around 6000 poises. An AR-6000 would roughly correspond to an AC-20 or a 60/70 pen. asphalt. However, it should be recognized that the conversion from an AR grade to an AC grade depends on the hardening characteristics of the asphalt.

ASTM D3381 [ASTM, 2001] provides a specification for asphalt cements graded by viscosity of aged residue after the RTFOT process. Table 3 shows the specification for AR-4000

and AR-8000 grade asphalts as examples. According to this specification, temperature susceptibility is specified through requiring a minimum penetration at 25 °C and a minimum kinematic viscosity at 135 °C of the residue after the RTFOT. Similar to the requirements in the specifications for the other two grading systems, there are requirements on (1) ductility at 25 °C of the aged residue, (2) minimum flash point of the original asphalt, and (3) minimum solubility in trichloroethylene of the original asphalt. Another requirement in this specification is a minimum percent of retained penetration after the RTFOT, which can serve as a check on the composition and aging characteristics of the asphalt.

Superpave Binder Tests

The Strategic Highway Research Program (SHRP) conducted a $50 million research effort from October 1987 through March 1993 to develop performance-based test methods and specifications for asphalts and asphalt mixtures. The resulting product is a new system called Superpave (SUperior PERforming asphalt PAVEments), which includes a binder specification and an asphalt mixture design method. The Superpave binder tests and specifications have been standardized by the American Association of State Highway and Transportation Officials (AASHTO). The significance of the Superpave binder tests are described in this section. The detailed procedures can be found in the AASHTO publications for these tests [AASHTO, 2004].

Pressure Aging Vessel

The Superpave Pressure Aging Vessel (PAV) procedure is used for simulation of long-term aging of asphalt binders in service. According to the method, the asphalt samples are first aged in the standard RTFOT. Pans containing 50 grams of RTFOT residue are then placed in the PAV, which is pressurized with air at 2.1 ± 0.1 MPa, and aged for 20 hours. As many as 10 pans can be placed in the PAV. The proposed range of PAV temperature to be used is between 90

and 110 °C. The PAV temperature to be used will depend on the climatic condition of the region where the binders will be used. A higher PAV temperature could be used for a warmer climatic condition, while a lower temperature could be used for a colder climatic condition.

Dynamic Shear Rheometer Test

The dynamic shear rheometer test measures the viscoelastic properties of an asphalt binder by testing it in an oscillatory mode. The general method had been used by researchers long before the SHRP researchers adopted and standardized the method for the purpose of asphalt specification. Typically, in a dynamic shear rheometer test, a sample of asphalt binder is placed between two parallel steel plates. The top plate is oscillated by a precision motor with a controlled angular velocity, w, while the bottom plate remains fixed. From the measured torque and angle of rotation, the shear stress and shear strain can be calculated. The oscillatory strain, γ, can be expressed as:

$$\gamma = \gamma_0 \sin wt \tag{6}$$

where: γ_0 = peak shear strain

w = angular velocity in radian/second

The shear stress, τ, can be expressed as:

$$\tau = \tau_0 \sin (wt + \delta) \tag{7}$$

where: τ_0 = peak shear stress

δ = phase shift angle

The following parameter is computed from the test data:

$$\text{Complex Shear Modulus, } G^* = \tau_0 / \gamma_0 \tag{8}$$

SHRP standardized the dynamic shear rheometer test for use in measuring the asphalt properties at high and intermediate service temperatures for specification purposes. In the standardized test method, the oscillation speed is specified to be 10 radians/second. The

amplitude of shear strain to be used depends on the stiffness of the binder, and varies from 1%

for hard materials tested at low temperatures to 13% for relatively softer materials tested at high

temperatures. There are two standard sample sizes. For relatively softer materials, a sample

thickness (gap) of 1 mm and a sample diameter (spindle diameter) of 25 mm are to be used. For

harder materials, a sample thickness of 2 mm and a sample diameter of 8 mm are to be used.

The two values to be measured from each test are the complex shear modulus, G^*, and the phase

angle, δ. These two test values are then used to compute $G^*/\sin \delta$ and $G^*\sin \delta$. In the Superpave

asphalt specification, permanent deformation is controlled by requiring the $G^*/\sin \delta$ of the binder

at the highest anticipated pavement temperature to be greater than 1.0 kPa before aging and 2.2

kPa after the RTFOT process. Fatigue cracking is controlled by requiring that the binder after

PAV aging should have a $G^*\sin \delta$ value of less than 5000 kPa at a specified intermediate

pavement temperature.

Bending Beam Rheometer Test

The bending beam rheometer test was used to measure the stiffness of asphalts at low

service temperatures. The standard asphalt test specimen is a rectangular prism with a width of

12.5 mm, a height of 6.25 mm and a length of 125 mm. The test specimen is to be submerged in

a temperature-controlled fluid bath and to be simply supported with a distance between supports

of 102 mm. For specification testing, the test samples are to be fabricated from PAV-aged

asphalt binders, which simulate the field-aged binders. In the standard testing procedure, after

the beam sample has been properly pre-conditioned, a vertical load of 100 gram-force is applied

to the middle of the beam for a total of 240 seconds. The deflection of the beam at the point of

load is recorded during this period, and used to compute for the creep stiffness of the asphalt by

the following equation:

$$S(t) = PL^3/4bh^3\delta(t) \qquad\qquad\qquad (9)$$

where: S(t) = creep stiffness at time t

P = applied load, 100 g

L = distance between beam supports, 102 mm

b = beam width, 12.5 mm

h = beam height, 6.25 mm

δ(t) = deflection at time t

The above equation is similar to the equation that relates the deflection at the center of the beam to the elastic modulus of an elastic beam according to the classical beam theory. The instantaneous deflection in the original equation is replaced by the time dependent deflection δ(t), while the elastic modulus in the original equation is replaced by the time dependent creep stiffness S(t).

For Superpave binder specification purpose, the bending beam rheometer test is to be run at 10 °C above the expected minimum pavement temperature, T_{min}. SHRP researchers [Anderson & Kennedy, 1993] claimed that the stiffness of an asphalt after 60 seconds at T_{min} + 10 °C is approximately equal to its stiffness at T_{min} after 2 hours loading time, which is related to low-temperature cracking potential. The Superpave binder specification as stated in AASHTO Designation M320-03 [AASHTO, 2004] requires the stiffness at the test temperature after 60 seconds to be less than 300 MPa to control low-temperature cracking.

The second parameter obtained from the bending beam rheometer test result is the m-value. The m-value is the slope of the log stiffness versus log time curve at a specified time. A higher m-value would mean that the asphalt would creep at a faster rate to reduce the thermal stress and would be more desirable to reduce low-temperature cracking. The Superpave binder specification as stated in AASHTO M320-03 requires the m-value at 60 seconds to be greater than or equal to 0.30.

Direct Tension Test

The Superpave direct tension test measures the stress-strain characteristics of an asphalt binder in direct tension at low temperature. In this test, a small "dog bone" shaped asphalt specimen is pulled at a constant rate of 1 mm/min until it breaks. The amount of elongation at failure is used to compute the failure strain. The maximum tensile load taken by the specimen is used to compute the failure stress. The test specimen is 30 mm long and has a cross section of 6 mm by 6 mm at the middle portion. For Superpave binder specification purpose, the direct tension test is to be run on PAV-aged binders at the same test temperature as for the bending beam rheometer test, which is run at 10 °C above the minimum expected pavement temperature. According to the Superpave binder specification as stated in AASHTO Designation M320-03, the failure strain at this condition should not be less than 1% in order to control low temperature cracking.

Brookfield Rotational Viscometer Test

The Superpave binder specification uses the Brookfield rotational viscometer test as specified by ASTM D4402 for use in measuring the viscosity of binders at elevated temperatures to ensure that the binders are sufficiently fluid when being pumped and mixed at the hot mix plants. In the Brookfield rotational viscometer test, the test binder sample is held in a temperature-controlled cylindrical sample chamber, and a cylindrical spindle, which is submerged in the sample, is rotated at a specified constant speed. The torque that is required to maintain the constant rotational speed is measured and used to calculate the shear stress according to the dimensions of the sample chamber and spindle. Similarly, the rotational speed is used to calculate the shear rate of the test. Viscosity is then calculated by dividing the computed shear stress by the computed shear rate.

As compared with the capillary tube viscometers, the rotational viscometer provides larger clearances between the components. Therefore, it can be used to test modified asphalts containing larger particles, which could plug up a capillary viscometer tube. Another advantage of the rotational viscometer is that the shear stress versus shear rate characteristics of a test binder can be characterized over a wide range of stress or strain levels.

For Superpave binder specification purpose, the rotational viscosity test is to be run on the original binder at 135 °C. The maximum allowable viscosity at this condition is 3 Pa-s.

Superpave Binder Specification

The Superpave performance graded asphalt specification (AASHTO Designation M320-03) uses grading designations which correspond to the maximum and minimum pavement

temperatures of the specified region. The designation starts with "PG", and is followed by the

maximum and the minimum anticipated service temperature in °C. For example, A "PG-64-22"

grade asphalt is intended for use in a region where the maximum pavement temperature (based

on average 7-day maximum) is 64 °C and the minimum pavement temperature is -22 °C. A

"PG-52-46" grade asphalt is for use where the maximum pavement temperature is 52 °C and the

minimum pavement temperature is -46 °C.

Table 4 shows the Superpave specification for three different performance grades of

asphalts (PG-52-16, PG-52-46 & PG-64-22) as examples. The specified properties are constant

for all grades, but the temperatures at which these properties must be achieved vary according to

the climate in which the binder is to be used. It is possible that an asphalt can meet the

requirements for several different grades.

All grades are required to have a flash point temperature of at least 230 °C for safety

purpose, and to have a viscosity of no greater than 3 Pa-s at 135 °C to ensure proper workability

during mixing and placement.

Dynamic shear rheometer tests are to be run on the original and RTFOT-aged binders at

the maximum pavement design temperature. The minimum required values of $G^*/\sin\delta$ at this

temperature are 1.0 kPa and 2.2 kPa for the original and RTFOT-aged binders, respectively.

These requirements are intended to control pavement **rutting**.

Dynamic shear rheometer tests are also to be run on PAV-aged binders at an intermediate

temperature, which is equal to 4 °C plus the mean of the maximum and minimum pavement

design temperatures. For example, for a PG-52-46 grade, the intermediate temperature is 7 °C.

The maximum allowable value of $G^*\sin\delta$ at this condition is 5000 kPa. This requirement is

intended to control pavement fatigue cracking.

Bending beam rheometer tests and direct tension tests are to be run on PAV-aged binders at a temperature which is 10 °C above the minimum pavement design temperature. For example, for a PG-52-46, the test temperature is -36 °C. At a loading time of 60 seconds, the stiffness is required to be no greater than 300 MPa, and the m-value is required to be no less than 0.3. The failure strain from the direct tension test is required to be at least 1%. However, the direct tension test criterion is applicable only if an asphalt does not meet the bending beam rheometer stiffness requirement and has a stiffness between 300 MPa and 600 MPa .

Effects of Properties of Asphalt Binders on the Performance of Asphalt Pavements

Effects of Viscoelastic Properties of Asphalt

When an asphalt concrete surface is cooled in winter time, stresses are induced in the asphalt concrete. These stresses can be relieved by the flowing of the asphalt binder within the asphalt mixture. However, if the viscosity of the asphalt binder is too high at this low temperature, the flow of the asphalt binder may not be fast enough to relieve the high induced stresses. Consequently, low-temperature cracking may occur. The viscosity of asphalt at which low-temperature cracking would occur is dependent on the cooling rate of the pavement as well as the characteristics of the asphalt concrete. However, as a rough prediction of low-temperature cracking, a limiting viscosity of 2×10^{10} poises could be used [Davis, 1987]. If the viscosity of the asphalt binder at the lowest anticipated temperature is kept lower than this limiting value, low-temperature cracking would be unlikely to occur.

Another critical condition of an asphalt concrete is at the highest pavement temperature,

at which the asphalt mixture is the weakest and most susceptible to plastic flow when stressed. When the other factors are kept constant, an increase in the viscosity of the asphalt binder will increase the shear strength and subsequently the resistance to plastic flow of the asphalt concrete. With respect to resistance to plastic flow of the asphalt concrete, it is preferable to have a high asphalt viscosity at the highest anticipated pavement temperature. Results by Goodrich [1988] indicate that a low tan δ value of the binder (as obtained from the dynamic rheometer test) tends to correlate with a low creep compliance of the asphalt mixture, which indicates high rutting resistance. Thus, a low tan δ value of the binder is desirable to reduce rutting potential.

The effectiveness of the mixing of asphalt cement and aggregate, and the effectiveness of the placement and compaction of the hot asphalt mix are affected greatly by the viscosity of the asphalt. The Asphalt Institute recommends that the mixing of asphalt cement and aggregate should be done at a temperature where the viscosity of the asphalt is 1.7 ± 0.2 poises. Compaction should be performed at a temperature where the viscosity of the asphalt cement is 2.8 ± 0.3 poises [Epps et al, 1983]. These viscosity ranges are only offered as guidelines. The actual optimum mixing and compaction temperatures will depend on the characteristics of the mixture as well as the construction environment.

In the selection of a suitable asphalt cement to be used in a certain asphalt paving project, the main concerns are (1) whether the viscosity of the asphalt at the lowest anticipated service temperature would not be low enough to avoid low-temperature cracking of the asphalt concrete, (2) whether the viscosity of the asphalt at the highest anticipated temperature would be high enough to resist rutting, and (3) whether the required temperatures for proper mixing and placement would not be too high.

Effects of Hardening Characteristics of Asphalt

An important factor that affects the durability of an asphalt concrete is the rate of hardening of the asphalt binder. The causes of hardening of asphalt have been attributed to oxidation, loss of volatile oils, and polymerization (changes in structure). Among all these possible factors, oxidation is generally considered to be the prime cause of asphalt hardening.

The most severe hardening of asphalt occurs during the mixing process. The viscosity of the asphalt binder immediately after the asphalt concrete is placed on the road is usually 2 to 4 times the viscosity of the original asphalt cement. The asphalt binder continues to harden through service; its viscosity could reach as high as 10 to 20 times the viscosity of the original asphalt cement. The rate of asphalt hardening is dependent on asphalt composition, mixing temperature, air voids content, and climatic conditions. It usually increases with increased mixing temperature, increased air voids content in the asphalt mix, and increased service (air) temperature.

Excessive hardening of the asphalt binder will cause the asphalt concrete to be too brittle and low-temperature cracking to occur. It may also cause the asphalt binder to partially lose its adhesion and cohesion, and subsequently it may cause raveling (progressive disintegration of pavement material and separation of aggregates from it) in the asphalt concrete.

A certain amount of hardening of the asphalt binder during the mixing process is usually expected and designed for. If an asphalt binder has not hardened sufficiently during the mixing process (due to low mixing temperature or the peculiar nature of the asphalt), the asphalt binder may be too soft at placement. This may cause the asphalt mix to be difficult to compact (tender mix) and to have a low resistance to rutting in service. If the tenderness of an asphalt concrete disappears within a few weeks after construction, the problem is most likely caused by slow setting asphalt. This type of asphalt requires an excessive amount of time to "set up" after they

are heated up and returned to normal ambient temperature. Asphalts containing less than 10 percent asphaltenes appear to have a greater probability of producing slow-setting asphalt mixtures. Asphaltenes are the high molecular weight fraction of asphalt which can be separated from the other asphalt fractions by dissolving an asphalt in a specified solvent (such as n-heptane as used in the ASTM D4124 Methods for Separation of Asphalt into Four Fractions) [ASTM, 2001]. The asphaltenes, which are insoluble in the solvent, would be precipitated out in this method.

Types and Grades of Cutback Asphalts

Cutback asphalts are classified into three main types on the basis of the relative speed of evaporation of the solvents in them. A Rapid-Curing (RC) cutback asphalt is composed of an asphalt cement and a solvent of a volatility similar to that of naphtha or gasoline, which evaporates at a fast speed. A Medium-Curing (MC) cutback asphalt contains a solvent of a volatility similar to that of kerosine, which evaporates at a medium speed. A Slow-Curing (SC) cutback asphalt contains an oil of relatively low volatility.

Within each type, cutback asphalts are graded by kinematic viscosity at 60 °C. It is designated by the type followed by the lower limit of the kinematic viscosity at 60 °C in units of centi-stokes (cSt). The upper limit for the viscosity is twice its lower limit. For example, an "RC-70" designates a rapid-curing cutback asphalt with a kinematic viscosity at 60 °C ranging between 70 and 140 cSt, while an "SC-800" designates a slow-curing cutback asphalt with a viscosity ranging between 800 and 1600 cSt. The standard specifications for SC, MC and RC cutback asphalts can be found in ASTM Designation D2026, D2027 and D2028, respectively

[ASTM, 2001].

The standard practice for selection of cutback asphalts for pavement construction and maintenance can be found in ASTM Designation D2399 [ASTM, 2001].

Types and Grades of Emulsified Asphalts

Emulsified asphalts (or asphalt emulsions) are divided into three major kinds, namely anionic, cationic and nonionic, on the basis of the electrical charges of the asphalt particles in the emulsion. An anionic asphalt emulsion has negatively-charged asphalt particles, and is usually more suitable for use with calcareous aggregates, which tend to have positive surface charges. A cationic asphalt emulsion has positively charged asphalt particles, and is usually more suitable for use with siliceous aggregates, which tend to have negative surface charges. A nonionic asphalt emulsion contains asphalt particles that are electrically neutral. Nonionic asphalt emulsions are not used in pavement applications.

Asphalt emulsions are further classified into three main types on the basis of how quickly the suspended asphalt particles revert to asphalt cement. The three types are Rapid-setting (RS), Medium-Setting (MS) and Slow-Setting (SS). An RS emulsion is designed to demulsify (to break away from the emulsion form such that asphalt particles are no longer in suspension) upon contact with an aggregate, and thus has little or no ability to mix with an aggregate. It is best used in spraying applications where mixing is not required but fast setting is desirable. An MS emulsion is designed to have good mixing characteristics with coarse aggregates and to demulsify after proper mixing. It is suitable for applications where mixing with coarse aggregate is required. An SS emulsion is designed to be very stable in the emulsion form, and is suitable for use where good flowing characteristics are desired or where mixing with fine aggregates is

required. The three types of cationic asphalt emulsions are denoted as CRS, CMS and CSS.

The absence of the letter "C" in front of the emulsion type denotes an anionic type.

Two other standard types of anionic asphalt emulsions available are High-Float Rapid

Setting (HFRS) and High-Float Medium Setting (HFMS).   This type of asphalt emulsion

contains an asphalt cement which has a Bingham plastic characteristic (resistant to flow at low

stress level). This flow property of the asphalt permits a thicker film coating on an aggregate

without danger of runoff.

Within each type, asphalt emulsions are graded by the viscosity of the emulsion and the

hardness of the asphalt cement. The lower viscosity grade is designated by a number "1" and the

higher viscosity grade is designated by a number "2", which is placed after the emulsion type. A

letter "h" that follows the number "1" or "2" designates that a harder asphalt cement is used. For

example, an "RS-1" designates a rapid-setting anionic type with a relatively low viscosity. An

"HFMS-2h" designates a high-float medium setting anionic type having a relatively higher

viscosity and containing a hard base asphalt. A "CSS-1h" designates a slow-setting cationic type

having a relatively lower viscosity and containing a hard base asphalt. Standard specifications

for anionic and cationic emulsified asphalts can be found in ASTM Designations D977, and

D2397, respectively.

The standard practice for selection and use of emulsified asphalts in pavement

construction and maintenance can be found in ASTM Designation D3628 [ASTM, 2001].

References

American Association of State Highway and Transportation Officials 2004. *Standard*

Specifications for Transportation Materials and Methods of Sampling and Testing, AASHTO,

Washington, D.C.

American Society for Testing and Materials 2004. *Annual Book of ASTM Standard*, ASTM, Philadelphia, Pennsylvania.

Anderson, D.A. and Kennedy, T.W. 1993. Development of SHRP Binder Specification, *Proceedings of Association of Asphalt Paving Technologists*, Vol. 62, p. 481-528, AAPT, Minneapolis, Minnesota.

Asphalt Institue 1997. *Mix Design Methods for Asphalt Concrete and Other Hot-Mix Types*, Manual Series No. 2, Sixth Edition, The Asphalt Institute, Lexington, Kentucky.

Asphalt Institute 1991. *Thickness Design - Asphalt Pavements for Highways and Streets*, Manual Series No.1, The Asphalt Institute, Lexington, Kentucky.

Collins-Garcia, H., Tia, M., Roque, R. and Choubane, B. 2000, Evaluation of an Alternative Solvent for Extraction of Asphalt to Reduce Health and Environmental Hazards, *Transportation Research Record* 1712, pp. 79-85, Transportation Research Board, Washington, D.C.

Davis, R.L. 1987. Relationship Between the Rheological Properties of Asphalt and the Rheological Properties of Mixtures and Pavements, *Asphalt Rheology: Relationship to Mixture*, ASTM STP 941, O.E. Briscoe, Ed., pp.28-50, American Society for Testing and Materials, Philadelphia.

Epps, J.A., Button J.W. and Gallaway B.M. 1983. *Paving with Asphalt Cements Produced in the 1980's*, NCHRP Report 269, Transportation Research Board, Washington, D.C.

Goetz, W.H. and Wood, L.E. 1960. Bituminous Materials and Mixtures, *Highway Engineering Handbook*, Ed. K.B. Woods, Section 18, McGraw-Hill Book Company, New York.

Goodrich, J.L. 1988. Asphalt and Polymer Modified Asphalt Properties Related to the Performance of Asphalt Concrete Mixes, *Proc. of Association of Asphalt Paving Technologists*, Vol. 57, p. 116-175, AAPT, Minneapolis, Minnesota.

Sirin, O., Shih, C.T., Tia, M., Ruth, B.E., "Development of a Modified Rotavapor Apparatus and Method for Short-Term Aging of Modified Asphalts*," Transportation Research Record 1638*, 1998, pp.72-84, Washington, D.C.

Table 1 Requirements for 60/70 and 85/100 penetration asphalt cements

Test	Penetration Grade			
	60/70		85/100	
	Min	Max	Min	Max
Penetration at 25 °C, 0.1mm	60	70	85	100
Flash point (Cleveland open cup), °C	232	-	232	-
Ductility at 25 °C, cm	100	-	100	-
Solubility in trichloroethylene, %	99.0	-	99.0	-
Retained penetration after TFOT, %	52	-	47	-
Ductility at 25 °C after TFOT, cm	50	-	75	-

Source: ASTM 2004. ASTM D 946 Standard Specification for Penetration-Graded Asphalt Cement for Use in Pavement Construction, *Annual Book of ASTM Standards*, Volume 04.03, 2004, Philadelphia, Pennsylvania.

Table 2 Requirements for AC-10 and AC-20 asphalt cements

Test on Original Asphalt		Viscosity Grade			
		AC-10		AC-20	
		Spec. 1	Spec. 2	Spec. 1	Spec. 2
Absolute Viscosity at 60 °C, poises		1000±200	1000±200	2000±400	2000±400
Kinematic Viscosity at 135 °C, cSt		150	250	210	300
Penetration at 25 °C, 0.1mm		70	80	40	60
Flash point (Cleveland open cup), °C		219	219	232	232
Solubility in trichloroethylene, min., %		99.0	99.0	99.0	99.0
Tests on Residue from TFOT	Viscosity at 60 °C, max., poises	5000	5000	10000	10000
	Ductility at 25 °C, min., cm	50	75	20	50

Source: ASTM 2004. ASTM D 3381 Standard Specification for Viscosity-Graded Asphalt Cement for Use in Pavement Construction, *Annual Book of ASTM Standards*, Volume 04.03, 2004, Philadelphia, Pennsylvania.

Table 3 Requirements for AR-4000 and AR-8000 asphalt cements

Test on Residue from RTFOT		Viscosity Grade	
		AR-4000	AR-8000
Absolute Viscosity at 60 °C, poises		4000±1000	8000±2000
Kinematic Viscosity at 135 °C, min., cSt		275	400
Penetration at 25 °C, min., 0.1mm		25	20
% of original penetration, min.		45	50
Ductility at 25 °C, min., cm		75	75
Test on Original Asphalt	Flash point (Cleveland open cup), min., °C	227	232
	Solubility in trichloroethylene, min., %	99.0	99.0

Source: ASTM 2004. ASTM D 3381 Standard Specification for Viscosity-Graded Asphalt Cement for Use in Pavement Construction, *Annual Book of ASTM Standards*, Volume 04.03, 2004, Philadelphia, Pennsylvania.

Table 4 Examples of Superpave Performance Graded Binder Specification

Performance Grade	PG-52-16	PG-52-46	PG-64-22
Average 7-Day Maximum Pavement Design Temperature, °C	52	52	64
Minimum Pavement Design Temperature, °C	-16	-46	-22
Original Binder			
Flash Point Temperature, Minimum, °C	230		
Viscosity: Maximum, 3 Pa·s Test Temperature, °C	135		
Dynamic Shear @ 10 rad/s : $G^*/\sin\delta$, Minimum, 1.00 kPa Test Temperature, °C	52	52	64
Rolling Thin Film Oven Residue			
Mass Loss, Maximum, %	1.00		
Dynamic Shear @ 10 rad/s : $G^*/\sin\delta$, Minimum, 2.20 kPa Test Temperature, °C	52	52	64
Pressure Aging Vessel Residue			
PAV Aging Temperature, °C	90	100	100
Dynamic Shear @ 10 rad/s : $G^* \sin\delta$, Maximum, 5000 kPa Test Temperature, °C	22	7	25
Creep Stiffness @ 60 s : S, Maximum, 300 MPa m-value, Minimum, 0.30 Test Temperature, °C	-6	-36	-12
Direct Tension @ 1.0 mm/min : Failure Strain, Minimum, 1.0% Test Temperature, °C	-6	-36	-12

Source: AASHTO 2004. AASHTO Designation M320-03 Standard Specification for Performance Graded Asphalt Binder, *Specifications for Transportation Materials and Methods of Sampling and Testing*, Washington, D.C.
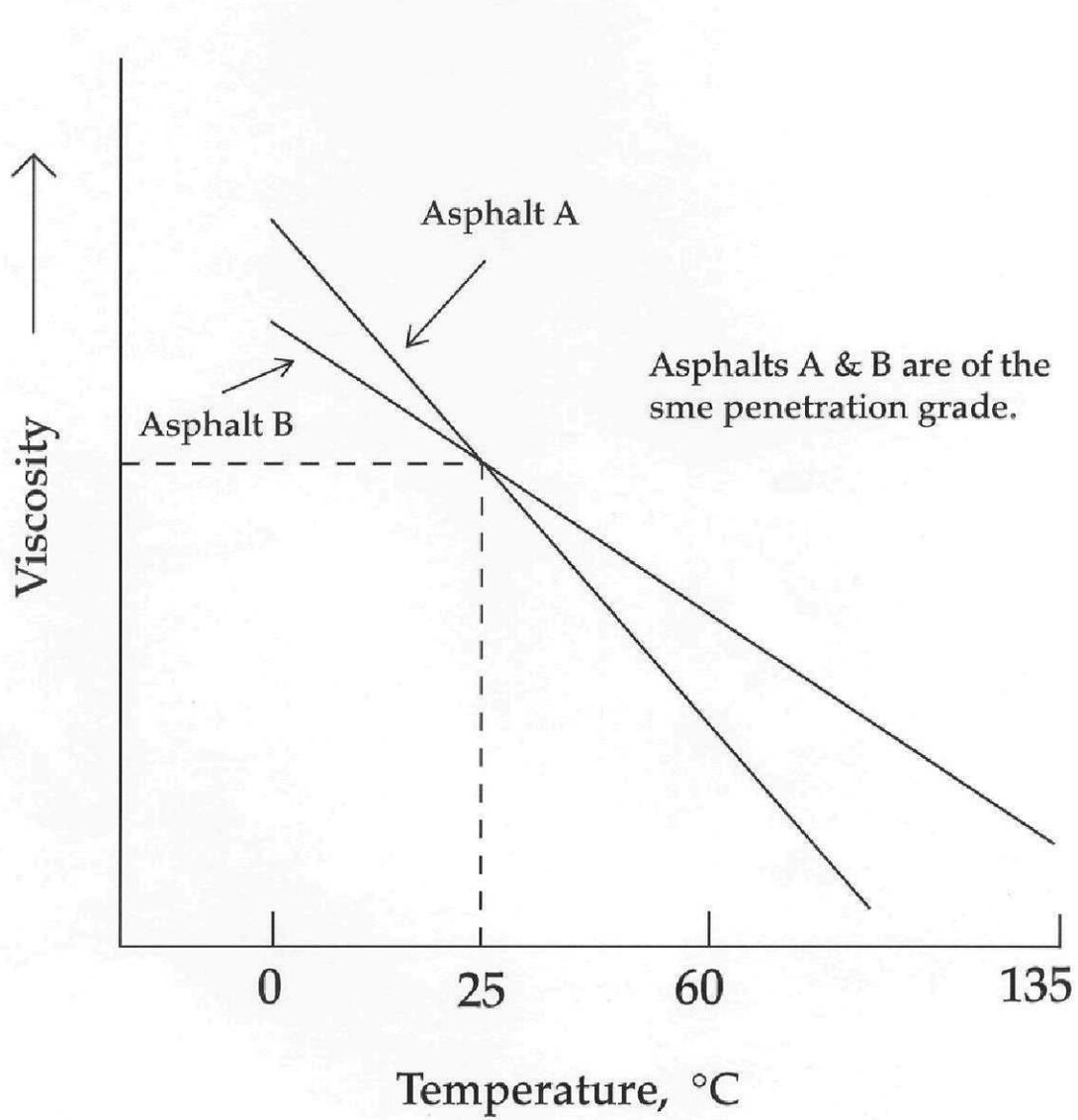
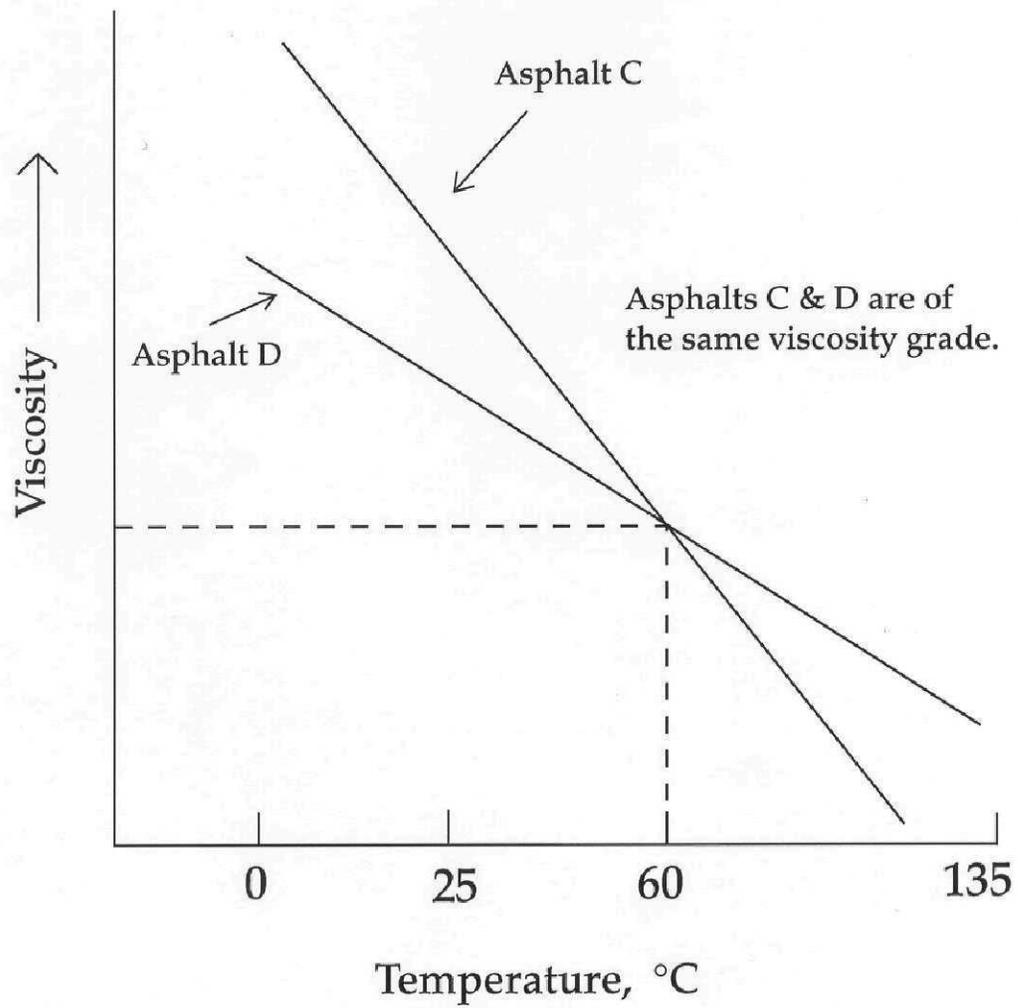Figure 1 Variation in viscosity of two penetration-graded asphalts at different temperatures

Figure 2 Variation in viscosity of two viscosity-graded asphalts at different temperatures