Durability of Bituminous Paving Mixtures

by

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Abstract

Bitumens are used as a binder in roadway pavements largely because they are relatively inexpensive and generally provide good adhesion and waterproofing characteristics. They are generally rather resistant to the detrimental effects of the environment and usually remain relatively pliant for many years. In other words, bitumens are relatively inexpensive binders that generally provide good durability (or longevity of service) in pavement mixtures.

Bitumens are no panacea, however. Many factors affect the durability of bitumens and, thus, bituminous mixtures. However, assuming that a pavement layer is constructed according to specifications (which attempt to account for durability), it is generally agreed that the two primary factors affecting the durability of bituminous paving mixtures are damage due to water and embrittlement of the bitumen due to age hardening.

Much effort has been afforded to the study of age hardening and water damage and much has been learned. However, the exact mechanisms of ageing and water damage in bituminous mixtures remain an enigma. This thesis attempts to provide an improved understanding of these mechanisms through a comprehensive literature review, development of performance tests to assess mixture durability and investigation of the rheological characteristics of bitumens aged and tested whilst in contact with mineral aggregate.

Key words: bitumen, bitumen chemistry, bituminous mixture, durability, oxidative ageing, age hardening, moisture damage, water sensitivity, rheology, rheological properties, viscoelastic properties, stiffness modulus, complex modulus, mineral aggregate

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1 Introduction

1.1 BACKGROUND

Bitumens have been used for millennia as adhesives, waterproofing agents and, in some cases, preservatives (1, 2). Early applications made use of "natural" bitumens obtained from surface seepages such as the lake of natural asphalt in Trinidad. Although natural asphalts are still used, most present-day applications make use of bitumens manufactured from crude oils.

Most bitumens are the product of distillation of crude oils, as is gasoline and other fuel oils. Some bitumens are products of solvent precipitation processes. Regardless of the process, however, the components of the crude oil with the highest molecular weight and chemical complexity become concentrated in bitumen. The four principal crude oil producing areas in the world include the United States, the Middle East, the Caribbean countries and Russia. Nearly 1,500 crude oils are produced worldwide (1). Crude oils from different sources are exceptionally diverse in their chemical make-up and, as a consequence, so are the bitumens produced from the oils. Bitumens are principally comprised of organic compounds consisting of primarily hydrogen and carbon, referred to as hydrocarbons, but most also contain other atomic particles (e.g., sulphur or oxygen) which significantly influence the chemical and physical properties of the bitumen.

Bitumens are used in a wide variety of applications—for fabricating clay pigeons and flower pots, for insulating paints and joint fillers, as mirror backing and coffin liners, to name just a few (3)—but the principal use is for building roads and, to a lesser extent, airfield pavements* which, together, account for approximately 85% of

*The term *pavement* is used throughout this thesis to mean a paved roadway surface, such as that found on a motorway, not the footway (sidewalk) adjacent to the roadway.

For a bituminous paving mixture to survive its design life (generally 10 to 20 years) it must provide, in addition to adequate skid resistance, ride quality and resistance to load-associated distresses, good durability characteristics. While the principal failure mechanisms of roads result from traffic loading (e.g., cracking, permanent deformation and reduction in skid resistance), adverse environmental effects can accelerate the deterioration process. Many factors affect the durability of bituminous mixtures. However, assuming that a pavement layer is constructed according to specifications (which attempt to account for durability), it is generally agreed that the two primary factors that affect the durability of bituminous paving mixtures are embrittlement of the bitumen due to age hardening and damage due to water.

Bitumens become stiffer (increase in viscosity) primarily due to oxidation which occurs rapidly during construction of bituminous mixtures but more slowly whilst the mixture is in service. This phenomenon is referred to as oxidative ageing or just ageing and, thus, bitumens and bituminous mixtures are said to age or age harden. Moderate ageing is generally expected and is usually acceptable, but significant ageing can result in embrittlement of the bitumen. This can significantly affect the adhesion characteristics of the bitumen and is usually manifested in reduced cracking resistance of the mixture. Thus, age hardening can lead to early failure of bituminous mixtures.

Water can degrade the structural integrity of a bituminous mixture through loss of adhesion between the bitumen and the aggregate and/or through loss of cohesion in the mixture. Both mechanisms generally result in a reduction of strength and/or stiffness of the mixture and, thus, its effectiveness to accommodate traffic-induced stresses and strains. Consequently, the water-damaged pavement layer is prone to stripping (i.e., physical separation of the bitumen from the aggregate) and permanent deformations. Thus, water damage can also lead to early failure.

Much effort has been afforded to the study of age hardening and water damage and much has been learned. However, the exact mechanisms of ageing and water damage in bituminous mixtures remain an enigma.

In conjunction with the development and evaluation of practical durability tests, evaluation of the ageing characteristics of bitumens in contact with mineral aggregate was carried out on a fundamental level. This involved development and use of a novel way to test bitumens in a dynamic shear rheometer and confirmed that the mineralogy of the surface with which bitumen comes into contact affects its physical (rheological) properties.

1.3 SCOPE

Prior to embarking on development of test methods for the durability of bituminous paving mixtures, a literature review was conducted to ascertain knowledge about what has been previously learned about durability as well as any tests developed to assess durability. The principal findings of this review are summarised in Chapter 2 of this thesis while the development and evaluation of test methods and practices are presented in Chapters 3 and 4. Chapter 5 presents the development and use of a novel experimental arrangement involving a dynamic shear rheometer to evaluate the rheological changes occurring to bitumens subjected to accelerated oven ageing and Chapter 6 provides a discussion of the contents of this thesis including significant conclusions and recommendations.

1.4 REFERENCES

1. Whiteoak, D., *The Shell Bitumen Handbook*, Surrey, UK, 1990.

2. Morgan, P. and Mulder, A., *The Shell Bitumen Industrial Handbook*, Surrey, UK, 1995.

3. "The Asphalt Handbook," *Manual Series No.4, (MS-4)*, The Asphalt Institute, Lexington, Kentucky, 1989.

4. Edited by Harrigan, E.T., Leahy, R.B. and Youtcheff, J.S., "The SUPERPAVE Mix Design System Manual of Specifications, Test Methods, and Practices," *SHRP-A-379*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1994.

5. "SUPERPAVE Performance Graded Asphalt Binder Specification and Testing," *Superpave Series No.1 (SP-1)*, The Asphalt Institute, Lexington, Kentucky, 1994.

pavement is in service. This hardening, referred to as ageing or age hardening, is manifested in the stiffening of the bituminous layer(s) which, to some degree, is beneficial (e.g., a moderate amount of hardening improves the load spreading capabilities of the pavement layer which may result in better resistance to permanent deformation, often referred to as rutting). However, if the hardening of the bitumen is excessive, the mixture can become brittle and crack resulting in partial or significant failure of the bound layer. That is, excessive hardening of the binder in a bituminous mixture generally results in a reduction of the pavements ability to support traffic-induced stresses and strains. Excessive hardening of bitumen can also result in decreased adhesion between the bitumen and aggregate (1), often resulting in loss of the material at the layer surface. The changes that occur in a bitumen (i.e., the reduction in its ability to flow under shear loading as a result of age hardening) can significantly influence the durability of bituminous mixtures.

Damage due to moisture can also significantly influence the durability of bituminous mixtures. It is generally agreed that moisture can degrade the structural integrity of bitumen-aggregate mixtures through loss of cohesion[2] or through failure of the adhesion (or bond) between the bitumen and the aggregate (2, 3). Reduction of cohesion in the bitumen-aggregate mixture results in a reduction of the strength and stiffness of the mixture and thus a reduction of the pavement's ability to support traffic-induced stresses and strains. Failure of the bond between the bitumen and the aggregate (i.e., physical separation of the binder from the aggregate, referred to as stripping) also results in a reduction of the pavement's ability to support traffic-induced stresses and strains. Stripping, which is often characterised by migration of the bitumen to the surface of the layer (i.e., flushing or bleeding), results in a reduction of cohesion in the lower portions of the stripped layer as well as instability in the upper portion of the layer due to excessive amounts of bitumen. Both mechanisms of water damage result in a weaker pavement layer and one which is prone to deform under the influence of traffic loading. In addition, stripping can

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[2]*Cohesion*, as used here, may be defined as the overall attraction by which particles of bodies stick together to make up a compatible mixture.

in the procedures they have developed and Terrel and Al-Swailmi include repeated loading to simulate the effects of traffic. For the purposes of this thesis the following definition will be used:

> *Durability as it applies to bituminous paving mixtures is defined as the ability of the materials comprising the mixture to resist the effects of water, ageing and temperature variations, in the context of a given amount of traffic loading, without significant deterioration for an extended period.*

2.1.3 Purpose

The purpose of the literature review is to provide a synopsis of selected literature regarding age hardening and water sensitivity of bituminous mixtures with emphasis on their relation to durability. The literature review serves three important functions: 1) it synthesises what has previously been learned about the factors which influence durability; 2) it synthesises the efforts (i.e., test methods) conducted to predict whether or not a particular bituminous mixture will be adversely affected by embrittlement due to ageing and/or damage due to moisture and 3) it provides information which will steer further research work. More specifically, the literature review provides the following:

1) a synopsis of the factors which affect durability and, in particular, the mechanisms and consequences of age hardening and water sensitivity;
2) a review of test methods which address durability;
3) a review of studies which have attempted to link laboratory testing methods to field performance and
4) significant conclusions arising from the literature review.

2.2 BITUMEN

The durability of bitumen and, therefore, bitumen-aggregate mixtures is largely determined by the physical properties of the bitumen which, in turn, are determined by its chemical composition. Thus, a review of the chemical composition of bitumen is warranted prior to discussing the factors which affect the durability of bituminous mixtures.

Table 2.1. Elemental Composition of Several Paving Grade Bitumens (13).

Bitumen	AAA-1	AAB-1	AAC-1	AAD-1	AAF-1	AAG-1	AAK-1	AAM-1
Grade	150/200	AC-10	AC-8	AR-4000	AC-20	AR-4000	AC-30	AC-20
Crude Oil Source	Lloydminister	Wyoming Sour	Red Water	California	West Texas Sour	California Valley	Boscan	West Texas Intermediate
Carbon, %	83.9	82.3	86.5	81.6	84.5	85.6	83.7	86.8
Hydrogen, %	10	10.6	11.3	10.8	10.4	10.5	10.2	11.2
Oxygen, %	0.6	0.8	0.9	0.9	1.1	1.1	0.8	0.5
Nitrogen, %	0.5	0.54	0.66	0.77	0.55	1.1	0.7	0.55
Sulphur, %	5.5	4.7	1.9	6.9	3.4	1.3	6.4	1.2
Vanadium, ppm	174	220	146	310	87	37	1480	58
Nickel, ppm	86	56	63	145	35	95	142	36
Iron, ppm	<1	16	—	13	100	48	24	255
Aromatic Carbon, %	28.1	31.9	24.7	23.7	32.8	28.3	31.9	24.7
Aromatic Hydrogen, %	7.68	7.12	6.41	6.81	8.66	7.27	6.83	6.51
Molecular Wt. (Toluene)	790	840	870	700	840	710	860	1300

reformed, but these may not necessarily result in the same chemical structure that was present before heating.

2.2.4 Fractional Composition of Bitumen

Because the number of molecules with different chemical structures is astronomically large, chemists have not seriously attempted to separate and identify all the different molecules in bitumen (11, 12). Instead, various techniques have been developed to separate bitumen into less complex and more homogenous fractions. An attempt will not be made to summarise the techniques as the literature abounds with descriptions of these techniques (e.g., 11, 12, 14). However, it is important to note that the techniques divide the bitumen into groups or generic fractions based on molecular size, chemical reactivity and/or polarity and that the different separation techniques lead to fractions having different characteristics (i.e., the fractions from one separation technique differ from those from another technique with regard to chemical and physical characteristics). The method developed by Corbett (15) has probably seen the widest use as a separation technique for research purposes. For this reason, as well as to avoid confusion, the subsequent discussion with regard to the fractional composition of bitumen is be based on this technique.

According to Corbett's fractionation technique (15), bitumen is separated into asphaltenes and maltenes. The asphaltenes are considered to be the most complex fraction containing the molecules with the highest polarity and tendency to interact and associate. Asphaltenes, primarily comprised of hydrocarbons and some heteroatoms, are brittle solids when isolated. It is generally believed that the asphaltenes are primarily responsible for bitumen viscosity (12, 16, 17). Whiteoak (5) states that, "The asphaltene content has a large effect on the rheological characteristics of a bitumen. Increasing the asphaltene content produces a harder bitumen with a lower penetration, higher softening point and consequently higher viscosity."

The asphaltenes, of high molecular weight, are dispersed or dissolved in a lower molecular weight oily medium referred to by most authors as maltenes. The maltenes

2.2.6 Microstructural Model of Bitumen

As part of the A-002A contract of the recently completed Strategic Highway Research Program (SHRP) in the United States, the research team[3] developed a conceptual model, referred to as the *microstructural model*, to relate the physical and chemical properties of bitumen. Development of the model was governed by the premise that the important performance-related physical properties of bitumens are related to bitumen composition (18). The following discussion provides a brief review of this model.

The microstructural model proposed by the A-002A research team suggests that bitumens consist of *microstructures* (comprised of polar, aromatic molecules that tend to form associations) dispersed in a bulk solvent moiety consisting of relatively nonpolar, aliphatic molecules (18, 19). The model postulates that many of the molecules comprising the dispersed phase are polyfunctional and capable of associating with one another through hydogen bonds, dipole interactions and π-π interactions to form primary microstructures. It further postulates that the primary microstructures associate to form, under the proper conditions, three-dimensional networks which can become disjoined, as alluded to earlier, by heat and/or shear stress. The model suggests that the physical properties of bitumens can best be described by the effectiveness with which the microstructures are dispersed by the solvent moiety, rather than being described by global chemical parameters such as elemental composition.

Efforts to validate the microstructural model required separation of bitumen into solvent and dispersed moieties. Separation, by ion exchange chromatography (IEC), of bitumens into polar components, comprised of strong and weak acids, strong and weak bases and amphoterics (which are compounds with both acidic and basic

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[3]The A-002A research team was comprised of Western Research Institute (prime contractor) at Laramie, Wyoming, the Pennsylvania Transportation Institute at Pennsylvania State University, SRI International at Menlo Park, California and Texas Transportation Institute at Texas A&M University.

2.3 FACTORS AFFECTING DURABILITY

The following paragraphs provide a synopsis of the factors which affect the durability of bituminous mixtures and, in particular, the mechanisms and consequences of age hardening and water sensitivity.

2.3.1 Factors Affecting Ageing

Age hardening of bitumen occurs as a result of compositional changes in the bitumen. The changes that occur are, as yet, not clearly understood, primarily due to bitumen being a rather complex mixture of organic molecules that vary widely in composition; no two crude oils, and there are nearly 1,500 (5), are exactly alike. However, many researchers have investigated age hardening of bitumens and bituminous mixtures and have provided significant advances toward a better understanding of the mechanisms of age hardening.

Mechanisms of Age Hardening

Traxler (1) identifies 15 effects which may influence the chemical, rheological and adhesion characteristics of bitumen as shown in Table 2.2. Traxler provides experimental data for some of the effects but notes that some of those listed had not been given experimental consideration. He also notes that the effects are not necessarily given in order of importance and that time, temperature and film thickness are factors in all of the effects.

Petersen (11) states that, "Durability is determined by the physical properties of the [bitumen], which in turn are determined directly by chemical composition. An understanding of the chemical factors affecting physical properties is thus fundamental to an understanding of the factors that control [bitumen] durability." He identifies three composition-related factors which govern the changes that could cause hardening of bitumen in pavements as follows:

1) loss of the oily components of bitumen by volatility or absorption by porous aggregates;
2) changes in chemical composition of bitumen molecules from reaction with atmospheric oxygen and
3) molecular structuring that produces thixotropic effects (steric hardening).

Of the three factors listed he identifies reaction with atmospheric oxygen as probably being the major and best understood cause of age hardening. In pavements where bitumen exists in thin films exposed to atmospheric oxygen, rapid and irreversible oxidation occurs resulting in the formation of polar, strongly interacting, oxygen-containing chemical functional groups that greatly increase viscosity and alter complex flow properties, a phenomenon which often leads to embrittlement of the bitumen and ultimately pavement failure. Figure 2.2 provides structural formulas of important chemical functionalities in bitumens. The chemical functional groups formed on oxidative ageing include sulphoxides, anhydrides, carboxylic acids and ketones. Table 2.3 presents data from tests conducted on four bitumens from different crude oils that had been aged under identical conditions. The data indicates that ketones and sulphoxides are the major oxidation products while anhydrides and carboxylic acids are formed in smaller amounts. Table 2.4 presents data which shows that the concentration of ketones formed on oxidative ageing is greatest in the asphaltene and polar aromatic fractions; smaller concentrations are found in the naphthene aromatic (shown as aromatic in Table 2.4) and saturate fractions. Petersen (11) explains that, because the polar aromatic and asphaltene fractions are known to contain the highest concentrations of aromatic ring systems, they have the highest content of hydrocarbon types sensitive to air oxidation.

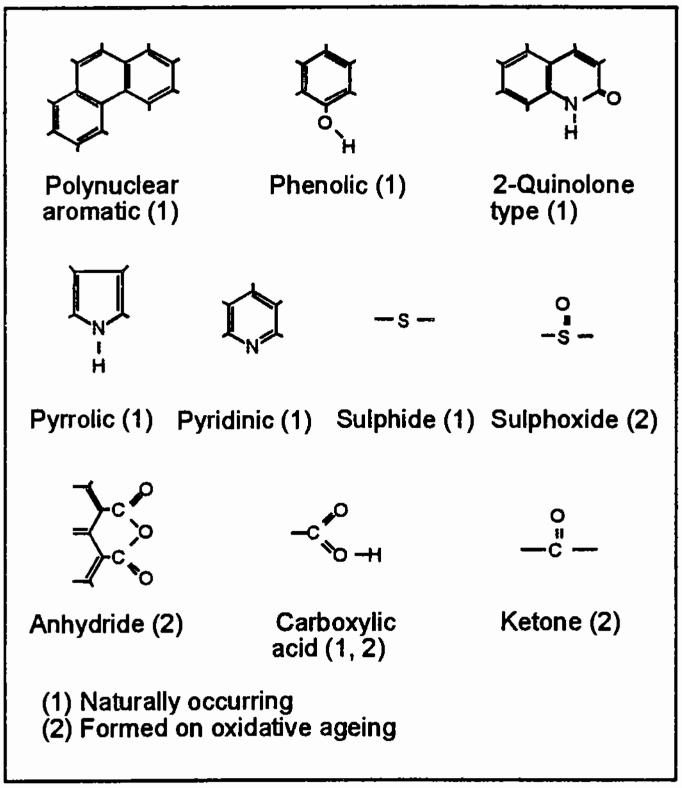

Figure 2.2. Chemical Functionalities in Bitumen Molecules Normally Present or Formed on Oxidative Ageing (11).

formed in bitumen, the strength of the associations of the polar molecules and the dispersing capacity of the non-associating components in the solvent moiety (19).

It must be stressed, however, that oxidative ageing requires the presence of oxygen. Thus, in pavements having very low void contents (or, more correctly, very low permeabilities), oxidative ageing is not likely to significantly affect the rheological properties of the pavement. For example, Vallerga and Halstead (22) found that, for pavements with void contents less than 2%, ageing during 11 to 13 years of service subsequent to hardening that occurred during mixing, transport and laydown appeared to be negligible.

Molecular structuring, a slow and largely reversible phenomenon which appears to occur concurrently and synergistically with oxidative ageing, can produce changes in the flow properties of a bitumen without changing its chemical composition. Consequently, it may be a significant factor contributing to embrittlement of the bitumen and, thus, reduced durability of the bituminous mixture. Petersen (11) stresses, however, that this phenomenon is difficult to quantify as the recovery processes (i.e., use of solvents, heat and mechanical working to obtain neat bitumen from bituminous mixtures) destroys most or all of the structuring.

The loss of volatile components (i.e., the nonpolar saturate or oily fraction of bitumen) occurs during the mixing, storage, transport and laydown of the bituminous mixture (i.e., while the bitumen is in a thin film at an elevated temperature) as well as due to absorption of the polar components by porous aggregate. Petersen (11) states that, "With current specifications and construction practices, volatility is probably not a significant contributor to pavement hardening." Similarly, Whiteoak (5) states that, "Penetration grade bitumens are relatively involatile and therefore the amount of hardening resulting from loss of volatiles is usually fairly small." The absorption of the polar components by porous aggregate, an irreversible process which might not be expected to harden bitumen, will nevertheless result in compositional changes in the bitumen which may significantly affect its properties and ageing characteristics. Traxler (1) suggests that chemical reactions or catalytic effects at the bitumen-

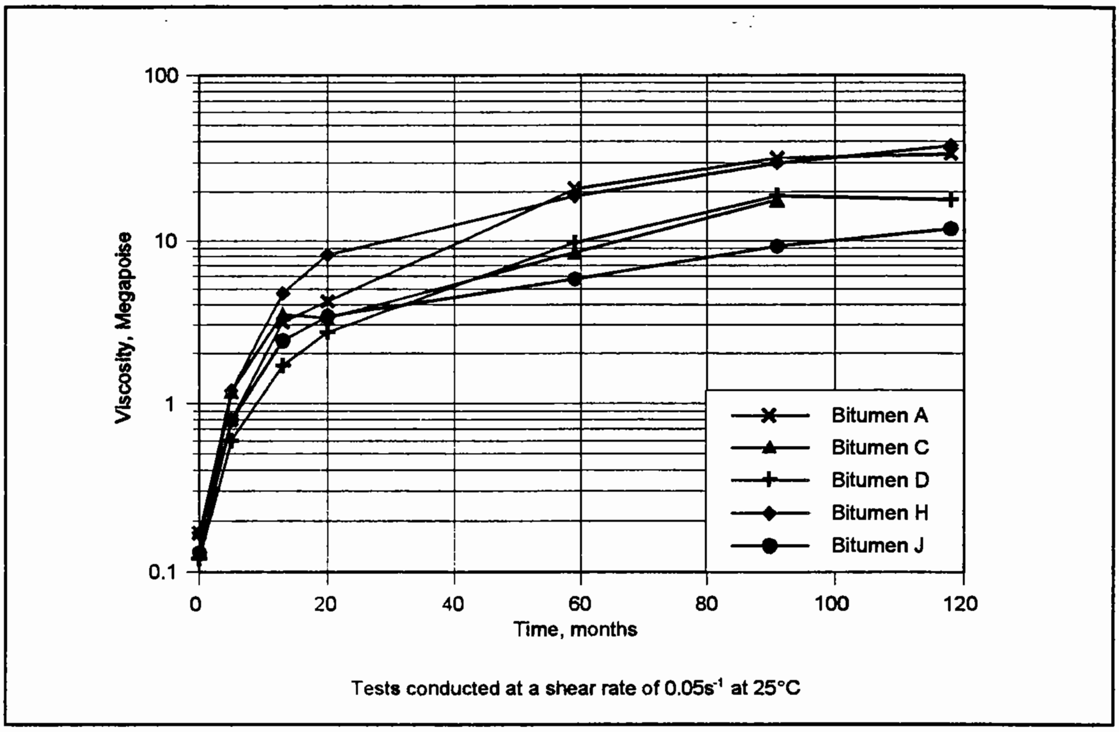

Figure 2.3. Viscosity Change of Several Bitumens During Service in Pavements (23).

cracks in the existing roadway, hence the term "reflective." Reflective cracking generally occurs as a result of stresses developed in the overlay via differential movement of the portions of the existing roadway immediately adjacent to a crack.

Age hardening reduces, through embrittlement of the bitumen, the ability of the bituminous mixture to support traffic- and thermally-induced stresses and strains. That is, age-hardened bitumen has a reduced ability to flow, by virtue of increased stiffness, under the influence of external loading. This reduction in the flow characteristics of the bitumen directly affects its vulnerability to cracking. For example, Figure 2.4 shows that oven-aged bituminous mixtures have a higher fracture temperature than do unaged mixtures, as determined by the thermal stress restrained specimen test where a 50 × 50 × 250mm specimen is held at constant length while its temperature is reduced at a constant rate until fracture occurs.

However, Lottman (4) provides a more comprehensive list of the moisture damage mechanisms that cause stripping and mixture softening as follows:

1) Pore pressure of water in the mixture voids due to wheel-loading repetitions; thermal expansion-contraction differences produced by ice formation, temperature cycling above freezing, freeze-thaw, and thermal shock; or a combination of these factors;
2) Bitumen removal by water in the mixture at moderate to higher temperatures;
3) Water-vapour interaction with the bitumen-filler mastic and larger aggregate interfaces and
4) Water interaction with clay minerals in the aggregate fines.

Of the mechanisms identified, stripping has been given, by far, the greatest attention. It has traditionally been thought that stripping is related to rupture of the adhesive bond at the bitumen-aggregate interface, a complex phenomenon involving physical and chemical properties of both the bitumen and the aggregate, with the properties of the aggregate surface playing an important role in determining the adhesive properties of the bitumen-aggregate bond (30). However, recent research has shown that cohesive failures within the aggregate or the bulk bitumen (or both), rather than separation at the bitumen-aggregate interface, are major causes of stripping (31).

Curtis et al (32) indicate that stripping of bitumen from aggregate stems from the intrusion of water into the bitumen-aggregate system. They report that the modes of failure are many and dependent on the bitumen-aggregate system. The most important modes were identified to be:

1) Separation of the bond at the interface;
2) Failure within the bitumen where soluble components are removed;
3) Cohesive failure within the aggregate and
4) Phase separation of components when the presence of water increases the solubility of polar components of the bitumen through hydrogen bonding.

Water intrusion can occur by diffusion through the bitumen film, possibly removing soluble components of the bitumen in the process, or through cracks in the bitumen

Consequences of Moisture Damage

Damage due to moisture occurs in various forms and degrees of severity. As alluded to earlier, the primary consequence of moisture damage is that of stripping, characterised by failure of the bitumen-aggregate bond. Stripping is often initially manifested in localised areas where the bitumen has migrated to the surface of the bituminous layer, referred to as flushing or bleeding. This migration of bitumen results in an unstable matrix in the lower portions of the bituminous layer which can lead to permanent deformation in the form of rutting and/or shoving as well as the development of potholes and cracking under the action of traffic loading. Subsequent intrusion of water into these localised water-damaged areas, coupled with traffic loading, further degrades the structural integrity of the pavement layer, and possibly underlying layers which, if not repaired, can lead to substantial localised failure of the pavement structure. Stripping can also result in ravelling which is characterised by loss of material at the surface of the bituminous layer.

The other major consequence of moisture damage is that of a reduction of stiffness and strength in the bituminous layer which decreases the load spreading capabilities of the pavement. Under the action of traffic loading, a pavement with reduced stiffness due to water damage is prone to rutting as a result of increased stresses and strains in the underlying layers. Loss of strength in the bitumen-aggregate matrix may also encourage stripping (2).

2.4 TESTS ADDRESSING DURABILITY

The following paragraphs provide an overview of many of the various laboratory tests which have been developed to assess the durability of bituminous mixtures.

2.4.1 Ageing Tests

Research work regarding the ageing of bituminous mixtures has been reported as early as the beginning of the 20th century (34). Since that time a majority of the research endeavours have concentrated on the ageing of the binder alone, not on mixtures. Welborn (35) and Bell (14) have produced excellent summaries of the test methods used in an attempt to predict the age hardening of bitumen and bituminous evaluated on the basis of viscosity before and after the test providing an "ageing index."

It should be pointed out here that many researchers refer to the ageing index. Its generic form can be expressed as shown in the following equation:

$$Ageing\ Index = \frac{P_{AGED}}{P_{UNAGED}} \qquad \textbf{2.1}$$

where:

P_{UNAGED} = some physical property (e.g., penetration, viscosity, softening point, etc.) measured on the unaged bitumen,

P_{AGED} = the same physical property as measured on the unaged bitumen but performed after the bitumen has been aged in some fashion (e.g., thin film oven, field ageing, etc.).

Probably the most significant modification to the TFOT involves placing bitumen in a glass jar and rotating it such that thinner films of bitumen than the 3.2mm film used in the TFOT can be aged. The **Rolling Thin Film Oven Test** (RTFOT), developed by the California Division of Highways (40), involves rotating glass bottles containing 35g samples of bitumen in an oven at 163°C for 75 minutes. Film thicknesses of 1.25mm are obtainable under these conditions. The RTFOT was adopted by ASTM in 1970 as ASTM D2872 (37). Several modifications have also been made to the RTFOT, most of them minor, as reported below.

Much thinner films of bitumen than those in the RTFOT were obtained in the **Rolling Microfilm Oven Test** (RMFO) developed by Schmidt and Santucci (41). Twenty micron film thicknesses were obtained by dissolving bitumen in benzene, coating the glass bottles with the solution, then allowing the benzene to evaporate. The bitumen was then heated to 99°C for 24 hours. The primary disadvantage of this test is that only 0.5g of bitumen is obtained from each bottle.

opposed to the 0.5g sample provided by the RMFO. Petersen presents data which shows that the TFAAT is much more severe than the TFOT and that the TFAAT causes a similar level of chemical oxidation to that occurring in pavements 11 to 13 years old. However, he cautions that the kinetics of oxidation in the TFAAT are different from that occurring in the field by virtue of temperature differences and the effects of molecular structuring and steric hardening. Petersen suggests the rate of hardening due to these two phenomena is significantly reduced after two to three years of service, however.

Similar in concept to the rolling thin film oven tests is the **Accelerated Ageing Test Device** developed at the Belgian Road Research Centre (BRRC) or more commonly known in the UK as the Centre for Road Research (CRR). This device, described by Verhasselt and Choquet (45), consists of a fairly large cylinder (124mm internal diameter, 300mm in length) capped at both ends, with one end having a 43mm diameter central aperture through which bitumen is introduced and extracted. After charging the cylinder with up to 500g of bitumen, a roller 296mm in length and 34mm in diameter is placed in the cylinder. The cylinder is then placed in a frame which rotates the cylinder as well as flows oxygen at a rate of 4 to 5 litres per hour into the aperture of the end cap. Rotation of roller within the cylinder distributes the bitumen into an even film 2mm thick on the inner wall of the cylinder. Tests can be conducted at temperatures ranging between about 65 and 110°C. At discrete periods throughout the test small portions (20 to 25g) of the aged bitumen are removed from the cylinder for evaluation (i.e., ring and ball softening point, penetration and asphaltenes content determination). Due to the large initial quantity of bitumen, the procedure allows numerous evaluations to be made and, thus, a progression of the changes in softening point, penetration and asphaltenes content are ascertained.

Using this device Choquet (46) showed that ageing the bitumen at 85°C for 144 hours reflects ageing in several field pavements with regard to formation of asphaltenes. He notes that the use of temperatures below 100°C are essential in accelerated ageing tests in order to produce chemical and rheological changes similar to those observed

Extended Heating Procedures. Pauls and Welborn (53) exposed 51 × 51mm cylinders of Ottawa sand mixtures to 163°C for various periods. The compressive strength of the cylinders was determined. Also, consistency of the recovered bitumen was compared with that of the original bitumen. Bitumens representing major sources produced in the 1930s were used in the study. The conclusions of the study included the following:

1) The hardening properties of bitumens can be determined either by measuring the compressive strength of laboratory oven-aged, moulded specimens, by tests on bitumen recovered from the laboratory-aged specimens or by the TFOT.
2) Because the TFOT procedure is relatively simple, it is highly valuable for predicting high temperature hardening of bitumens.

It should be noted that there is no suggestion that the TFOT was suitable for predicting long-term hardening due to field weathering.

Plancher et al (20) also used an oven ageing procedure on 25mm thick by 38mm diameter samples as a part of a study to evaluate the effect of lime on oxidative hardening of bitumen. It was found that the indirect tensile stiffness of lime-treated mixtures was changed less than non-treated mixtures by the ageing process. It should be noted that Plancher et al present an explanation of the chemistry of lime action. Bell (14) notes the study should also be considered with that by Edler et al (38), which found that the lime had a considerable effect in retarding ageing in bituminous samples.

Hugo and Kennedy (54) describe a method of oven ageing mixture "briquettes" at 100°C. They note that this procedure is similar to an Australian standard (Standards Association of Australia, 1980). This procedure was carried out for 4 and 7 days in a dry atmosphere and in an atmosphere of 80% relative humidity, due to the need to assess a project located near the ocean. Bitumen was recovered for viscosity determination from 100mm diameter samples cored from laboratory-produced slabs. Also, samples were weighed before and after ageing, and the weight loss used to authors emphasised that tensile strain at break was a better indicator of the effect of ageing than the tensile strength. Bell notes that this is logical since the dominant effect of ageing is embrittlement and failure to accommodate traffic- and environmentally-induced strains.

Oxidation Tests. Kumar and Goetz (56) describe a study of the effects of film thickness, voids and permeability on bitumen hardening in bituminous mixtures. Their method of hardening the mixture involved "pulling" air through a set of compacted specimens at a constant head of 0.5mm of water. The low head was used to avoid turbulence in the air flow through the specimen. The specimens were maintained at a temperature of 60°C for ten days. At 1, 2, 4, 6 and 10 days the specimens were tested in simple creep. Bell (14) notes that, because these were the only data obtained, assessment of the extent of ageing achieved is very difficult. However, he points out that quantifying the binder film thickness and the permeability of the mixtures was a valuable feature of this research work. Kumar and Goetz evaluated dense- and open-graded mixtures produced with a range of air voids, permeabilities and film thicknesses. They concluded that, for open-graded mixtures, the ratio of a film thickness factor to mixture permeability was the best predictor of resistance to hardening. For dense-graded mixtures, permeability was found to be the best indicator. Bell (14) makes reference to Goode and Lufsey (57) who also concluded that permeability was a better indicator of ageing susceptibility than air voids.

Kim et al (58) utilised pressure oxidation to age laboratory-prepared specimens representative of mixtures used in Oregon. The specimens were aged in oxygen at a pressure of 0.7MPa and a temperature of 60°C for 0, 1, 2, 3 and 5 days and the effects of ageing were evaluated by indirect tensile stiffness and indirect tensile fatigue. Bell (14) notes that stiffness ratios (i.e., the ratio of aged to unaged stiffness) generally increased with ageing time and more rapidly for poorly compacted mixtures. He also points out that some of the results demonstrated that a potential problem of ageing compacted mixtures under pressure at elevated temperatures was loss of cohesion in the matrix resulting in decreased stiffness and therefore stiffness ratios less than unity.

They identify UV light as being a major source of mixture ageing but only at the surface.

Steric Hardening. Hveem et al (40) describe a cohesiograph test to measure the "setting" quality of paving grade bitumens. The test involves making four 305mm long semi-cylindrical specimens using Ottawa sand. Two of these specimens are tested immediately in the cohesiograph whereby the long, slender specimens are extruded out of a support such that they act as cantilevers and break into short sections. The remaining two specimens are tested in the same manner after a 24 hour cure at 60°C. If there exists a difference in the "length of break," defined as the average length of the broken sections, between the two sets of specimens, then this reflects the tendency of the bitumen to "structure." The authors note that remoulding the specimens that had been cured reduced the 24 hour reading and, in some cases, the reading was reduced to that of the unaged specimens.

Recently Developed Methods

The above presentation of mixture tests for ageing was modeled after Professor Bell's literature review (14) as part of his work on the recently completed SHRP asphalt program, a portion of which was devoted to the development of an ageing technique which accurately predicts field performance. Research efforts on this extensive project considered the chemical and physical properties of several bitumens of widely varying characteristics as well as the physical properties of bitumen-aggregate mixtures. The SHRP A-002A contractor (Western Research Institute), commissioned to develop predictions of bitumen-aggregate performance based on the chemical properties of the binder, developed the TFO-PAV method, described earlier, to rapidly simulate in the laboratory oxidative age-hardening as it occurs in the field within a pavement (49). While this technique ages neat bitumen, Professor Bell's efforts involved developing an ageing technique for the bitumen-aggregate mixture. It should be noted, however, that this work was carried out solely on dense-graded mixtures.

nitrogen) is passed through a pre-heating chamber beneath the specimen which dries the "air." The dried synthetic air passes through the specimen and exhausts through an aperture in the lid of the pot. The "air" can be collected and further analysed for reaction products and/or distillation products. Potschka notes that use of synthetic air allows the investigation of oxidation and distillation (i.e., polymerisation and volatilisation) and that pure nitrogen can be used to investigate the separate influences of oxidation and distillation. He suggests that selection of test parameters such as air flow, temperature and time permits simulation of various conditions in practice (e.g., silo storage conditions, in-situ conditions, etc.).

2.4.2 Water Sensitivity Tests

Numerous methods have been developed to determine if a bituminous mixture is prone to damage due to moisture. Terrel and Shute (7) identify eight methods which have received the most attention in the United States as follows:

1) Indirect Tensile Strength Test and/or Indirect Tensile Stiffness Test with Lottman conditioning (9);
2) Indirect Tensile Strength Test with Tunnicliff and Root Conditioning (10);
3) AASHTO T283 which combines features of the above tests (commonly referred to as the Modified Lottman Test);
4) Boiling water tests;
5) Immersion-compression tests (ASTM D1075; 37);
6) Freeze-Thaw Pedestal Test;
7) Static Immersion Test (ASTM D1664; 37) and
8) Marshall Stability with conditioning.

Terrel and Shute (7) divide these tests into two general categories:

1) Tests conducted on coated aggregate whereby the loose, uncompacted mixture is immersed in water which is either held at room temperature or brought to a boil. Assessment of the separation of the bitumen from the aggregate is then made by visual inspection.
2) Tests conducted on compacted mixtures which can be laboratory-prepared specimens or cores taken from existing pavements. Assessment of moisture

whether the methods can be improved or combined such that they more accurately reflect in-service performance.

Tests on Loose Mixtures

Several methods have been developed to assess the amount of bitumen loss which occurs as a result of uncompacted coated aggregate being immersed in water. The methods include the Boiling Water Test, the Static Immersion Test, dynamic immersion tests and chemical immersion tests.

The **Boiling Water Test** involves placing a 200 to 300g sample of coated aggregate (single size aggregate or aggregate graded to design specifications) in boiling water for 1 to 10 minutes. For the 10 minute version, the mixture is stirred 3 times with a glass rod whilst it is being boiled. After boiling, the mixture is dried and the amount of bitumen loss is determined by visual assessment. Terrel and Shute (7) note that some researchers have found the test useful in assessing the effectiveness of antistripping additives while others found the test provided poor results in identifying mixtures known to be sensitive to water. They also note that a limitation of the test is that it reflects only the loss of adhesion and does not address loss of cohesion.

The **Static Immersion Test** (ASTM D1664; 37) involves coating 100g of aggregate with bitumen, immersing it in 400ml of distilled water with a pH of 6 to 7 for 16 to 18 hours, then visually estimating the total visible area of the coated aggregate as above or below 95%. The visual assessment is made while the mixture is still immersed in the water. It should be noted that the method is applicable to cutback, emulsified and semi-solid bitumens and tars. Terrel and Shute (7) point out that, although the method may indicate mixtures showing some degree of water sensitivity, it is doubtful that the long-term potential of stripping is addressed.

Whiteoak (5) indicates that **dynamic immersion tests** are similar to static immersion tests except the mixture is mechanically agitated by shaking or kneading. A visual assessment is made to estimate the degree of stripping. He notes that the reproducibility of this type of test is very poor.

which withstand 20 to 25 cycles are deemed to be moisture resistant. Terrel and Shute (7) note that the variation or effect of physical properties such as aggregate gradation, density, and interlock are minimized through use of the single size aggregate so that the test primarily evaluates the strength of bonding and binder cohesion. They also note that some researchers have found the test of little potential for identifying moisture susceptible mixtures while others have found it to be useful in evaluating aggregates for moisture sensitivity and in determining the effectiveness of antistripping additives.

The **Immersion Compression Test** (ASTM D1075; 37) is widely used throughout the United States to evaluate the loss of cohesion in compacted bituminous mixtures. In this test the index of retained strength (IRS) is obtained by comparing the compressive strength of freshly moulded specimens with the compressive strength of duplicate specimens that have been immersed in water for 4 days at 49°C. Terrel and Shute (7) note that the Asphalt Institute recommends that mixtures be rejected if they have an IRS less than or equal to 75%.

Whiteoak (5) describes **immersion mechanical tests** whereby measurement of the change in mechanical properties of bituminous mixtures as a result of immersion in water is determined. He states that a number of mechanical properties can be measured including flexural strength, shear strength and compressive strength but that the Marshall stability is probably the most popular test.

The **Marshall Stability Test** (AASHTO T245) is widely used for evaluating the relative performance of bituminous mixtures (e.g., evaluation of additives or modifiers). Several agencies have used the Marshall Stability Test in an attempt to evaluate moisture sensitivity of mixtures whereby the stability of unconditioned specimens are compared with the stability of duplicate specimens which have been subjected to some sort of water conditioning. Terrel and Shute (7) note that the conditioning procedure varies amongst agencies and is usually an adaptation from one of the procedures previously mentioned. Whiteoak (5) describes the Shell version of the test whereby eight specimens are fabricated using a prescribed aggregate type, water), freezing them for 15 hours at -18 to -12°C, then heating them to 60°C in a distilled water bath for 24 hours after having removed the plastic wrap.

In this method three specimens are tested dry (unconditioned), three are tested after vacuum saturation and three are tested after vacuum saturation plus freeze-thaw. The results are averaged for each set of specimens. The specimens are optionally tested for indirect tensile stiffness prior to determining the indirect tensile strength—Lottman (4) notes that loss of bond due to stripping seems to be measured more directly by tensile-type tests. From the averaged results two ratios are determined for each test method: the ratio of vacuum saturated to dry and the ratio of vacuum saturated plus freeze-thaw to dry. Lottman (4) termed the vacuum saturated to dry ratio the *short-term ratio* and found that, in a 5-year study of eight dense-graded pavements representing a variety of materials and climatic regions in the United States, the short-term ratios were reached within four years. He termed the vacuum saturated plus freeze-thaw to dry ratio the *long-term ratio* and found that, for some pavements, this ratio was reached within five years. Terrel and Shute (7) indicate that this ratio is intended to represent the field performance of the mixture from 4 to 12 years.

Tunnicliff and Root (10) report a method similar to the Lottman procedure. Like the Lottman procedure the method consists of obtaining a conditioned to unconditioned ratio of indirect tensile strength (the stiffness ratio is excluded). However, unlike the Lottman procedure, conditioning involves submerging the specimens in distilled water and incrementally applying a partial vacuum of 508mm Hg (5 minutes for each increment) until a degree of saturation of 55 to 80% is achieved followed by heating the specimens in a distilled water bath at 60°C for 24 hours. Thus, the method developed by Tunnicliff and Root is similar to the Lottman procedure in that the conditioned specimen is wetted prior to subjecting it to thermal cycling but differs with regard to vacuum saturation and thermal treatment and excludes evaluation on the basis of indirect tensile stiffness. Note that the Tunnicliff and Root procedure carefully controls the degree of saturation and excludes freezing the wetted specimen.

Table 2.5. Summary of the ECS Test Procedure (13).

Step	Description
1	Prepare test specimens using SHRP protocol.
2	Determine the geometric and gravimetric properties of the specimen.
3	Encapsulate the specimen in silicone sealant and latex rubber membrane, allow to cure for 24 hours.
4	Place the specimen in the ECS load frame and determine the air permeability.
5	Determine the unconditioned (dry) triaxial resilient modulus.
6	Apply 508mm Hg vacuum for 10 minutes.
7	Wet the specimen by pulling distilled water through the specimen for 30 minutes using a 508mm Hg vacuum.
8	Determine the unconditioned water permeability of the specimen.
9	Heat the specimen to 60°C for 6 hours, under repeated loading (124kPa).
10	Cool the specimen to 25°C for at least 2 hours. Measure the water permeability and triaxial resilient modulus. This constitutes a hot cycle.
11	Repeat Steps 9 and 10 for two more hot cycles.
12	Cool the specimen to -18°C for 6 hours, without repeated loading.
13	Heat the specimen to 25°C for at least 2 hours and measure the water permeability and triaxial resilient modulus. This constitutes a freeze cycle.
14	Split the specimen and assess the percentage of stripping.
15	Plot the triaxial resilient modulus and water permeability ratios (conditioned to unconditioned).

2.5.1 Ageing

Zube and Skog (23) published a final report on the Zaca-Wigmore Test Road, a study of newly constructed pavements on a major highway incorporating ten different 200-300 penetration grade bitumens. With one exception all crude oil sources and methods of production represented that found in California at the time; the exception was a mid-continent crude source produced in a refinery in Arkansas. All sections were constructed under nearly identical procedures beginning in October 1954

2) Relate these changes to the properties of the original bitumens, rationalising the effects of mixture properties and their variability and
3) Correlate the measured changes in field-aged bitumens with the corresponding changes in laboratory-aged bitumens from the same sources.

The physical properties of bitumens were evaluated by penetration at 15.6 and 25°C, ductility at 15.6 and 25°C, viscosity at 15.6, 25, 60°C, and ring and ball softening point. Chemical properties were determined by the Rostler precipitation method (16). Vallerga and Halstead (22) found that the most important factor in hardening of the binder in a pavement is void content of the pavement mixture. In pavements having void contents below 2%, field ageing during 11 to 13 years of service subsequent to hardening in pug-mill mixing and laydown operations appeared to be negligible. Above this level, hardening increased with higher void contents.

Kemp and Sherman (25) and Kemp and Predoehl (42) report a study whereby laboratory-prepared specimens were aged for 1, 2 and 4 years in four distinct climates in the field. The objectives of the study were to determine the relationship between bitumen properties, degree of compaction (voids), aggregate porosity and weathering under various climatic conditions. Bitumens of the same grade from three crude sources representing low, moderate and high temperature susceptibilities and two aggregate types (absorptive and nonabsorptive) were used to fabricate specimens of three void ranges: 3 to 5%, 7 to 9% and 10 to 12%. The climatic regions in California where the compacted specimens were aged included:

1) A high mountain climate characterised by mild dry summers and severe and wet snowy winters;
2) A coastal climate characterised by mild humid summers and mild wet winters;
3) An interior valley climate characterised by hot summers and cold wet winters and
4) A low desert climate characterised by mild to warm winters and very hot dry summers.

3) Pavement performance was affected significantly by the extent of air voids in a pavement. The rate of hardening of bitumens was reduced considerably in the pavements that compacted under traffic loading during the first 1½ to 2 years, such that they had air voids of less than 5%.

Bell (14) identifies several other studies which essentially identify similar findings to the studies reviewed above. These can be summarised as follows:

1) With all other factors (e.g., aggregate type, void content, climatic conditions, construction practices, etc.) being the same, or nearly so, bitumens from different sources show different rates of age hardening.
2) Void content of the compacted mixture is considered to be a major factor affecting the rate of age hardening of the bitumen. The rate of age hardening in mixtures having very low void contents appears to be negligible.
3) Changes in the physical properties of bitumen (e.g., penetration, viscosity, ductility, etc.) follow a hyperbolic relationship with time, which appears to be related to the characteristic decrease in void content with time as a result of compaction due to traffic.
4) Although many studies indicate there exists good correlations between the durability tests evaluated (typically thin film oven tests) and field performance, the correlations are generally limited to specific materials and/or conditions; no single test has shown good correlation over a wide range of materials and conditions.

Bell et al (65) describe the validation of the short- and long-term ageing procedures developed during the SHRP asphalt program. Preliminary evaluation of the short-term ageing procedure using a limited number of field sites indicated that the stiffness modulus of compacted mixtures that had been aged for 4 hours at 135°C prior to compaction closely corresponded to that of "field" specimens without additives but underestimated the stiffness modulus of specimens with additives (lime and rubber). A good correlation was shown between laboratory specimens aged for 8 to 12 hours at 135°C and "field" specimens with additives. The "field" specimens in this study as the short- and long-term ratios. Comparison of these ratios were made to the ratios of field cores obtained periodically throughout the study as well as to the ratios of laboratory-fabricated specimens comprised of the same materials used in the pavements. In most cases, predictive ratios determined from the initial cores were greater than the ratios predicted by tests conducted on the laboratory-fabricated specimens indicating that moisture damage is overestimated by the use of the laboratory-fabricated specimens.

For the mixtures which showed low long-term ratios (i.e., the ratio of vacuum saturated plus freeze-thaw strength to dry strength) when they were initially cored, the ratios for the field cores obtained thereafter began to show a decrease after two to three years. Also at this time, the onset of stripping was observed in these mixtures which later became so severe that disintegration of the field cores occurred.

Six of the eight pavement sections developed ratios greater than unity during the first year the pavements were in service indicating that the saturated cores had strengths (and stiffnesses) which were greater than the dry cores. Lottman notes that this was not always predicted by the ratios obtained from laboratory-fabricated specimens and that there appears to be an initial strengthening and stiffening effect in the field due to the early phases of moisture conditioning. He further suggests that field predictions may be difficult to make by using laboratory specimens because of the complexities of interaction between early moisture conditioning, bitumen ageing mechanisms and aggregate surface reactions.

Where the long-term ratios obtained from laboratory-fabricated specimens predicted mixtures which were prone to stripping, damage in the form of stripping was first observed when the ratios from field cores decreased to 0.80 and became more severe as the ratios decreased further.

Short-term ratios (i.e., the ratio of vacuum saturated strength to dry strength) can indicate the onset of stripping within the first four years of the pavement's life. Long-term ratios (i.e., the ratio of vacuum saturated plus freeze-thaw strength to dry

2.6 CONCLUDING DISCUSSION

The effects of moisture can adversely affect bituminous mixtures, particularly if proper construction practices are not followed (e.g., poor compaction). It is generally agreed that moisture damage can be manifested in the loss of adhesion between the bitumen and the aggregate (stripping) and/or loss of cohesion in the mixture. Clear evidence of the mechanisms of the loss of cohesion was not presented but it is apparent that moisture can result in a reduction in the stiffness and strength of compacted bituminous mixtures. Loss of adhesion is presently believed to be the result of failure in the aggregate, failure in the bulk bitumen or a combination of the two. The adhesion and adsorption characteristics of bitumen-aggregate systems are dependent more on the aggregate surface chemistry than the composition of the bitumen, but bitumen composition has some influence. Thus, the adhesion and debonding characteristics of the bitumen-aggregate system must be determined by the physical and chemical nature of the bond (i.e., stripping cannot be determined solely by the generic aggregate type). However, there is evidence that aggregate surfaces rich in alkaline earth metals (e.g., calcium and magnesium) are less susceptible to adhesive debonding of the bitumen in the presence of water than are surfaces rich in alkali metal elements (e.g., sodium and potassium).

The tests which have attempted to predict moisture damage to bituminous pavements have largely been empirical and, as a consequence, generally fall short of accurately predicting field performance. The Lottman procedure (NCHRP 246) and its variations (i.e., AASHTO T283 and NCHRP 274) have seen wide use in the United States but no studies in the literature search were found which conclusively validated the methods over a wide range of materials, climatic conditions and times. The Net Adsorption Test was developed for the SHRP asphalt program as a screening method for selecting bitumen-aggregate combinations as well as to evaluate the efficacy of antistripping additives. The Environmental Conditioning System (ECS), which was also developed for the SHRP asphalt program, is a performance-related test to evaluate the long-term performance of bituminous mixtures. It should be pointed out that these tests have not been conclusively validated over a wide range of materials, climatic conditions and times. Thus, the conclusion which can be drawn is that there neat bitumen, has emerged as the binder specification test for the SHRP SUPERPAVE mixture design system. It is this Author's opinion that a test method used to evaluate the performance of a mixture must be conducted on all components of the mixture in proportions mixed and compacted to in-service conditions and that the evaluation of its performance be based on fundamental engineering properties. It is also this Author's opinion that the efforts by Professor Bell in his work to develop ageing techniques carried out on bituminous mixtures and evaluated by tangible engineering properties is closer to reality than tests conducted on neat bitumen. Thus, the conclusion that can be drawn is that, although tests on the binder alone have shown a degree of correlation to field performance for a limited number of materials and under specific conditions, the effect of the aggregate is neglected even though the effect of the aggregate has been shown to have a significant influence on the ageing characteristics of bituminous mixtures. It can further be concluded that tests on the neat bitumen are not tests which evaluate the fundamental properties of bitumen-aggregate mixtures.

2.7 REFERENCES

1. Traxler, R.N., "Durability of Asphalt Cements," *Association of Asphalt Paving Technologists*, Vol. 32, pp 44-63, 1963.

2. Kennedy, T.W., "Prevention of Water Damage in Asphalt Mixtures," *STP 899*, American Society for Testing and Materials, pp 119-133, Philadelphia, 1985.

3. Terrel, R.L. and Al-Swailmi, S., "Water Sensitivity of Asphalt-Aggregate Mixes: Test Selection," *SHRP-A-403*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1994.

4. Lottman, R.P., "Laboratory Test Method for Predicting Moisture-Induced Damage to Asphalt Concrete," *TRR 843*, pp 88-95, Transportation Research Board, Washington, D.C., 1982.

5. Whiteoak, D., *The Shell Bitumen Handbook*, Surrey, UK, 1990.

6. "Mix Design Methods for Asphalt Concrete and Other Hot-Mix Types," Manual Series No.2 (MS-2), The Asphalt Institute, Lexington, Kentucky, 1988.

7. Terrel, R.L. and Shute, J.W., "Summary Report on Water Sensitivity," *SHRP-A/IR-89-003*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1989.

20. Plancher, H., Green, E.L. and Petersen, J.C., "Reduction of Oxidative Hardening of Asphalts by Treatment with Hydrated Lime-A Mechanistic Study," *Association of Asphalt Paving Technologists*, Vol. 45, pp 1-24, 1976.

21. Petersen, J.C., Barbour, F.A. and Dorrence, S.M., "Catalysis of Asphalt Oxidation by Mineral Aggregate Surfaces and Asphalt Components," *Association of Asphalt Paving Technologists*, Vol. 43, pp 162-177, 1974.

22. Vallerga, B.A. and Halstead, W.J., "Effects of Field Aging on Fundamental Properties of Paving Asphalts," *HRR 361*, pp 71-92, Highway Research Board, Washington, D.C., 1971

23. Zube, E. and Skog, J., "Final Report on the Zaca-Wigmore Asphalt Road Test," *Association of Asphalt Paving Technologists*, Vol. 38, pp 1-38, 1969.

24. Lee, D.Y., "Development of a Laboratory Durability Test for Asphalts," *HRR 231*, pp 34-49, Highway Research Record, 1968.

25. Kemp, G.R. and Sherman, G.B., " Significant Studies on Asphalt Durability: California Experience," *TRR 999*, pp 36-41, Transportation Research Board, Washington D.C., 1984.

26. Kandhal, P.S. and Koehler, W.C., "Significant Studies on Asphalt Durability: Pennsylvania Experience," *TRR 999*, pp 41-50, Transportation Research Board, Washington D.C., 1984.

27. Shah, S.C., "Asphalt Cement Consistency," *TRR 695*, pp 1-7, Transportation Research Board, Washington, D.C., 1978.

28. Culley, R.W., "Relationships Between Hardening of Asphalt Cements and Transverse Cracking of Pavements in Saskatchewan," *Association of Asphalt Paving Technologists*, Vol. 38, pp 629-659, 1969.

29. Vinson, T.S., Jackson, N.M. and Jung, D., "Thermal Cracking Resistance of Asphalt Concrete: an Experimental Approach," *7th International Conference on Asphalt Pavements*, Vol. 3, pp 395-409, International Society for Asphalt Pavements, Nottingham, 1992.

30. Petersen, J.C., Plancher, H., Ensley, E.K., Venable, R.L. and Miyake, G., "Chemistry of Asphalt-Aggregate Interaction: Relationship with Pavement Moisture-Damage Prediction Test," *TRR 843*, pp 95-104, Transportation Research Board, Washington, D.C., 1982.

31. Jamieson, I.L., Jones, D.R. and Moulthrop, J.S., "Advances in the Understanding of Binder-Aggregate Adhesion and Resistance to Stripping," *Highways and Transport*, January, 1993.

45. Verhasselt, A.F. and Choquet, F.S., "A New Approach to Studying the Kinetics of Bitumen Ageing," *International Symposium on Chemistry of Bitumens*, Vol. II, pp 686-705, Rome, 1991.

46. Choquet, F.S., "Bitumen Ageing," Centre de Recherches Routières, Brussels, May, 1993.

47. Bell, C.A. and Sosnovske, D., "Validation of the A-002A Hypothesis for Aging," *TM-OSU-A-003A-92-24, Final Summary Report to SHRP: 92-2*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1992.

48. Robertson, R.E., Branthaver, J.F., Plancher, H., Duvall, J.J., Ensley, K., Harnsberger, P.M. and Petersen, J.C., "Chemical Properties of Asphalts and Their Relationships to Pavement Performance," *Association of Asphalt Paving Technologists*, Vol. 60, pp 413-436, 1991.

49. Christensen, D.W. and Anderson, D.A., "Interpretation of Dynamic Mechanical Test Data for Paving Grade Asphalt Cements," *Association of Asphalt Paving Technologists*, Vol. 61, pp 67-116, 1992.

50. Anderson, D.A., Christensen, D.W., Bahia, H.U., Dongre, R., Sharma, M.G., Antle, C.E. and Button, J., "Binder Characterization, Volume 3: Physical Characterization," *SHRP-A-369*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1994.

51. Edited by Harrigan, E.T., Leahy, R.B. and Youtcheff, J.S., "The SUPERPAVE Mix Design System Manual of Specifications, Test Methods, and Practices," *SHRP-A-379*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1994.

52. Abson, G., "Apparatus for the Recovery of Asphalts," *American Society for Testing and Materials*, Part II, Vol. 33, 1933.

53. Pauls, J.T. and Welborn, J.Y., "Studies of the Hardening Properties of Asphaltic Materials," *Association of Asphalt Paving Technologists*, Vol. 21, pp 48-75, 1952.

54. Hugo, F. and Kennedy, T.W., "Surface Cracking of Asphalt Mixtures in Southern Africa," *Association of Asphalt Paving Technologists,* Vol. 54, pp 454-501, 1985.

55. Von Quintas, H.J., Scherocman, J.A, Hughes, C.S. and Kennedy, T.W., "Asphalt-Aggregate Mixture Analysis System," *NCHRP 338*, National Research Council, Washington, D.C., 1991.

56. Kumar, A. and Goetz, W.H., "Asphalt Hardening as Affected by Film Thickness, Voids and Permeability in Asphaltic Mixtures," *Association of Asphalt Paving Technologists*, Vol. 46, pp 571-605, 1977.

3 Development of a Sample Preparation Protocol for Compacted Bituminous Mixtures

3.1 INTRODUCTION

Slabs of bituminous mixtures are frequently fabricated in the laboratories at the University of Nottingham. In preparing these slabs the quantity of material required exceeds that which can be mixed at one time. Thus, several batches are mixed to make up the quantity required. After the first batch is mixed, it is placed in an oven set to the compaction temperature for the mixture. Subsequently, the second batch is mixed and placed in the oven and the process is repeated until the required number of batches are mixed. The logistics of this process results in the first batch of material undergoing a longer storage period relative to all subsequent batches. Similarly, the second batch is subjected to a longer storage period than all subsequent batches, and so on.

It was hypothesised that the variation in curing periods amongst the batches of bituminous materials that made up a large test specimen resulted in a variation in binder stiffness amongst the batches. Since the first batch of material was subjected to the longest storage period, it was likely that the binder in this batch had the greatest stiffness. Clearly, variation in binder stiffness amongst batches is undesirable in that non-uniformity within the slab is likely to result. Non-uniformity amongst test specimens fabricated for research purposes introduces an unnecessary variable which may create or contribute to increased scatter in test results derived from the test specimens. The same effect may occur in producing moulded cylindrical specimens from single batches if variable periods of oven storage are used. Consequently, it was considered important to develop a laboratory procedure which resulted in test specimens having been exposed to elevated temperatures for a uniform period and to ensure that this resulted in hardening of the binder representative of that which occurred during normal construction of actual paving mixtures.

The materials (bitumen and aggregate) were obtained from six sources so as to adequately embrace interactions between bitumen and aggregate. Two mixture types (continuously- and gap-graded mixtures) were used to account for differences in binder content and gradation. Several ageing periods were utilized to provide a strong relationship between short-term ageing and stiffness modulus. Three specimens per ageing period were fabricated so as to generate reasonably reliable results which provided information for both parts of this study (i.e., to determine whether or not storage time affects material properties as well as to determine the duration of time the mixture should be stored so as to simulate that which occurs in actual field construction).

3.3.2 Field Samples

The experiment design for the investigation to correlate the laboratory ageing period with that which occurs in a mixing plant was as follows:

- Number of contractors: 6
- Number of mixtures: 1 per contractor
- Number of specimens per mixture type: 6
- $6 \times 1 \times 6 = 36$ test specimens

These test specimens did not undergo additional storage at elevated temperatures beyond that which occured during mixing, storage, and transport; thus, oven ageing periods were not specified. At least six test specimens were fabricated for each mixture type so as to provide good reliability in the test results.

3.4 WORK PLAN

3.4.1 Laboratory Samples

Virgin materials (bitumen and aggregate) were sampled at the plant prior to mixing. The aggregates were sampled from the hot bin feed, thus accounting for loss of fines in the drum dryer in all but one plant which was a drum mixer plant where aggregates were sampled from the stock piles. The materials were taken to the laboratory at the University where they were mixed and oven aged for various periods (1, 2, 5, and 10 hours at 135°C) prior to compaction. They were then compacted in accordance with the percentage refusal density method. In addition, a control group was established in

Table 3.1. Summary of Results for the 20mm DBM Mixture.

Field Specimens			Laboratory Specimens			
					Stiffness (MPa)	
Sample ID	Void Content (%)	Stiffness (MPa)	Storage Period (Hours)	Average Void Content (%)	Mean	95% Confidence Interval
F1	3.3	2440	0	3.9	1440	1290 - 1580
F2	3.8	1750	1	4.3	2250	1980 - 2520
F3	4.7	1490	2	4.2	2290	1720 - 2870
F4	4.5	1290	5	4.8	2840	2520 - 3250
F5	3.7	2090	10	5.5	4060	3730 - 4390
F6	4.3	1370				
F7	4.3	1720				
F8	4.3	1720				

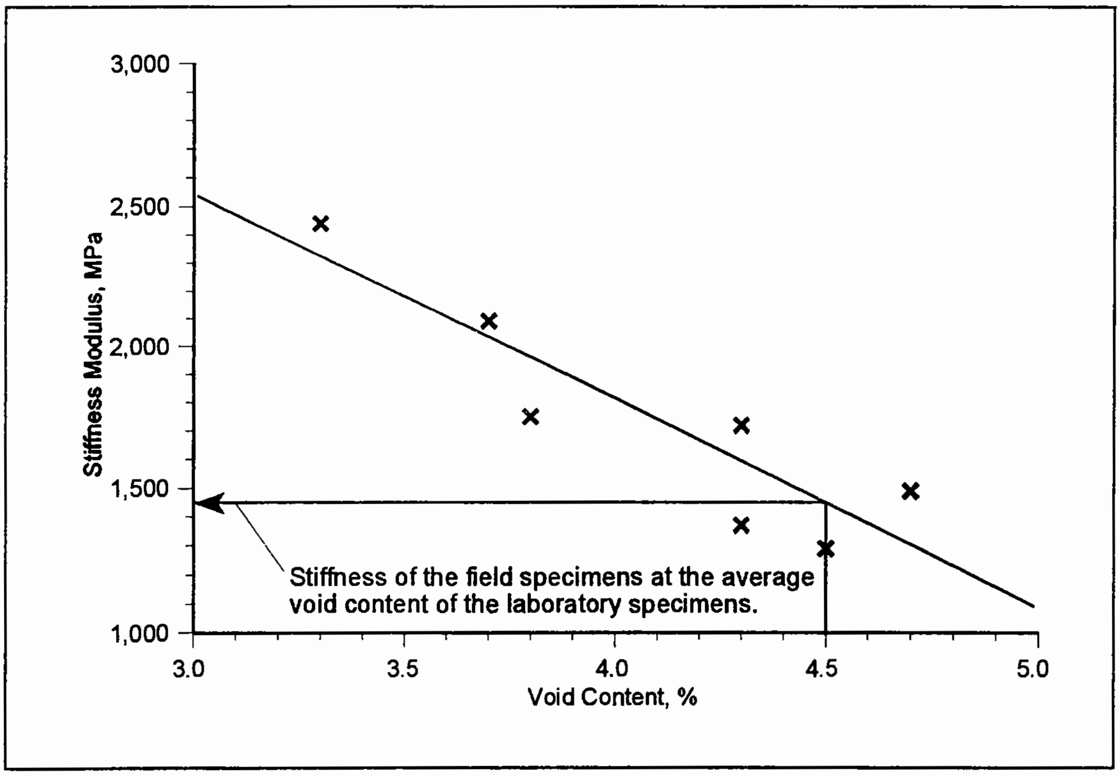

Figure 3.1. Test Results for the 20mm DBM Field Specimens.

Table 3.2. Summary of Results for the 28mm DBM Mixture.

Field Specimens			Laboratory Specimens			
Sample ID	Void Content (%)	Stiffness (MPa)	Storage Period (Hours)	Average Void Content (%)	Stiffness (MPa) Mean	Stiffness (MPa) 95% Confidence Interval
F1	4.5	3830	0	2.7	2830	2450 - 3200
F2	3.6	3370	1	2.9	3220	2930 - 3500
F3	5.1	3640	2	3.3	3660	3490 - 3830
F4	5.4	3030	5	3.2	5650	5210 - 6100
F5	5.3	3110	10	3.1	6620	6190 - 7040
F6	6.9	2310				
F7	7.5	2080				
F8	9.0	1650				

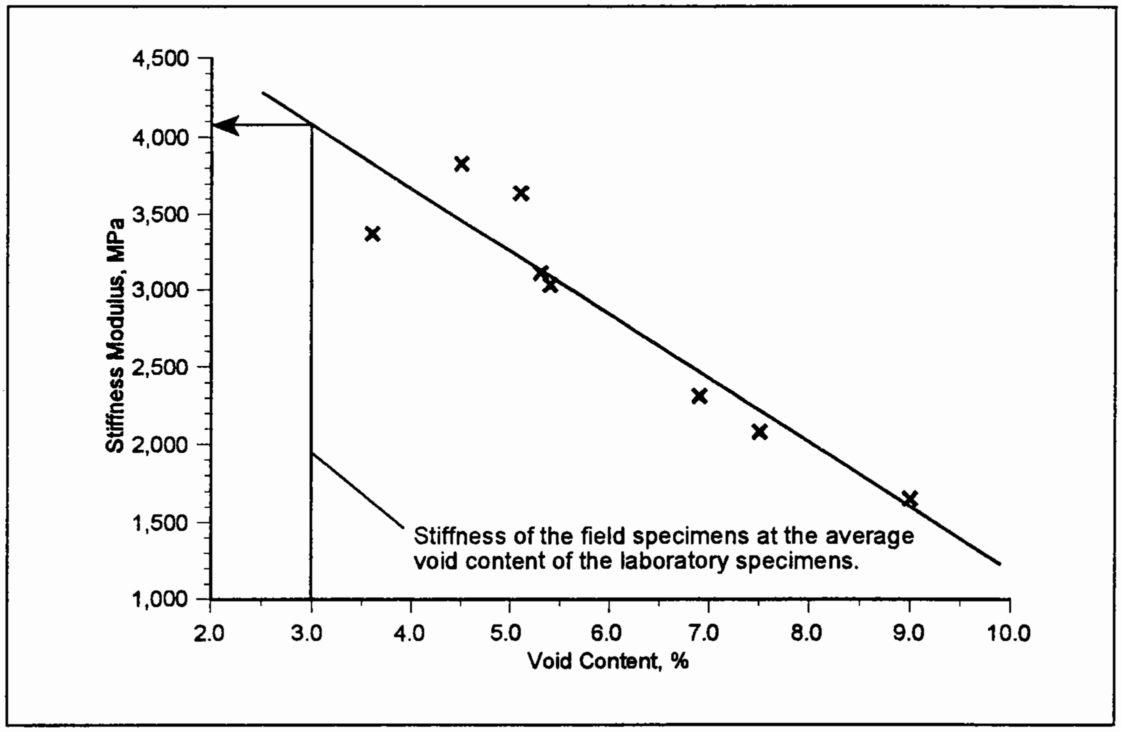

Figure 3.3. Test Results for the 28mm DBM Field Specimens.

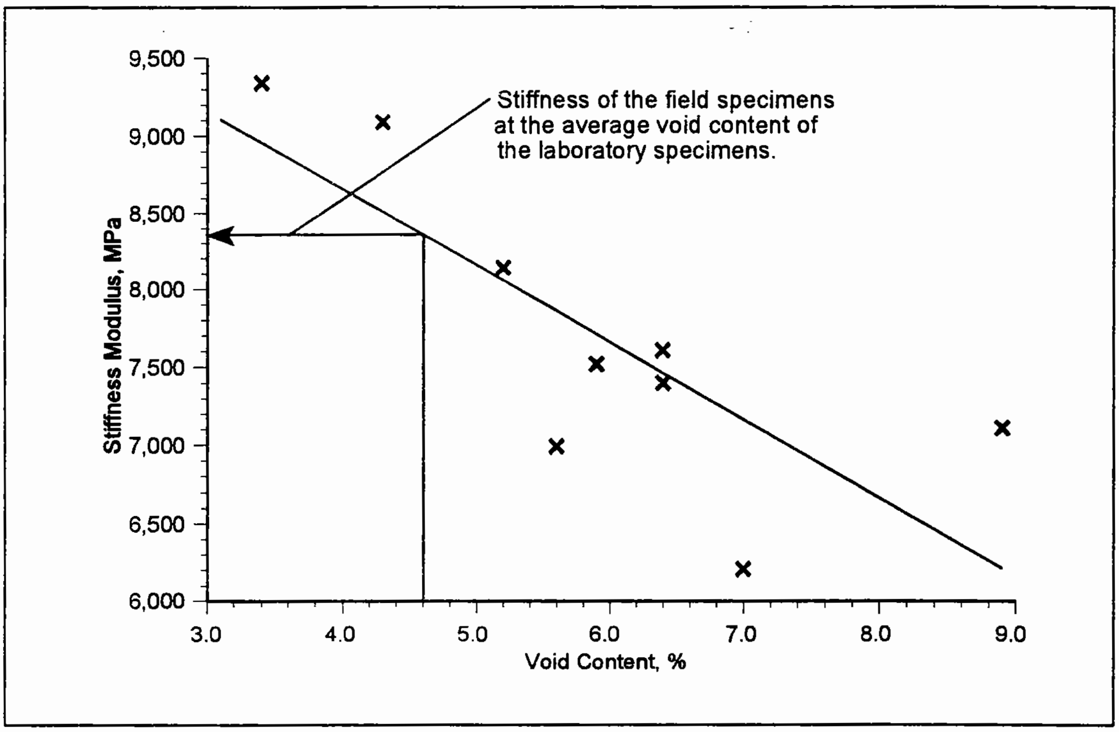

Figure 3.5. Test Results for the 28mm HDM Field Specimens.

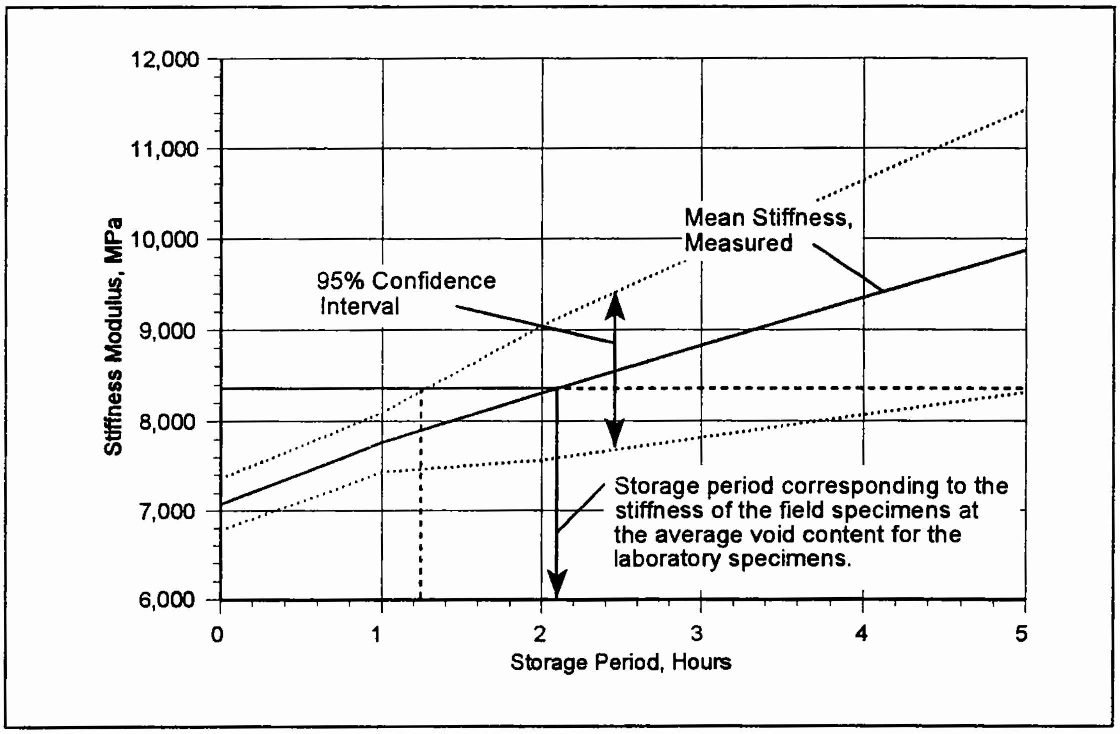

Figure 3.6. Tests Results for the 28mm HDM Laboratory Specimens.

Table 3.4. Summary of Results for the 30/10 HRA Wearing Course Materials.

Field Specimens			Laboratory Specimens			
					Stiffness (MPa)	
Sample ID	Void Content (%)	Stiffness (MPa)	Storage Period (Hours)	Average Void Content (%)	Mean	95% Confidence Interval
F1	2.0	1680	0	2.3	1840	1710 - 1970
F2	2.3	1840	1	2.5	2600	2280 - 2910
F3	3.2	1690	2	3.2	3120	2950 - 3290
F4	2.9	1790	5	2.6	4140	3420 - 4860
F5	3.1	1710	10	5.1	5430	5110 - 5760
F6	2.7	1820				
F7	3.5	1760				
F8	3.2	1880				

Mean stiffness for the field samples = 1770 MPa with a standard deviation of 72.5MPa and a coefficient of variation of 4.1%

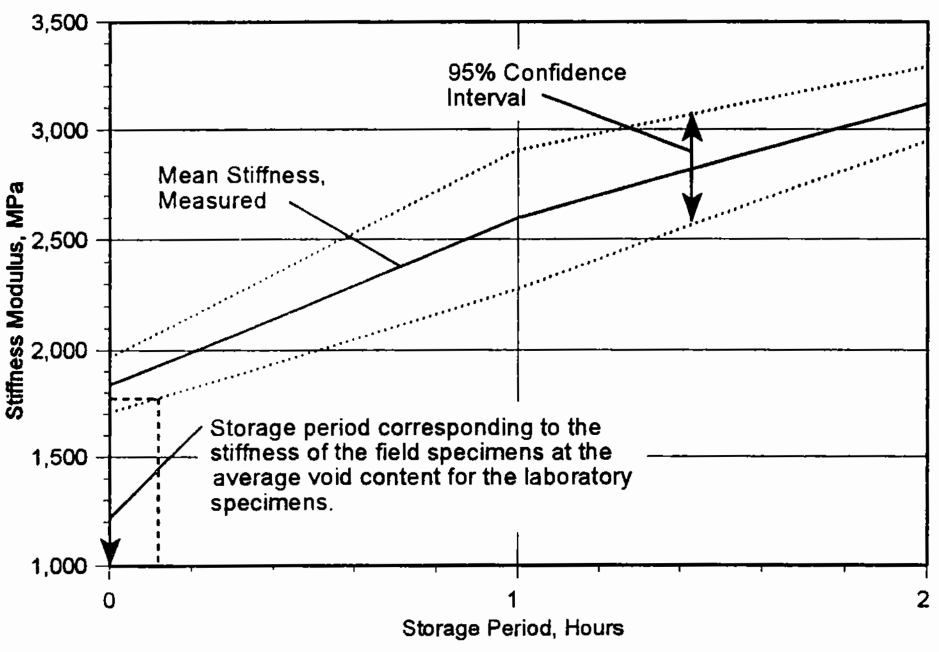

Figure 3.7. Test Results for the 30/10 HRA Laboratory Specimens.

Table 3.6. Summary of Results for the 30/14 Wearing Course Materials (Drum Mixer Plant).

Field Specimens			Laboratory Specimens			
Sample ID	Void Content (%)	Stiffness (MPa)	Storage Period (Hours)	Average Void Content (%)	Stiffness (MPa) Mean	Stiffness (MPa) 95% Confidence Interval
F1	1.7	1470	0	2.8	2750	2430 - 3080
F2	1.7	1520	1	3.1	3140	2900 - 3380
F3	1.6	1540	2	3.3	3500	3210 - 3790
F4	1.4	1740				
F5	1.4	1690				
F6	1.5	1680				
F7	1.3	1450				
F8	1.7	1640				

Mean stiffness for the field samples = 1590MPa with a standard deviation of 110MPa and a coefficient of variation of 7.0%.

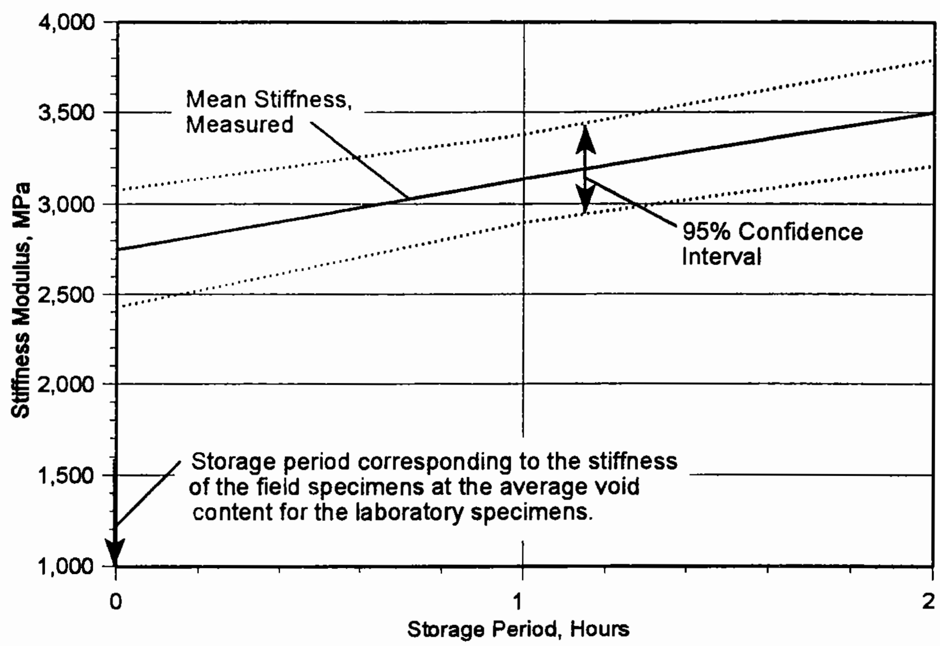

Figure 3.9. Test Results for the 30/14 HRA Laboratory Specimens from the Drum Mixer Plant.

4 Development and Evaluation of Test Protocols for Durability

4.1 INTRODUCTION

Bituminous paving mixtures are presently supplied in the UK according to recipe specifications. Based on past experience, these call for a minimum binder content to ensure good durability characteristics and fatigue resistance which, for the most part, appears to be an adequate specification. However, with the highway industry in the UK moving away from recipe specifications to end-product, performance-based specifications, there exists a need for test methods that can accurately evaluate the durability characteristics of bituminous mixtures. Although numerous tests have been developed for this purpose, standard tests have yet to be universally accepted.

One of the objectives of the Bitutest project was to develop durability test methods and practices for use in the UK. These were to address the specific areas of ageing and water sensitivity of wearing course mixtures as it was determined from the literature review (Chapter 2) that the wearing course is the pavement layer most susceptible to the effects of the environment. It was also determined from the literature review that ageing is generally divided into two distinct phases; short-term and long-term ageing, where the former refers to the amount of binder hardening which occurs during the construction process whereas the latter refers to the hardening of the binder that occurs whilst the mixture is in service (i.e., after construction). Chapter 3 described the investigation undertaken to develop a method for simulating short-term ageing. This chapter describes the efforts undertaken to develop and evaluate protocols for long-term ageing and water sensitivity.

4.2 LONG-TERM AGEING

4.2.1 Development of Long-Term Ageing Protocol

Long-term ageing is concerned with the hardening of the binder in compacted bituminous mixtures occurring over a period of many years. Simulation of long-term content and void content were varied in the mixtures to ascertain any effect that could be attributable to these variables. In addition, because it is likely that layers other than just the surface course are affected by age hardening, the effects of the ageing procedure on dense base course mixtures was also investigated.

Table 4.1 summarises the mixtures used in the study. All of the mixtures, which were comprised of materials considered to provide good durability characteristics, satisfied the design criteria specified in BS 598 (2) except where noted (i.e., some of the mixtures were intentionally fabricated with a low binder content and/or high void content). Mixtures 1 - 5 were comprised of the same materials and all mixtures were prepared in accordance with the *Standard Practice for Laboratory Preparation of Compacted Bituminous Mixtures* (Appendix A).

Table 4.1. Mixtures Used for the Evaluation of the Long-Term Ageing Protocol.

Mixture Number	Mixture Type	Materials	Binder Content by Volume (%)	Average Void Content (%)
1	30/14 HRA	Gritstone Aggregate, Asphalt Sand, Limestone Filler	7.5	2.0
2	30/14 HRA	Gritstone Aggregate, Asphalt Sand, Limestone Filler	7.5	2.3
3	30/14 HRA	Gritstone Aggregate, Asphalt Sand, Limestone Filler	7.5	4.8
4	30/14 HRA	Gritstone Aggregate, Asphalt Sand, Limestone Filler	6.5	4.8
5	20mm DBM	Gritstone Aggregate, Asphalt Sand, Limestone Filler	4.7	7.2
6	20mm DBM	Granite	4.7	5.9
7	28mm DBM	Limestone	4.5	4.8

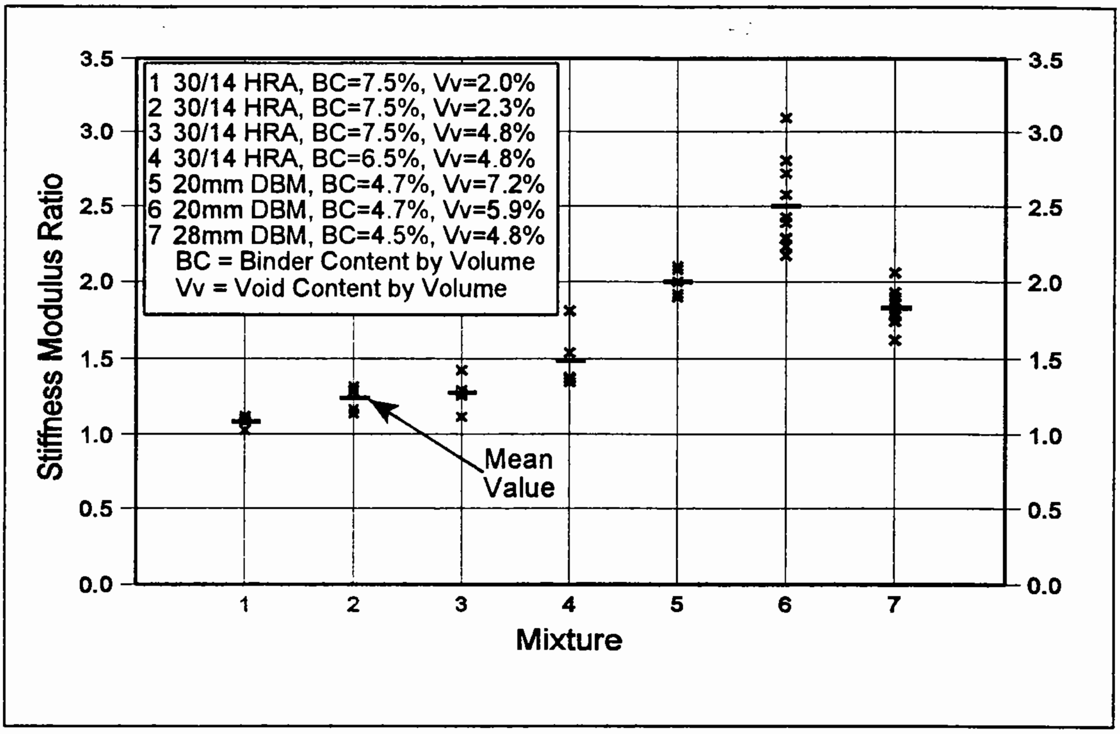

Figure 4.1. Summary of Stiffness Modulus Ratios for the Mixtures Used to Evaluate the Long-Term Ageing Protocol.

stiffness by 50% less than Mixture 6, which contained solely granite aggregate, even though the void content of Mixture 5 was greater than that of Mixture 6. That is, if ageing characterstics were based solely on volumetric proportions of binder and air voids, then it would be expected that Mixture 5, by virtue of a greater void content, would have experienced a greater stiffness modulus increase relative to Mixture 6. Instead, the opposite occurred indicating an aggregate or, more likely, bitumen-aggregate influence. The data for Mixture 7, which contained solely limestone aggregate, showed an even greater difference in stiffness increase relative to Mixture 6 supporting the bitumen-aggregate interaction theory, but it could be argued that the observed difference may be attributable, in part or in whole, to the difference in void content.

Discussion of Results

It is clear from the results that the long-term ageing protocol is effective in producing changes in bituminous mixtures which result in an increase in stiffness modulus. It is also evident that the procedure is more effective in producing greater changes in into a procedure adapted for the Nottingham Asphalt Tester. The following section presents a summary of the work carried out to evaluate the efficacy of the method.

4.3.2 Evaluation of Water Sensitivity Protocol

Mixtures Evaluated

Evaluation of the water sensitivity protocol concentrated primarily on HRA wearing course mixtures as it was presumed that this layer would be the most affected by water damage. The mixtures were comprised of materials which were considered to provide good durability characteristics but parameters such as binder content and void content were varied in the mixtures to ascertain any effect that could be attributable to these variables. Some work was also carried out on a DBM base course mixture as it was assumed that layers other than just the surface course would be affected by water damage. All mixtures met the design criteria specified in BS 598 (2) except where noted (i.e., some of the mixtures were intentionally fabricated with a low binder content and/or high void content) and were prepared in accordance with the *Standard Practice for Laboratory Preparation of Compacted Bituminous Mixtures* (Appendix A).

Evaluation Methodology

The basic methodology for evaluating the water sensitivity protocol was firstly, to carry out the procedure on mixtures representative of both properly and improperly constructed pavement materials to determine if the test was sufficiently sensitive to such differences and secondly, to vary the test conditions to determine the effect of certain test variables. Evaluations were based primarily on the change in stiffness modulus (3) of the mixture.

Test Results

Tests were initially conducted on mixtures of varying binder and/or void contents to ascertain if the water sensitivity protocol could detect such variations. The results of these tests are shown in Figure 4.2. Each data point represents the average of the results from five test specimens. The data indicate that the procedure did not appear to induce much moisture damage to the 30/14 HRA mixtures even though two of the

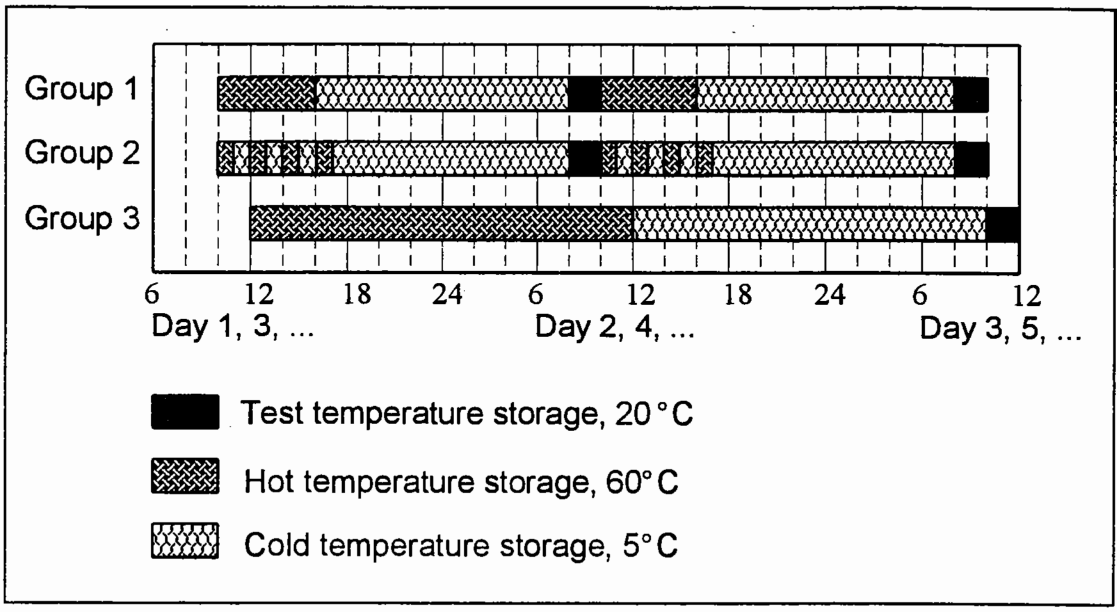

Figure 4.3. Moisture Damage Regimes for the Three 30/14 HRA Mixtures Used to Evaluate the Effects of Thermal Cycling.

subjected to the water damage regime as indicated by the water sensitivity protocol. Groups 2 and 3 formed the experimental groups with rapid and slow thermal cycling, respectively.

The results of tests for the three groups are shown in Figures 4.4 to 4.6. The results indicate a fair amount of variation amongst specimens within each group. This appears to be related to the degree of saturation as specimens with a relatively high saturation level showed a greater propensity to damage by thermal cycling. This is evident from the figures when the heavy solid line representing the average results for all five specimens is compared with the heavy dashed line representing the average results for those specimens with a relatively high degree of saturation.

A summary of results from the three groups is shown in Figure 4.7, where each data point represents the average result for the five specimens from each group. There appears to be little difference amongst the three groups suggesting that rapid or slow thermal cycling is no more effective at inducing moisture damage than the thermal cycling regime indicated by the water sensitivity protocol. Furthermore, additional cycles beyond that indicated by the water sensitivity protocol do not appear to induce

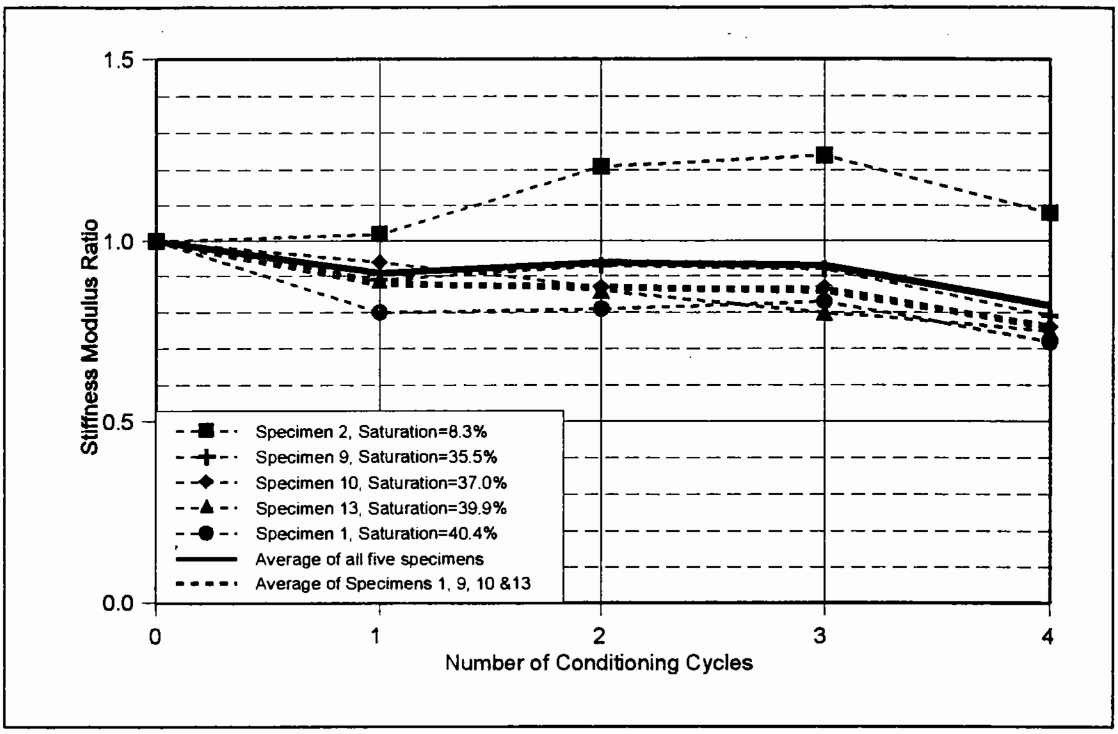

Figure 4.6. Summary of Test Results for the Specimens from Group 3 Used to Evaluate the Effects of Thermal Cycling.

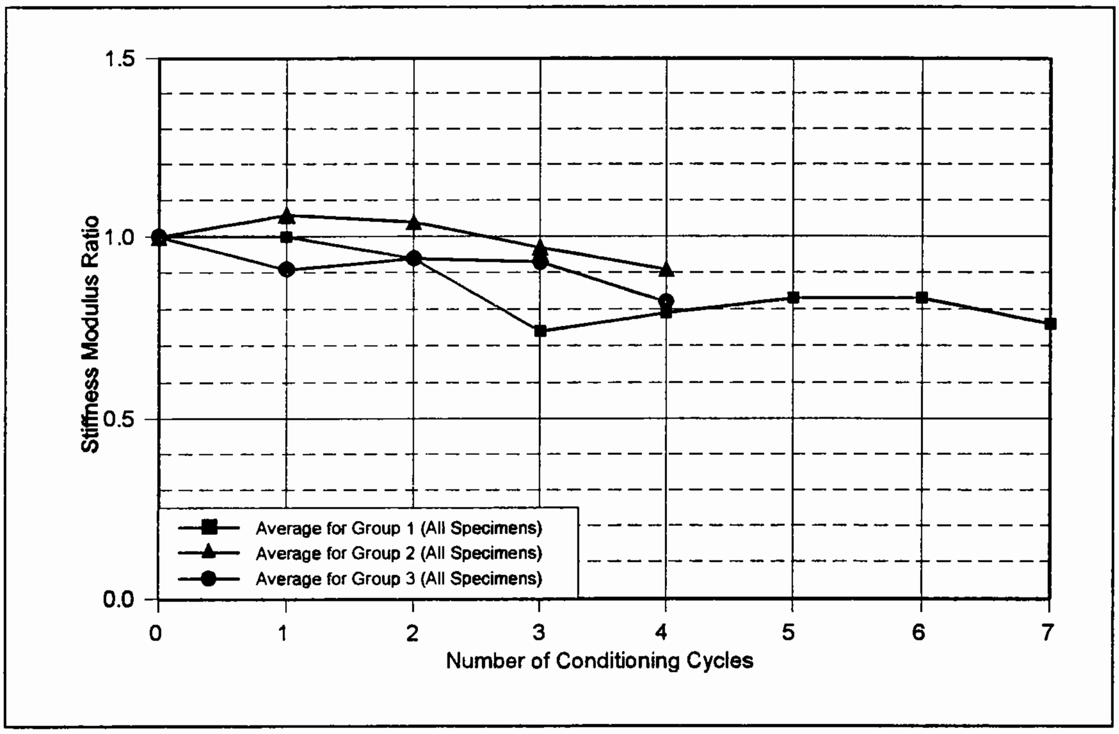

Figure 4.7. Summary of Test Results for the Three Groups of 30/14 HRA Test Specimens Used to Evaluate the Effects of Thermal Cycling.

The test results, shown in Figure 4.8, indicate that there was a significant decline in stiffness with increased conditioning cycles (on average ≈17% per cycle) for the experimental group but little change for the control group. This clearly shows that the degree of saturation is an important factor for water sensitivity of this mixture. However, it also shows that a normally durable mixture can be damaged by water provided the damage mechanism is severe enough. It is believed that the rate and magnitude of damage that occurred to the experimental group is not likely to be representative of what actually occurs on site. Instead, it is more likely that the response shown by the control group is representative of what happens on site. However, these statements are based on the presumption that HRA materials are quite impermeable and, therefore, would not have high degrees of saturation in situ.

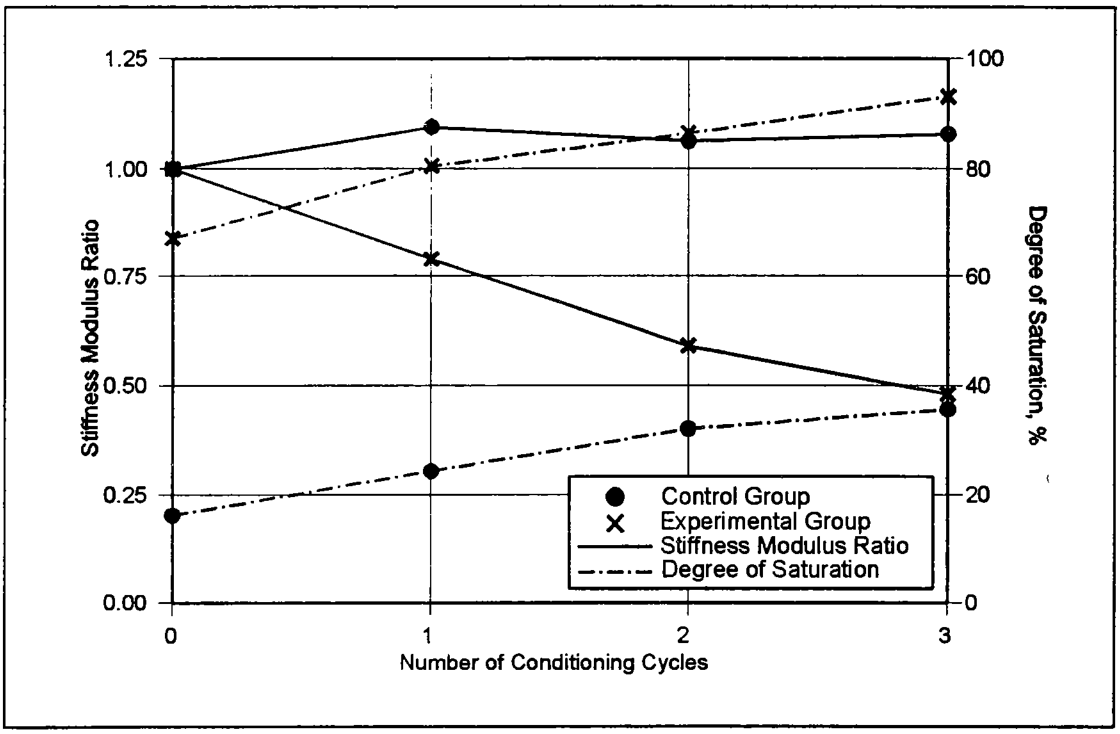

Figure 4.8. Summary of Results for Tests Investigating the Importance of the Degree of Saturation.

Monitoring the mass of the test specimens revealed that the degree of saturation appeared to increase with increased conditioning cycles (see Figure 4.8). It is believed that this is probably due to the intrusion of water into voids resulting from

Table 4.3. Summary of Specimen Void Contents and Initial Degrees of Saturation for the DBM Mixture Used to Investigate the IWTT Conditioning Regime.

Group	Partial Vacuum Level & Duration	Sample ID	Void Content (%)	Initial Degree of Saturation (%)
Control	510mm Hg (≈670 mbar) for 30 minutes	DR5-5	8.0	50.7
		DR5-6	7.3	55.5
		DR5-10	6.2	58.2
		DR5-14	7.1	63.5
		DR5-15	6.9	64.8
Experimental	510mm Hg (≈670 mbar) for 60 minutes	DR5-1	7.4	55.0
		DR5-3	7.6	53.8
		DR5-4	6.5	51.4
		DR5-7	6.5	55.0
		DR5-13	7.6	55.5

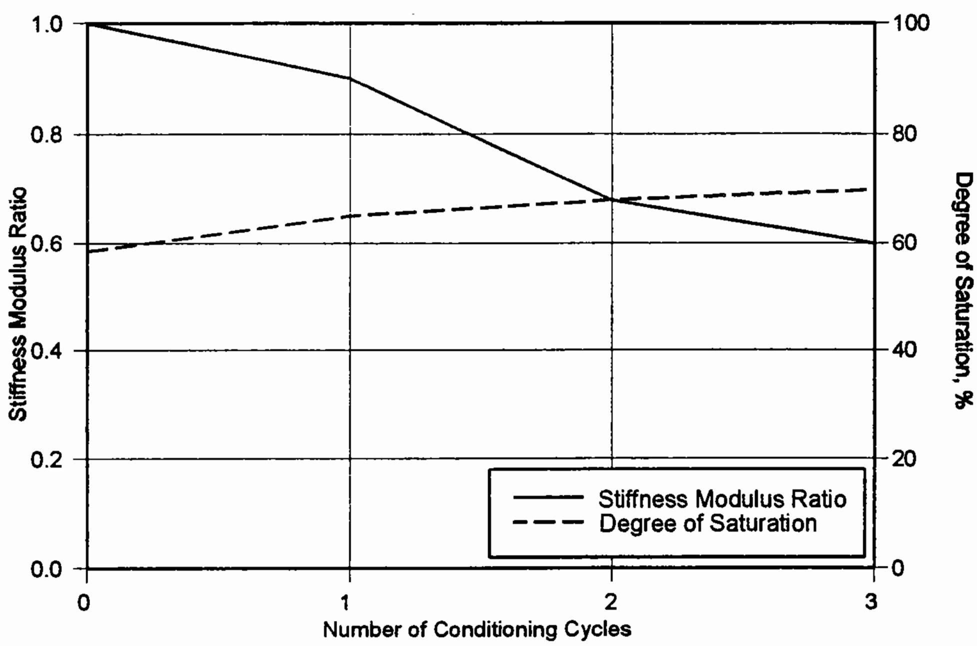

Figure 4.9. Results for the Specimens from the Control Group for Comparison With the Specimens Subjected to the IWTT Conditioning Regime.

Two 30/14 HRA wearing course mixtures incorporating an aggregate type known to be prone to water damage when used in bituminous mixtures were used in the investigation. The mixtures had like materials but different binder contents: one had a design binder content of 7% (2) while the other had a 5% binder content. A total of 36 specimens, 18 for each mixture, were fabricated using Marshall compaction (2). The 18 specimens from each mixture were divided into three groups of 6 specimens with approximately equivalent void contents as shown in Tables 4.4 and 4.5. One of these (Group I), which formed the control group, was tested in accordance with the water sensitivity protocol except that the "conditioned" stiffness modulus tests were only carried out after the third conditioning cycle. The other groups, which formed the experimental groups, were tested as follows:

1) The specimens were tested to determine the unconditioned stiffness modulus at 20°C and at 120ms rise time.
2) The specimens were vacuum saturated for 30 minutes using two different levels: Group II specimens were subjected to a partial vacuum of 510mm Hg while those in Group III were subjected to 670mm Hg.
3) The specimens were then subjected to a conditioning cycle consisting of a hot (60°C) water soak for 1 day followed by a warm (20°C) water soak for 2 hours.
4) They were then tested to determine the conditioned stiffness modulus at the same temperature and rise time as in Step 1.
5) Steps 3 and 4 were repeated until the specimens were subjected to a total of four conditioning cycles.

Comparison of the data in Tables 4.4 and 4.5 indicates that the higher partial vacuum level (i.e., 670mm Hg) did not result in an increased degree of saturation, as would be expected, relative to the lower partial vacuum level. For both mixture types, the degrees of saturation for Group I, which were subjected to a partial vacuum level of 510mm Hg, were actually higher than for Group III, which were subjected to a partial vacuum level of 670mm Hg. However, when the data for the two mixtures are compared, it is clear that higher degrees of saturation can be obtained with higher void contents.

Table 4.5. Summary of Volumetric Properties and Degrees of Saturation for the Mixtures with Low Binder Content Used to Evaluate the Simpler Test Method.

Group	Sample ID	Bulk Specific Gravity	Max. (Rice) Specific Gravity	Void Content (%)	Average Void Content (%)	Initial Degree of Saturation (%)	Average Degree of Saturation (%)
I	13	2.267	2.485	8.8	9.2	66.2	67.0
	15	2.261	2.485	9.0		64.2	
	17	2.247	2.485	9.6		70.4	
	19	2.240	2.485	9.9		70.2	
	22	2.249	2.485	9.5		66.8	
	25	2.268	2.485	8.7		64.2	
II	10	2.254	2.485	9.3	9.2	62.2	60.2
	11	2.258	2.485	9.1		59.6	
	12	2.253	2.485	9.3		62.4	
	14	2.256	2.485	9.2		58.7	
	16	2.252	2.485	9.4		59.5	
	18	2.256	2.485	9.2		58.7	
III	20	2.254	2.485	9.3	9.2	56.0	63.0
	21	2.252	2.485	9.4		59.6	
	23	2.253	2.485	9.3		72.3	
	24	2.262	2.485	9.0		62.6	
	26	2.251	2.485	9.4		59.4	
	27	2.258	2.485	9.1		67.9	

indicate that it had the greatest propensity to be damaged, possibly indicating that subjecting the mixtures to a partial vacuum, in itself, causes damage. The amount of damage induced by the regime to which the Group II specimens were subjected in the first three cycles appears to be reasonable and consistent with the threshold value of 0.7 for deeming a mixture to be sensitive to water as established by Lottman (5) and later by Terrel and Al-Swailmi (7) for the US Strategic Highway Research Program. The same is true for the Group III specimens subjected to one conditioning cycle.

For the mixture with a low binder content, the data indicate that an equivalent amount of damage to that induced by the water sensitivity protocol occurred after two conditioning cycles for the Group III specimens and after about 2⅓ cycles for the Group II specimens (Figure 4.12). However, it is clear that the majority of damage occurred during the first cycle or possibly during the partial vacuum saturation phase and that the amount of damage was quite severe. Although these results are only slightly helpful for determining an alternate hot water soak period, they do unequivocally confirm that the protocol is able to detect a water sensitive mixture whatever experimental procedure is followed.

The data for the mixture with a design binder content not only confirms that the water sensitivity protocol is an effective method for inducing water damage in mixture specimens but also suggests that the thermal cycling phase can be replaced by a single soak period of 72 hours. The data also suggest that the duration of the procedure could be shortened to 24 hours provided that the wetting phase incorporates a partial vacuum level of 670mm Hg. The data for the mixture with the low binder content indicates that, for poorly manufactured mixtures, either of the alternate conditioning methods at any of the durations are effective in causing significant damage to the specimens and that either method would, therefore, be suitable. However, because the damage to these specimens was so severe and occurred in the first conditioning cycle, it is doubtful that the data are useful for determining a hot water soak period to give an equivalent amount of damage to that induced by the water senstivity protocol. Hence, only the data for the mixture with a design binder content should be considered when deciding the duration of the hot water soak. If the cyclic conditioning phase of the protocol is to be replaced with a static soak period, it is recommended that the partial vacuum level remain at 510mm Hg for the wetting phase and that the static soak duration be 72 hours.

Discussion of Results

The results of the investigations to evaluate the water sensitivity protocol indicate that the protocol (Appendix A) is effective in causing moisture damage to mixtures that are prone to such damage. The wetting and conditioning phases of the protocol subjected to shear loading. At moderate temperatures, at which bitumens are most commonly used, they behave in a viscoelastic manner somewhere between the two extremes of this continuum. That is, at moderate temperatures, bitumens possess both elastic and viscous properties, the relative proportions depending on many factors but dominated by temperature and rate of loading. It is this fundamental property of bitumens that makes them versatile binders for paving mixtures and, therefore, widely used as such in virtually all of the habitable climates found on earth.

The viscoelastic characteristics of bitumens directly and significantly influence the performance of mixtures comprising bitumens as a binder. Knowledge of these characteristics are therefore very important to ensure good long-term performance when designing such mixtures. Measurement of the viscoelastic characteristics of bitumens is fortunately relatively simple with an apparatus referred to as a dynamic shear rheometer (DSR) or sometimes oscillatory shear rheometer or just dynamic rheometer. DSRs measure the rheological characteristics of substances from which can be obtained elastic and viscous characteristics.

For bitumens, dynamic shear rheometers come in two configurations; sliding plate or torsional rheometers, with the latter being the more common of the two. The principles of rheometry tests are the same for either configuration and therefore the remainder of this section will refer only to torsional-type rheometry.

The principles involved in dynamic shear rheometry tests are illustrated in Figure 5.1 which shows bitumen placed between a spindle and a base plate. The spindle, which can be either a disc-shaped plate or a cone, is allowed to rotate while the base plate remains fixed during testing. A test is carried out by oscillating the spindle about its own axis such that a radial line through Point A moves to Point B, reverses direction and moves past Point A to Point C, reverses direction again and moves back to Point A. This oscillation, which is smooth and continuous as illustrated in the graph in Figure 5.1, comprises one cycle which can be continuously repeated during a test. Normally, tests are carried out over a range of frequencies, which are the number of cycles completed per second, and over a range of temperatures.

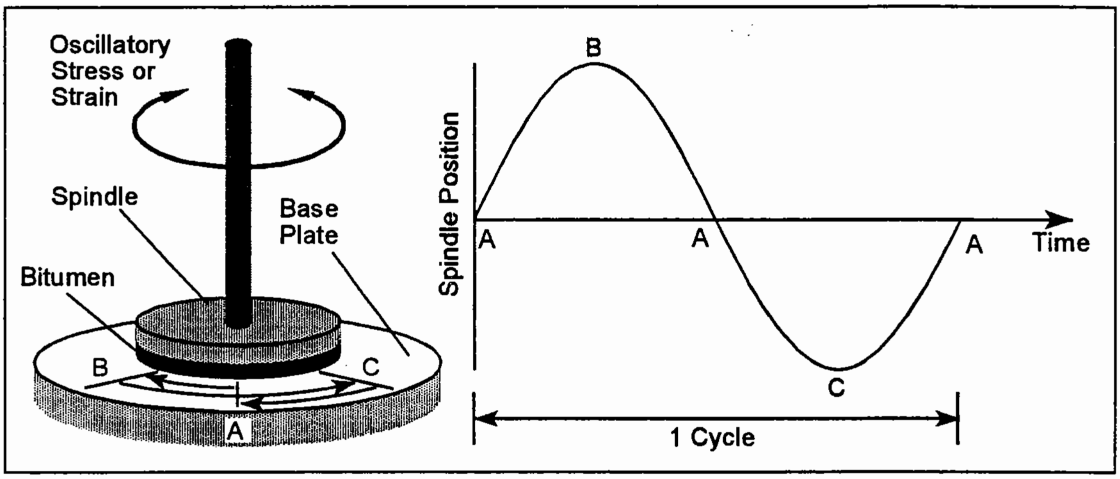

Figure 5.1. Principles of Operation of Torsional-Type Dynamic Shear Rheometers.

DSR tests can be carried out in controlled stress or controlled strain mode. In the controlled stress mode of testing, a specified magnitude of shear stress is applied to the bitumen by application of a torque to the spindle and the resultant spindle rotation is measured, from which the magnitude of shear strain is calculated. In the controlled strain mode of testing the magnitude of spindle rotation (i.e., magnitude of shear strain) is specified and the required torque needed to achieve this is measured, from which the magnitude of shear stress is calculated.

In either mode of testing the complex shear modulus (G^*) is calculated from the ratio of shear stress to shear strain as shown in Figure 5.2. The complex shear modulus, which provides a measure of the total resistance to deformation when the bitumen is subjected to shear loading, is comprised of elastic and viscous components. These are designated as the storage modulus (G') and loss modulus (G''), respectively, and are related to the complex shear modulus and to each other through the phase angle (δ), which is the phase lag between the shear stress and shear strain responses during a test.

The relationships amongst the moduli obtained from DSR tests are conveniently represented graphically on a plane Cartesian coordinate system such as that shown in Figure 5.3. The axes of the graph represent the extrema of the continuum of bitumen or very rapid shear loading. In the vast majority of circumstances to which bituminous paving mixtures are subjected, however, the temperature and loading conditions are such that the behaviour of the bitumen lies somewhere between the two axes, which is represented by a vector with magnitude G* and direction δ degrees anti-clockwise from the horizontal axis. The phase lag or phase angle (δ) indicates how much of the total modulus (G*) is attributable to viscous behaviour and how much is attributable to elastic behaviour.

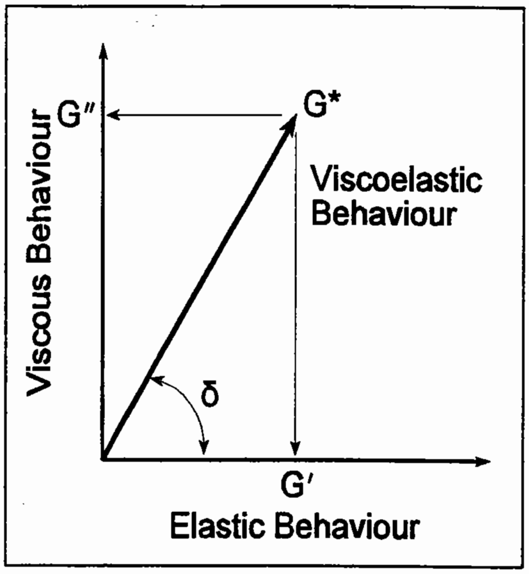

Figure 5.3. Viscoelastic Behaviour of Bitumen.

5.3 TEST METHOD DEVELOPMENT

5.3.1 Overview

Ordinarily, measurements in DSRs are made on bitumens placed between metal plates (i.e., the spindle and base plate) which are typically comprised of stainless steel and/or anodised aluminium (see Appendix C). While such measurements are useful for comparing bitumens on a "standard" material such as for specification purposes, they do not quantify any effects imparted to the bitumen by mineral aggregates. Hence, such tests may lead to inappropriate characterisation of the bitumen in the context of its performance in a bitumen-aggregate mixture.

A simple modification to the base plate of a Bohlin Model DSR50 dynamic shear rheometer allowed a small disc to be clamped and securely held in place directly below the parallel plate spindle. This, in turn, allowed the novel experimental arrangement shown in Figure 5.4 to be used for conducting dynamic shear modulus tests on bitumens coated on discs of mineral aggregate and also on stainless steel.

This arrangement presented several problems which needed to be overcome before reliable results could be obtained from the experiments. In particular, the of establishing the gap setting for the removable discs. The effect of these parameters on the measurements made in the DSR are discussed in more detail in the following paragraphs.

Temperature

Temperature control of the specimen in the Bohlin DSR50 was accomplished through submersion of the specimen in a fluid as indicated in Figure 5.4. The temperature control unit was capable of maintaining a temperature to within ±0.1°C, as recommended by Petersen et al (8).

Preliminary tests were carried out over a range of temperatures in order to define a reasonable test temperature that would allow accurate testing over the widest possible range of strain amplitudes and range of frequencies using an 8mm diameter parallel plate spindle.

The results of tests carried out on a 50pen bitumen at temperatures of 5, 25 and 40°C and at a gap setting of 50μm are shown in Figures 5.5, 5.6 and 5.7, respectively. The data clearly indicate that, at a temperature of 5°C (Figure 5.5), the DSR was not capable of applying sufficient torque to achieve the target strain amplitudes above about 2%. Figure 5.7 indicates that, at 40°C, the DSR could not apply a small enough torque to achieve the target strain amplitudes below 10%. Figure 5.6, on the other hand, indicates that the DSR was capable of applying the appropriate torque to achieve the target strain amplitudes over the majority of the frequency and strain amplitude ranges at 25°C.

Strain Amplitude and Frequency of Oscillation

The data shown in Figure 5.6 indicate that the DSR was capable of achieving target strain amplitudes from 0.5 to 5% in the frequency range of 0.01 to 10Hz for a 50pen bitumen at 25°C. However, when a 200pen bitumen was tested, the DSR output indicated that, at nearly all frequencies between 0.01 and 10Hz, the measured spindle rotation (used to calculate the strain) approached the lower limit of its practical range when target strains of 0.5% or less were specified.

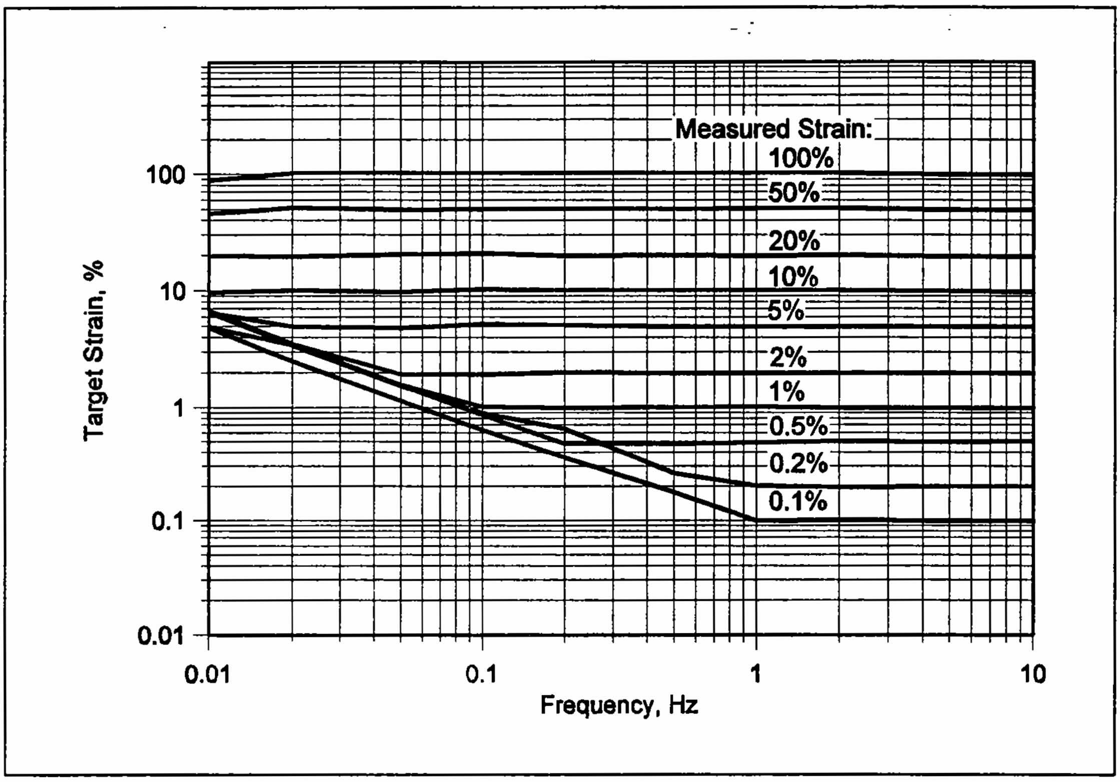

Figure 5.7. Measured Strain Versus Frequency for Various Target Strains at 40°C.

Immersion of the Spindle in the Bitumen

Figure 5.4 indicates that the bottom portion of the spindle was partially immersed in the bitumen during testing. Close examination of the bitumen around the perimeter of the spindle, however, revealed that the bitumen did not actually come into contact with the curved surface of the spindle unless sufficient time elapsed to allow it to flow (which was much longer than that required for testing). Ideally, the bitumen should have been trimmed from around the perimeter of the spindle. However, this proved to be impractical as trimming left insufficient quantities of bitumen on the aggregate surface for subsequent tests. Thus, an investigation was undertaken to determine if the test results from untrimmed specimens were significantly different from the test results from trimmed specimens.

To accomplish this, tests were carried out on a 200pen bitumen coated on three different mineral aggregates. These were the granite, limestone and greywacke,

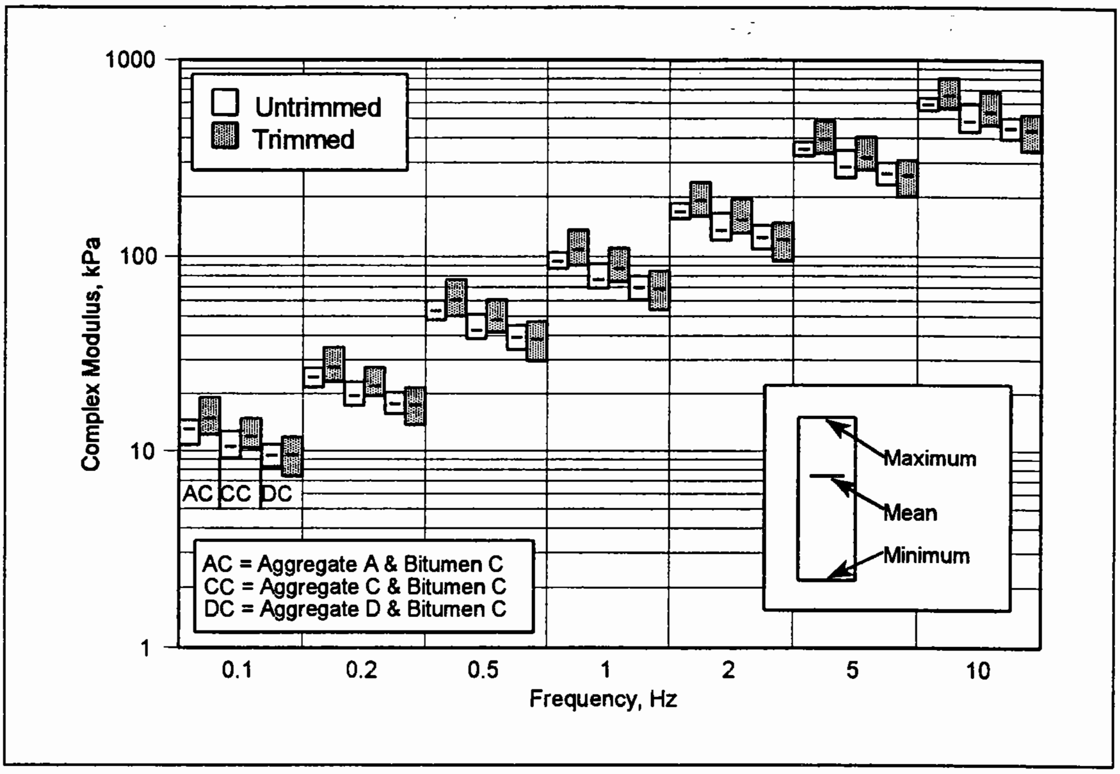

Figure 5.9. Effect of Trimmed Versus Untrimmed Bitumen Specimen on the Complex Shear Modulus.

specimens (i.e., tests were carried out on the same specimen before and after trimming and were, therefore, not independent). More specifically, the paired *t* test was carried out as follows:

1) μ_1 was used to denote the mean phase angle (or complex shear modulus) for the bitumen coated on mineral aggregate (either granite, greywacke or limestone) and trimmed prior to testing.

2) μ_2 was used to denote the mean phase angle (or complex shear modulus) for the bitumen coated on mineral aggregate (either granite, greywacke or limestone) but not trimmed prior to testing.

3) μ_d was used to denote the population mean difference between phase angles (or complex shear moduli) obtained from tests on trimmed and untrimmed bitumen specimens coated on mineral aggregate (i.e., μ_1 - μ_2)

4) The hypotheses of interest were:

H_0: $\mu_d = 0$ (i.e., $\mu_1 = \mu_2$) versus

H_a: $\mu_d \neq 0$ (i.e., $\mu_1 \neq \mu_2$)

Table 5.1. Paired *t* Statistic for Testing Differences Between DSR Tests Conducted on Trimmed and Untrimmed Bitumen Specimens.

	Aggregate A		Aggregate C		Aggregate D	
Frequency (Hz)	Phase Angle	Complex Modulus	Phase Angle	Complex Modulus	Phase Angle	Complex Modulus
0.1	-0.45	0.96	0.55	1.80	-1.24	0.02
0.2	1.04	1.00	0.59	1.76	-0.23	-0.20
0.5	-0.01	1.11	0.39	1.72	-2.85	-0.32
1	-1.11	1.12	-0.47	1.93	-0.25	-0.15
2	0.27	1.10	-0.19	1.85	-1.55	-0.42
5	-2.24	1.11	-1.55	2.04	-2.32	-0.43
10	-1.94	1.10	$\boxed{-5.78}$	1.90	$\boxed{-14.7}$	-0.38

Notes:

Boxed values indicate significance at a 95% confidence level.
$t_{critical} = 4.30$ at $\alpha = 0.05$ and 2 degrees of freedom.

film under test. This was necessary because experiments were to be conducted on bitumens coated on removable discs. To determine the magnitude of error that could be expected in establishing the gap setting, six discs were repeatedly clamped in the DSR base plate to establish the zero gap setting (see below). It was reasoned that the error associated with establishing the zero gap setting would be representative of the error associated with establishing a gap setting of 25μm. The measurements are shown in Table 5.2. They indicate that 95% of all intervals sized about ±6μm will contain the desired mean (e.g., 25μm).

Summary

Preliminary tests to evaluate the efficacy of the novel experimental arrangement shown in Figure 5.4 indicated that reliable dynamic shear modulus test results could be obtained provided that certain limitations in test conditions were observed. Collectively, the results presented above indicate that reasonable results can be obtained when tests are conducted at a temperature of 25°C, at strain amplitudes between 1 and 5% and at frequencies between 0.01 and 10Hz using an 8mm diameter the unaged bitumen. The bitumen on its disc was then aged in a forced-draft oven and tested again to obtain the rheological properties of the aged bitumen. Ageing of the bitumens was conducted at 85 °C in the absence of light for a total of 120 hours. These conditions were selected to correspond with those recommended by SHRP for long-term ageing of mixtures (10). The ageing process was interrupted after 48 hours to perform the complex modulus tests, thereby providing the rheological properties of the bitumen after 48 and 120 hours of ageing.

5.4.2 Variables Considered

The test programme involved four bitumens coated on four mineral aggregate types and on stainless steel as a control. The original programme included a fifth aggregate type but, because it possessed a relatively high porosity, it absorbed too much of the bitumen during the ageing periods such that insufficient quantities were left on the surface for subsequent DSR tests. The other variable considered in the test programme was duration of accelerated ageing in that two periods were used. Thus, the test programme, comprised of a 4 × 5 matrix, was formulated to investigate bitumens, aggregates, bitumen-aggregate interactions and ageing exposure time.

5.4.3 Materials

Aggregates

The four mineral aggregates selected for the test programme included a granite, a limestone, a greywacke (sandstone) and a basalt. The inorganic compositions of these aggregates are provided in Table 5.3. The approximate composition of the control "aggregate" (i.e., the stainless steel) was 19% chromium, 9% nickel, and 72% iron with trace quantities of carbon and nitrogen. The designations of the mineral aggregates are given in Table 5.3 while the stainless steel was given the designation of Aggregate F.

Bitumens

The bitumens were selected such that different grades from one crude oil source as well as different crude oil sources were investigated. The bitumens designated A and B were from the same source but were graded as 200pen and 50pen, respectively.

allowed to thoroughly evaporate prior to cementing an aluminium ring with 23mm outside diameter, 19mm inside diameter and height of 2mm to one surface of the disc (see Figure 5.4). Once the discs had been washed, particular care was taken when handling them so as not to touch the surface that was to be coated with bitumen.

Prior to coating the discs, they were labelled, weighed and placed in the DSR to determine the zero gap setting. This was determined by a trial-and-error procedure as follows:

1) The disc was carefully clamped in the base plate of the DSR ensuring that full contact was maintained between the disc and base plate.
2) With the torque motor in its raised position, an estimated gap setting was established on the DSR.
3) The spindle was set spinning by hand and the torque motor carefully lowered until the spindle either made contact with the disc or the torque motor reached the bottom of its travel.
4) The gap setting was adjusted and the above step repeated until the spindle just touched the disc when the torque motor was at the bottom of its travel. The spindle was deemed to be "just touching" if it remained spinning at Setting X but was slowed due to friction between the spindle and disc at Setting X minus 2.5μm (which was estimated to be half-way between adjacent finest divisions on the vernier scale).

This procedure was used throughout the test programme and, as shown in Table 5.2, allowed the gap setting to be established to well within the finest division of the vernier on the DSR, which was 5μm.

After establishing the zero gap setting, each disc was placed ring-side up on a balance accurate to 1mg and approximately 200mg of bitumen at room temperature (≈20°C) was placed on it. The discs (typically nine at a time) were then transferred to a level hot plate preheated to 163°C for 60s which was sufficient time for the straight-run bitumens to completely coat the aggregate surface within the aluminium ring. Unfortunately, the SBS-modified bitumen required a duration of 120s to uniformly data also clearly indicate that the accelerated ageing procedure resulted not only in a marked increase in stiffness (complex modulus) of the bitumen but also in a tendency for the bitumen to become more elastic as indicated by the decrease in phase angle at all frequencies. Although the results of tests from all bitumen-aggregate combinations exhibited similar trends, the magnitudes of the properties as well as the magnitudes of changes in the properties due to accelerated ageing proved to be markedly different.

Table 5.5. Number of Specimens Successfully Tested Per Bitumen-Aggregate Combination

Aggregate Type	Bitumen	Number of Specimens Successfully Tested
Granite (A)	A	3
	B	2
	C	3
	D	2
Limestone (C)	A	3
	B	3
	C	3
	D	1
Greywacke (D)	A	2
	B	3
	C	3
	D	2
Basalt (E)	A	3
	B	2
	C	3
	D	3
Stainless Steel (F)	A	3
	B	4
	C	3
	D	1

The coefficients of variation* for the phase angle and complex modulus measurements are shown in Figure 5.11. The figure indicates that, while significant variation occurred amongst some of the results, the vast majority of the measurements were

*The coefficient of variation, which provides a relative measure of variability, is the standard deviation expressed as a percentage of the mean. Low coefficients of variation indicate low variability amongst test results.

performed with good repeatability. For the complex modulus, 85% of the measurements had a coefficient of variation of less than 30%. The phase angle was measured with even better precision with nearly 95% of the measurements having a coefficient of variation of 10% or less.

5.5.1 General Observations

One of the aims of the test programme was to investigate bitumen-aggregate interactions to determine if mineral aggregates affect the physical properties of the bitumen. The effect of such interactions on the complex moduli of the unaged bitumens at oscillation frequencies of 0.01 and 10Hz are shown in Figures 5.12 and 5.13, respectively. As expected, different grades of bitumens coated on a particular aggregate exhibited significantly different modulus values. However, when data for a particular bitumen is considered, it can be seen that differences also existed amongst the various aggregates. Arguably, the differences are slight but appear different enough to warrant further analysis to determine if the data are statistically significantly different.

Figure 5.14 indicates that bitumen-aggregate interaction appears to also affect the phase angle of the unaged bitumens tested at an oscillation frequency of 0.01Hz. Although not shown, similar trends existed at the 10Hz test frequency.

Figures 5.15 and 5.16 show the average complex moduli at oscillation frequencies of 0.01 and 10Hz, respectively, of the four bitumens on the various base materials prior to and after 48 and 120 hours of accelerated ageing. It can be seen from these data that the complex modulus values for a particular bitumen after 120 hours of accelerated ageing appear to be quite different amongst the various aggregates. It is also evident that, for a particular bitumen, the aggregate type appears to influence whether the majority of modulus increase occurs in the 48 hour ageing period or in the subsequent 72 hour period. For example, the majority of the stiffness increase for Bitumen B occured in the first 48 hours of accelerated ageing on the limestone and greywacke aggregates while relatively little stiffness increase occurred during the same period on the basalt.

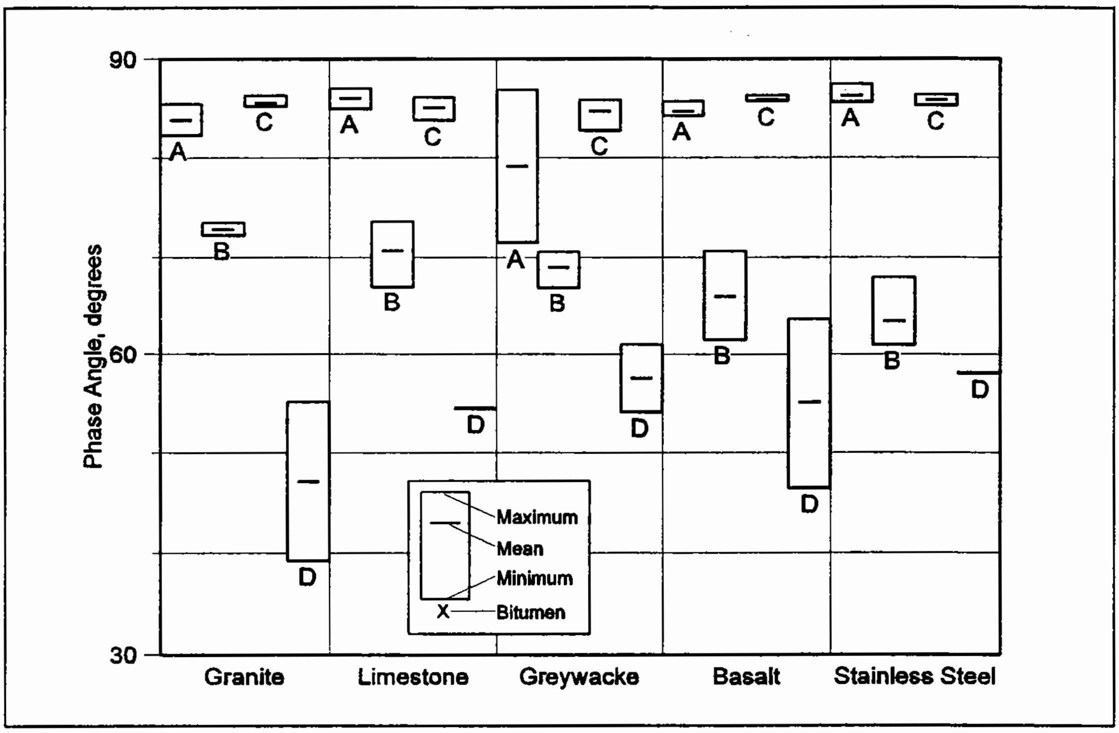

Figure 5.14. Effect of Bitumen-Aggregate Interaction on the Phase Angle at a Frequency of Oscillation of 0.01Hz.

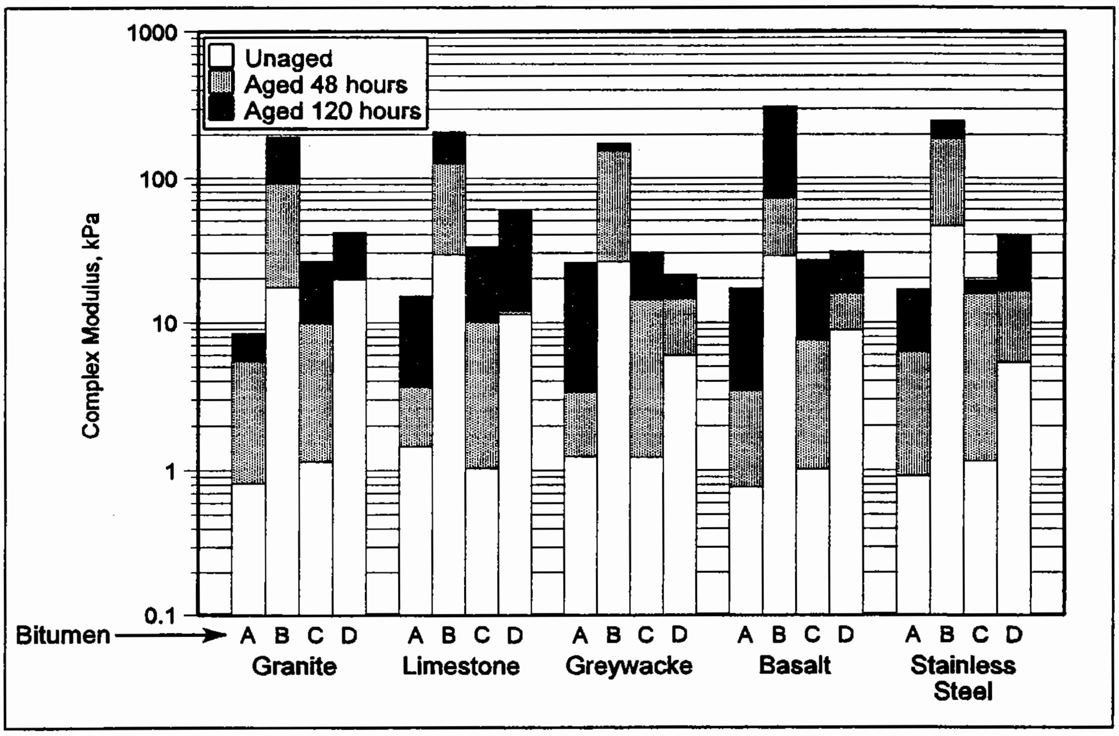

Figure 5.15. Effect of Accelerated Ageing as Indicated by the Complex Modulus at a Frequency of Oscillation of 0.01Hz.

Numerous well-known and commonly used procedures exist for making multiple comparisons of means but there is not always agreement as to which one should be used in any given situation. Devore and Peck (9) recommend the Bonferroni procedure whereas Milliken and Johnson (11) and Carmer and Swanson (12) recommend Fisher's least significant difference (LSD) method or the Waller-Duncan method, but the former also mentions the Bonferroni method. Analysis of data with unequal sample sizes cannot be accomplished with the Waller-Duncan method, however, as it has not yet been generalised to the unequal-sample-size case (11). Although either Fisher's LSD method or Bonferroni's method are appropriate to the task at hand, Fisher's LSD method was selected as it appeared to be slightly easier to apply.

In Fisher's least significant difference method an analysis of variance (*F* test) is performed first to test the null hypothesis H_0: $\mu_1 = \mu_2 = \cdots = \mu_k$ for equal means (μ_i; $i = 1, 2, \cdots, k$). If the null hypothesis is rejected by the *F* test (indicating that at least two of the means tested are different), then the means (μ_i; $i = 1, 2, \cdots, k$) are compared with one another using an ordinary least significant difference test.

The LSD method is used to compare each treatment mean to every other treatment mean where, for the present data set, mineral aggregate type is the "treatment" to which bitumen is subjected. The test statistic (i.e., least significant difference) for comparing μ_i to μ_j at a significance level $\alpha \times 100\%$ is:

$$LSD_\alpha = t_{\alpha/2,\, df} \times s \times \sqrt{\frac{1}{n_i} + \frac{1}{n_j}} \qquad \textbf{5.2}$$

where:

$t_{\alpha/2,\, df} = t_{critical}$ at level of significance $\alpha \times 100\%$ with *df* degrees of freedom

$s =$ weighted average sample standard deviation for the k treatment means

n_i, $n_j =$ sample size for treatments i and j, respectively

Table 5.6. *F* Statistic for Differences Amongst Treatment Means (Mineral Aggregate Types) According to Bitumen Type.

Bitumen	Frequency (Hz)	Unaged δ	Unaged G*	Aged 48 hours δ	Aged 48 hours G*	Aged 120 hours δ	Aged 120 hours G*
A	0.01	1.30	$\boxed{4.03}$	1.01	0.81	1.29	$\boxed{7.80}$
	0.02	1.55	3.58	0.56	0.44	$\boxed{6.33}$	$\boxed{9.12}$
$F_C = 3.63$	0.05	1.57	2.62	0.66	0.79	$\boxed{7.51}$	$\boxed{10.45}$
	0.1	1.07	3.19	0.85	0.85	$\boxed{9.81}$	$\boxed{8.32}$
	0.2	0.50	3.08	0.44	0.94	$\boxed{6.61}$	$\boxed{7.75}$
	0.5	0.75	3.44	0.31	0.88	$\boxed{7.23}$	$\boxed{6.38}$
	1	1.56	3.38	0.72	0.86	$\boxed{4.41}$	$\boxed{6.61}$
	2	0.38	3.02	0.87	0.93	$\boxed{6.90}$	$\boxed{5.94}$
	5	$\boxed{5.20}$	2.98	1.27	0.82	$\boxed{8.30}$	$\boxed{6.02}$
	10	$\boxed{5.66}$	2.68	1.33	0.74	$\boxed{10.06}$	$\boxed{5.85}$
B	0.01	3.51	3.10	2.45	1.71	1.34	2.69
	0.02	2.62	2.87	2.11	1.77	1.39	2.45
$F_C = 3.63$	0.05	$\boxed{5.30}$	2.39	1.88	1.78	1.79	2.68
	0.1	$\boxed{4.20}$	1.91	2.31	1.62	2.98	1.92
	0.2	$\boxed{4.39}$	1.52	1.75	1.69	2.28	1.40
	0.5	$\boxed{5.15}$	1.46	1.10	2.06	2.46	1.10
	1	3.01	1.26	1.42	2.50	2.86	1.08
	2	2.91	1.28	1.66	3.10	1.79	0.85
	5	2.00	1.15	2.11	$\boxed{4.23}$	1.82	0.76
	10	1.79	1.13	2.59	$\boxed{5.64}$	1.65	0.84
C	0.01	0.69	0.25	0.69	$\boxed{4.80}$	2.41	1.69
	0.02	1.57	0.29	1.37	$\boxed{6.29}$	0.83	2.11
$F_C = 3.48$	0.05	1.42	0.26	1.95	$\boxed{6.50}$	2.40	1.85
	0.1	0.15	0.23	1.30	$\boxed{7.08}$	1.55	1.42
	0.2	0.91	0.23	2.14	$\boxed{7.85}$	2.23	1.39
	0.5	0.24	0.23	$\boxed{5.81}$	$\boxed{7.44}$	0.89	1.46
	1	0.72	0.23	$\boxed{4.95}$	$\boxed{7.34}$	2.27	1.49
	2	1.61	0.23	3.11	$\boxed{7.42}$	2.87	1.47
	5	0.64	0.23	$\boxed{4.22}$	$\boxed{7.69}$	1.69	1.52
	10	0.61	0.23	$\boxed{5.92}$	$\boxed{7.88}$	1.70	1.51
D	0.01	0.81	3.65	2.36	1.13	0.63	1.69
	0.02	0.93	2.98	4.45	1.56	0.71	2.32
$F_C = 6.94$	0.05	0.93	3.09	0.88	1.77	3.14	2.07
	0.1	1.36	3.37	0.48	2.07	0.69	2.28
	0.2	1.74	3.64	1.60	2.19	1.13	2.47
	0.5	1.62	3.47	2.53	2.12	1.85	2.67
	1	2.24	3.29	2.45	2.40	1.00	2.57
	2	2.22	3.02	$\boxed{7.95}$	2.41	3.67	2.66
	5	2.77	3.31	5.20	2.52	2.71	2.73
	10	3.26	3.38	3.68	2.31	2.43	2.78

Notes:

Boxed values indicate significance at a 95% confidence level.

F_C = *F* critical value at 5% significance level.

δ = Phase angle.

G* = Complex shear modulus.

Comparisons of differences in mean phase angle and mean complex moduli for Bitumen A amongst the various mineral aggregates are listed in Tables 5.7 and 5.8, respectively, while Table 5.9 lists the comparisons of differences in mean complex moduli for Bitumen C. The top half of these tables list, for each comparison, the critical value at the 5% (i.e., $\alpha \times 100\%$) significance level whilst the bottom half lists the difference in sample means (i.e., $|\hat{\mu}_i - \hat{\mu}_j|$). The comparison between means is deemed to be significant (i.e., there exists a significant difference) if the values in the bottom half of the table are greater than those in the top half. Thus, as indicated in Table 5.7, the mean phase angle for Bitumen A on Aggregate A would not be considered different from the mean phase angle of Bitumen A on Aggregate C at a frequency of 0.02Hz but they would be considered different at a frequency of 0.05Hz. Note that the comparisons that were found to be significant are demarcated by a box.

The results indicate that there is strong evidence to support the claim that mineral aggregate affects the rheological properties of Bitumens A and C. The evidence is quite conclusive that the phase angle measurements on Bitumen A (Table 5.7) coated on Aggregate A were different from those of Bitumen A coated on Aggregates D, E or F. The phase angle results for this bitumen also indicate, albeit with less conviction, that differences existed between Aggregates A and C, Aggregates C and D, Aggregates C and E and Aggregates C and F.

The results for the comparisons between complex modulus values for Bitumen A (Table 5.8) conclusively demonstrate that there existed differences between Aggregates A and C, Aggregates A and D, Aggregates D and E and Aggregates D and F. These results also indicate that differences existed between Aggregates A and E, Aggregates A and F and Aggregates C and D, but not at all frequencies.

For Aggregate C the results of comparisons between complex moduli (Table 5.9) conclusively indicate that differences existed between Aggregates A and D, Aggregates A and F, Aggregates C and D, Aggregates C and F, Aggregates D and E and Aggregates E and F.

Table 5.8. Comparison of Differences Amongst Mean Complex Moduli of Bitumen A Coated on the Various Aggregates by Fisher's Least Significant Differences.

Frequency (Hz)	Critical Values (LSD_{α}) for Comparing Means at a 5% Significance Level									
	A - C	A - D	A - E	A - F	C - D	C - E	C - F	D - E	D - F	E - F
0.01	6.27	7.01	6.27	6.27	7.01	6.27	6.27	7.01	7.01	6.27
0.02	9.72	10.87	9.72	9.72	10.87	9.72	9.72	10.87	10.87	9.72
0.05	18.82	21.04	18.82	18.82	21.04	18.82	18.82	21.04	21.04	18.82
0.1	33.21	37.13	33.21	33.21	37.13	33.21	33.21	37.13	37.13	33.21
0.2	52.60	58.81	52.60	52.60	58.81	52.60	52.60	58.81	58.81	52.60
0.5	103.7	116.0	103.7	103.7	116.0	103.7	103.7	116.0	116.0	103.7
1	153.8	171.9	153.8	153.8	171.9	153.8	153.8	171.9	171.9	153.8
2	238.4	266.6	238.4	238.4	266.6	238.4	238.4	266.6	266.6	238.4
5	362.0	404.7	362.0	362.0	404.7	362.0	362.0	404.7	404.7	362.0
10	480.6	537.3	480.6	480.6	537.3	480.6	480.6	537.3	537.3	480.6

Frequency (Hz)	Absolute Value of Difference Between Means ($\lvert\hat{\mu}_i - \hat{\mu}_j\rvert$)									
	A - C	A - D	A - E	A - F	C - D	C - E	C - F	D - E	D - F	E - F
0.01	$\boxed{6.59}$	$\boxed{17.06}$	$\boxed{8.66}$	$\boxed{8.33}$	$\boxed{10.47}$	2.07	1.73	$\boxed{8.40}$	$\boxed{8.73}$	0.33
0.02	$\boxed{11.77}$	$\boxed{28.47}$	$\boxed{13.97}$	$\boxed{15.60}$	$\boxed{16.70}$	2.20	3.83	$\boxed{14.50}$	$\boxed{12.87}$	1.63
0.05	$\boxed{24.30}$	$\boxed{59.58}$	$\boxed{27.23}$	$\boxed{30.10}$	$\boxed{35.28}$	2.93	5.80	$\boxed{32.35}$	$\boxed{29.48}$	2.87
0.1	$\boxed{40.60}$	$\boxed{94.30}$	$\boxed{40.57}$	$\boxed{45.27}$	$\boxed{53.70}$	0.03	4.67	$\boxed{53.73}$	$\boxed{49.03}$	4.70
0.2	$\boxed{66.63}$	$\boxed{144.3}$	$\boxed{63.63}$	$\boxed{66.30}$	$\boxed{77.67}$	3.00	0.33	$\boxed{80.67}$	$\boxed{78.00}$	2.67
0.5	$\boxed{123.0}$	$\boxed{258.0}$	$\boxed{108.0}$	$\boxed{112.0}$	$\boxed{135.0}$	15.00	11.00	$\boxed{150.0}$	$\boxed{146.0}$	4.00
1	$\boxed{186.3}$	$\boxed{387.7}$	142.7	153.0	$\boxed{201.3}$	43.67	33.33	$\boxed{245.0}$	$\boxed{234.7}$	10.33
2	$\boxed{293.7}$	$\boxed{563.5}$	198.3	222.3	$\boxed{269.8}$	95.33	71.33	$\boxed{365.2}$	$\boxed{341.2}$	24.00
5	$\boxed{473.3}$	$\boxed{843.3}$	266.7	303.3	370.0	206.7	170.0	$\boxed{576.7}$	$\boxed{540.0}$	36.67
10	$\boxed{640.0}$	$\boxed{1076.7}$	306.7	353.3	436.7	333.3	286.7	$\boxed{770.0}$	$\boxed{723.3}$	46.67

Note: Boxed values indicate significance at a 95% confidence level.

5.5.3 Comparison of Results With Those on Compacted Mixtures

Two of the dense bitumen macadam mixtures used to evaluate the efficacy of the long-term ageing protocol (Chapter 4) contained the same materials as used for some of the rheology work. Mixtures 6 and 7 (see Table 4.1) were manufactured using Bitumen C and the granite and limestone aggregates (Aggregates A and C), respectively. Tests on these mixtures were conducted in the Nottingham Asphalt Tester (NAT) before and after accelerated oven ageing—either 48 or 120 hours at 85°C. The NAT tests were carried out using a load pulse rise time of 120ms which is defined as the time from the onset of the pulse load to maximum load and roughly corresponds to one-quarter of a cycle in DSR tests (i.e., from Point A to Point B in Figure 5.1). Thus, a load pulse rise time of 120ms in the NAT roughly corresponds to an oscillation frequency of 2Hz in the DSR (i.e., ¼ cycle ÷ 0.12s = $2.083s^{-1} \approx 2Hz$). The NAT tests were carried out at 20°C whereas those in the DSR were carried out at 25°C. For this reason direct comparisons of the moduli obtained from the two tests cannot be made. However, because the accelerated oven ageing procedure for the DBM mixtures was exactly the same as that for the discs coated with bitumen, comparisons based on stiffness ratios are valid.

The results of tests on the DBM mixtures are tabulated in Tables 5.10 and 5.11, respectively, while those for the corresponding combinations tested in the DSR (i.e., AC and CC, respectively) are tabulated in Appendix C. The mean stiffness ratios (i.e., ratio of aged stiffness to unaged stiffness) are summarised graphically in Figure 5.17; those shown for the DSR tests correspond to an oscillation frequency of 2Hz. The results indicate that the ranking of results by aggregate type is the same for both test methods for bitumens aged for 120 hours. Ranking of the results for bitumens aged for 48 hours is not possible due to the near equality of mean stiffness ratios. The NAT results support those shown earlier in Table 5.9 which indicated that there did not exist a significant difference between complex moduli of Bitumen C coated on Aggregate A and those of Bitumen C coated on Aggregate C. Although these results indicate that tests on compacted mixtures in the NAT may correlate reasonably well with tests on the same bitumens coated on the same aggregates in the DSR, further tests on a wider range of mixtures are needed to confirm this.

Table 5.11. Summary of Results for the 28mm DBM Mixture with Limestone Aggregate Tested in the NAT.

Sample ID	Void Content (%)	Mean Void Content for Group (%)	Stiffness Modulus, Unaged (MPa)		Duration of Ageing (hours)	Stiffness Modulus, Aged (MPa)	
			Sample	Group Mean		Sample	Group Mean
L2	5.8	4.80	1857	2247	48	2682	3232
L3	3.5		2838			4380	
L5	3.1		2714			3494	
L8	3.2		2218			2900	
L14	8.4		1606			2706	
L1	5.6	4.80	1960	2070	120	3416	3755
L4	4.9		1986			3632	
L6	5.4		1860			3312	
L7	5.4		1925			3458	
L9	4.0		1898			3534	
L10	3.3		2984			4840	
L11	4.2		1856			3583	
L12	5.7		1994			3786	
L13	5.2		1631			3357	
L15	4.3		2610			4634	

results indicated that the viscoelastic properties (complex moduli and phase angles) of the low viscosity straight-run bitumens (i.e., Bitumens A and C) were different amongst several of the aggregate types. A summary of these results is provided in Table 5.12 which shows that nearly all aggregate types gave rise to differences in response variables for both bitumens. The analyses also showed that the viscoelastic properties of the high viscosity straight-run bitumen and the polymer-modified bitumen (i.e., Bitumens B and D, respectively) were, for the most part, not affected by the mineral aggregate; for Bitumen B, however, it was shown that some influence was effected by aggregate type (Table 5.6). It is not obvious why mineral aggregate appears to only affect the low viscosity bitumens but a possible explanation follows.

A possible explanation of the results concerns the chemical composition of the surface (i.e., aggregate) with which the bitumen comes into contact which may result in preferential associations between bitumen molecules and "active" sites on the surface. That is, when bitumen is adsorbed onto aggregate (e.g., during plant mixing), the polar molecules in bitumens compete for sites on the surface of the aggregate that contain metals or charged species. Strongly polar species (e.g., sulphoxides and carboxylic acids) are more competitive than less polar (e.g., ketones) or nonpolar species. It is not likely that associations of any strength are formed until the bitumen begins to cool (due to the relative weakness of the bonding capacity of the molecules, particularly at elevated temperatures). However, once formed these associations hold the polar molecules of bitumen to the aggregate surface and become the foundation on which microstructures build their network. This would account for the structuring within a bitumen and the "catalytic" effect of the aggregate surface proposed by Branthaver (7).

It is evident from Table 5.3 that the various aggregates used in this study had widely varying mineralogical compositions. Thus, it would be expected that the different compounds, as well as different proportions of a given compound, in the aggregates would give rise to differences in electrokinetic properties amongst the aggregates. These differences would, in turn, give rise to differences in the number and strength of bonds formed with the polar molecules in bitumens.

Hence, tests conducted on bitumens coated on stainless steel or anodised aluminium may neglect the effects imparted to the bitumen by the mineral aggregate which, in turn, may lead to inappropriate characterisation of the bitumen in the context of its potential performance in a bitumen-aggregate mixture. While it is not being suggested that routine testing in a dynamic shear rheometer be conducted on bitumens coated on aggregates, the results do indicate a potential shortcoming of measurements made on a material other than that with which the bitumen would normally come into contact.

For specification purposes, testing on metal platens and use of the oven ageing procedures as recommended by the SHRP SUPERPAVE mixture design system (11) may well be appropriate but the work reported in this chapter suggests that some additional "aggregate susceptibility" tests should be considered. This suggestion is made notwithstanding the procedures for oven ageing bitumen-aggregate mixtures during the design of such mixtures.

An aggregate susceptibility test would alert mixture designers to combinations of bitumen and aggregate which are particularly "active" or "inactive" in terms of their mutual chemistry. Appropriate steps could then be taken to accommodate, avoid or encourage specific combinations.

Clearly further research is needed in light of the results reported here. This should include a wider range of bitumen and aggregate combinations and further investigation of the chemistry involved in the ageing process, concentrating particularly on the combinations which are very "active" or "inactive." It is also important to recognise that the more intimate contact between bitumen and aggregate in a paving mixture involves the fine aggregate and filler, whereas the tests reported here effectively only considered coarse aggregate. A limited number of performance-related (stiffness modulus) tests carried out on mixtures comprised of the same materials as those tested in the DSR indicated that there exists a potential link between the relative ageing characteristics measured in the DSR tests and those exhibited by paving mixtures. Further tests are needed, however, to establish a firm

9. Devore, J. and Peck, R., "Statistics, the exploration and analysis of data," West Publishing Company, St. Paul, Minnesota, 1986.

10. Edited by Harrigan, E.T., Leahy, R.B. and Youtcheff, J.S., "The SUPERPAVE Mix Design System Manual of Specifications, Test Methods, and Practices," *SHRP-A-379*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1994.

11. Milliken, G.A. and Johnson, D.E., "Analysis of messy data," Van Nostrand Reinhold Company, New York, 1984.

12. Carmer, S.G. and Swanson, M.R., "An evaluation of ten pairwise multiple comparison procedures by Monte Carlo methods," *Journal of American Statistical Association*, Vol. 68, pp 66-74, 1973.

believed that the high binder contents of the gap-graded mixtures relative to those for the dense-graded mixtures is the reason for this observed difference. Although the proposed method needs to be validated further using a wider range of materials and mixture types, sufficient evidence was provided to indicate that a standard procedure should be adopted for routine preparation of mixture specimens in the laboratory and that, at present, the proposed method is the best candidate for use in the UK.

The work presented in Chapter 4 was concerned primarily with the evaluation of the efficacy of the long-term ageing and water sensitivity protocols. This involved testing mixtures manufactured to specification as well as mixtures intentionally fabricated such that they did not satisfy specification criteria. Tests were principally carried out on wearing course mixtures as it was presumed that this layer would be most susceptible to environmental effects. However, some evaluations were carried out using base course materials as it was assumed that these would not necessarily be immune to the effects of the environment.

The evaluation of the long-term ageing protocol showed conclusively that the procedure is effective in simulating the effects of long-term ageing as measured by increase in stiffness modulus of the mixture. The evaluation also showed that the procedure is sufficiently sensitive to differences in volumetric proportions of binder and air voids in the mixture to be confidently used for comparative purposes. That is, sufficient evidence was provided to show that the procedure could be successfully used for assessing the relative performance of newly constructed bituminous paving mixtures with regard to ageing susceptibility. Although assessment of the change in performance was determined by change in stiffness modulus, other tests which measure mixture performance (e.g., tensile strength) could be used equally effectively.

Considerably more effort was afforded to evaluation of the water sensitivity protocol largely because procedural variations were investigated in addition to differences in volumetric proportions of binder and air voids in mixtures. The evaluations indicated that the procedure, as originally proposed (see Appendix A), was as effective in

These results indicate that tests conducted on bitumens coated on stainless steel (or, by inference, anodised aluminium) may neglect the effects imparted to the bitumen by the mineral aggregate which, in turn, may lead to inappropriate characterisation of the bitumen in the context of its potential performance in a bitumen-aggregate mixture. While it is not being suggested that routine testing in a dynamic shear rheometer be conducted on bitumens coated on aggregates, the results do indicate a potential shortcoming of measurements made on a material other than that with which the bitumen would normally come into contact.

For specification purposes, testing on metal platens may well be appropriate but the work reported in Chapter 5 suggests that some additional "aggregate susceptibility" tests should be considered. This suggestion is made notwithstanding the procedures for oven ageing bitumen-aggregate mixtures during the design of such mixtures.

An aggregate susceptibility test would alert mixture designers to combinations of bitumen and aggregate which are particularly "active" or "inactive" in terms of their mutual chemistry. Appropriate steps could then be taken to accommodate, avoid or encourage specific combinations.

6.2 CONCLUSIONS

The principal conclusions which can be drawn from the literature review summarised in Chapter 2 of this thesis include:

1) A bituminous paving mixture must provide adequate protection against detrimental environmental factors for it to survive its design life—inadequate protection encourages deterioration due to other factors such as cracking and permanent deformation.
2) The primary factors affecting the durability (or longevity) of bituminous paving mixtures, assuming they are constructed according to current specifications (which attempt to account for durability), are damage due to moisture and age hardening.
3) Water damage is generally manifested in loss of cohesion in the mixture and/or loss of adhesion between the bitumen and aggregate near the interface

shows promise but, again, validation has been limited and the procedure neglects the effect of the aggregate.

8) Several mixture tests have been developed to assess the ageing susceptibility of bituminous paving mixtures but most of these have been limited to a narrow range of materials and mixture types; in many cases on mixtures that in no way represent mixtures used in actual practice. The long-term oven ageing procedure developed for the SHRP asphalt programme, however, uses fundamental engineering properties (i.e., stiffness) to assess the ageing characteristics of materials fabricated to in-service specifications.

The principal conclusions which can be drawn from the experimental work presented in this thesis include:

1) Evidence was provided in Chapters 4 and 5 to show that mineral aggregate can affect the ageing characteristics of bitumens (as determined by indirect tensile stiffness modulus tests on mixtures before and after accelerated ageing and by rheological measurements carried out on bitumens in contact with mineral aggregate, also before and after accelerated ageing). The results support the finding from the literature review that the performance of bituminous mixtures is influenced by bitumen-aggregate interaction.
2) The water sensitivity test developed for the Bitutest project (Appendix A), which incorporates the strengths of the Lottman procedure and of the ECS method, was shown to be sufficiently sensitive to the variations in binder and void contents, the two mixture variables which most affect susceptibility to moisture damage and, thus, was shown to be a viable method.
3) The long-term ageing protocol developed for the Bitutest project (Appendix A) was shown to be effective in simulating the effects of long-term ageing as measured by increase in stiffness modulus of the mixture. Evaluation of the procedure showed that it is sufficiently sensitive to differences in volumetric proportions of binder and air voids in the mixture to be confidently used for comparative purposes.
4) Rheology tests on three straight-run bitumens and one polymer-modified bitumen coated on four different mineral aggregates and on stainless steel

3) The long-term ageing protocol contained in Appendix A is recommended for accelerated ageing of bituminous mixtures to be representative of changes occurring in the mixture over a long period. Coupled with tests which measure mixture performance (e.g., stiffness and/or strength), the long-term ageing protocol can be used to make relative comparisons of mixture performance with regard to long-term ageing characteristics; say, for example, to assess the performance of polymer-modified bitumens relative to straight-run bitumens.
4) The water sensitivity protocol contained in Appendix A is recommended for assessing the relative performance of bituminous mixtures with regard to their susceptibility to damage due to moisture. It can be confidently used to investigate the effects of variations in volumetric proportions of binder and air voids or to detect moisture sensitive aggregate types and could feasibly be used to assess the relative performance of binder types (including polymer-modified bitumens) and/or mixture types; say, for example, open-graded versus dense-graded mixtures.
5) Although evidence was provided which established that the long-term ageing and water sensitivity protocols contained in Appendix A pass the test of reasonableness for assessing the relative durability charactersitics of bituminous mixtures, further evaluation is necessary to establish if the methods are able to assess the actual durability charactersitics of in-service pavement mixtures. In other words, the methods need to be validated to gain confidence in using the methods for assessment of long-term durability. This would require monitoring the performance of in-service paving mixtures over a long period (say, 10 years or more). The results from fundamental mixture performance tests (e.g., dynamic stiffness modulus, indirect tensile stiffness modulus, etc.) could be used to monitor changes occurring to the paving mixtures which could be compared with those arising from the accelerated durability tests to establish correlations between the laboratory tests and field performance.
6) Although the novel experimental arrangement using the dynamic shear rheometer to test bitumens coated on mineral aggregate proved feasible, it is

Appendices

A Test Protocols and Practices

B Indirect Tensile Stiffness Modulus

C Rheology Test Data

Standard Practice for

Laboratory Preparation of Compacted Bituminous Mixtures

1 SCOPE

This standard practice is used to prepare compacted bituminous mixture specimens in the laboratory such that they are simulative of newly constructed bituminous pavements. The practice includes a short-term ageing procedure for continuously-graded mixtures which is intended to simulate the amount of hardening which occurs during the construction process (i.e., the hardening of the bitumen which occurs during mixing, storage, transport and laydown).

2 DEFINITIONS

2.1 *Ageing* refers to the hardening or embrittlement of the bitumen in bitumen-aggregate mixtures as a result of compositional changes in the bitumen due to many factors such as time, temperature, oxidation, steric hardening, ultraviolet radiation, etc.

2.2 *Short-term ageing* refers to the hardening or embrittlement of the bitumen which occurs during the construction of bituminous mixtures. Short-term ageing of the bitumen may occur during any or all of the following: mixing of the bitumen and aggregate, silo storage of the mixture, transport of the mixture or placement of the mixture on site.

3 APPARATUS

3.1 *Oven*—forced-draft oven which is thermostatically controlled and capable of being set to maintain any desired temperature from room temperature to 260 °C with an accuracy of ±1 °C or less.

3.2 *Mixing Apparatus*—any type of mechanical mixer which: 1) can be maintained at the required mixing temperature; 2) will provide a well coated, homogenous mixture of the required amount of bituminous mixture in the allowable time and 3) allows essentially all of the mixture to be recovered.

3.3 *Compactor*—any type of mechanical compactor which can compact the mixture to the desired density without causing damage to the aggregate. Suitable compactors include the Marshall hammer, Kango hammers, kneading compactors, rolling wheel compactors or gyratory compactors.

3.4 *Digital thermometer* capable of measuring temperatures from room temperature to 260 °C and having an accuracy of ±1 °C or less.

3.5 *Metal oven pans* of sufficient size to heat the required amount of aggregate for each mixture.

6.4 Preheat the metal oven pans to 135°C.

6.5 When the aggregate and bitumen are at the desired mixing temperature, place the aggregate in the mixer and dry mix it thoroughly.

6.6 Form a crater in the blended aggregate and add the required mass of bitumen.

6.7 Mix the bitumen and aggregate as quickly and thoroughly as possible to yield a mixture having a uniform distribution of bitumen throughout. For most mixtures 2 to 4 minutes, depending on mixer type, should be sufficient for thorough mixing. It is recommended that a standard mixing time (e.g., 4 minutes) be established for all mixtures.

6.8 For continuously-graded mixtures (e.g., dense bitumen macadams), place the mixture in a metal oven pan preheated to 135°C. Spread the mixture in the pan to an even depth approximately equal to the maximum aggregate size (e.g., for a mixture with a maximum aggregate size of 20 mm, spread the mixture to a depth of approximately 20 mm). If the desired mixing temperature is greater than 135°C, allow the mixture to cool to 135°C, then place it in a forced-draft oven at 135±1°C for 2 hours ±5 minutes. If the compaction temperature for the mixture is greater than 135°C, then cure the mixture at the compaction temperature for 2 hours ±5 minutes. Thoroughly stir the mixture using the spatula or spoon after 1 hour.

NOTE: At present it is recommended that gap-graded mixtures such as hot rolled asphalt wearing course mixtures be compacted without subjecting the mixture to the 2 hour cure at 135 °C.

6.9 Ensure the temperature of the mixture is at the desired compaction temperature (i.e., allow the mixture to cool if the compaction temperature is less than 135°C) and compact the mixture to the desired density or in accordance with the established procedures for the particular compactor being used.

NOTE: The minimum time necessary should be used to heat the mixture if heating is required.

6.10 After compaction allow the compacted mixture specimen to cool to room temperature prior to extrusion or removal from the compaction mould.

Standard Practice for

Long-Term Oven Ageing of Compacted Bituminous Mixtures

1 SCOPE

This standard practice is used to simulate the long-term ageing of compacted bituminous mixtures. Long-term ageing considers the hardening of the bitumen in the mixture subsequent to construction. The practice should result in ageing representative of 15 years or more in service for dense-graded mixtures.

2 DEFINITIONS

2.1 *Ageing* refers to the hardening or embrittlement of the bitumen in bitumen-aggregate mixtures as a result of compositional changes in the bitumen due to many factors such as time, temperature, oxidation, steric hardening, ultraviolet radiation, etc.

2.2 *Long-term ageing* refers to the hardening or embrittlement of the bitumen in bitumen-aggregate mixtures subsequent to construction of the pavement layer incorporating the bituminous mixture.

3 APPARATUS

3.1 *Oven*—forced-draft oven which is thermostatically controlled and capable of being set to maintain any desired temperature from room temperature to 260°C with an accuracy of ±1°C or less.

3.2 *Digital thermometer* capable of measuring temperatures from room temperature to 260°C and having an accuracy of ±1°C or less.

4 SAMPLING

Plant-mixed materials shall be sampled in accordance with relevant local standards (e.g., BS 598 : Part 100 : 1987).

5 PREPARATION

5.1 Laboratory-prepared mixtures shall be fabricated in accordance with the Standard Practice for Laboratory Preparation of Compacted Bituminous Mixtures or other suitable method such as BS 598 : Part 107 : 1990.

5.2 Plant-mixed materials shall be heated to a temperature which corresponds to a kinematic viscosity of 50±1 Poise for the bitumen in the mixture and compacted in accordance with the Standard Practice for Laboratory Preparation of Compacted Bituminous Mixtures or other suitable method such as BS 598 : Part 107 : 1990.

Test Method for

Measurement of the Water Sensitivity of Compacted Bituminous Mixtures

1 SCOPE

This test method determines the water sensitivity of compacted bituminous mixtures under warm and cold climatic conditions. This method is applicable to laboratory-moulded specimens and core specimens obtained from existing roads.

2 DEFINITIONS

2.1 *Water sensitivity* is the quality or state of a compacted bituminous mixture being susceptible to damage due to moisture.

2.2 The *unconditioned stiffness* is defined as the indirect tensile stiffness modulus (ITSM) of the compacted mixture as determined in the Nottingham Asphalt Tester (NAT) prior to water and thermal conditioning.

2.3 The *conditioned stiffness* is defined as the indirect tensile stiffness modulus of the compacted mixture as determined by the NAT after the compacted mixture has been subjected to one or more cycles of water and thermal conditioning.

2.4 The *stiffness ratio* is defined as the ratio of conditioned stiffness to unconditioned stiffness.

3 APPARATUS

3.1 *Vacuum desiccator* capable of accommodating at least one compacted bituminous mixture specimen (102mm in diameter by approximately 64mm in height) and capable of withstanding a negative pressure (i.e., vacuum) of 760mm Hg (1 atmosphere).

3.2 *Balance* with sufficient capacity and accuracy to 1g.

3.3 *Water baths* of suitable size to accommodate at least one compacted bituminous mixture specimen (102mm in diameter by approximately 64mm in height) and thermostatically controlled such that temperatures of 5, 20 and 60°C can be maintained. It is preferrable that three baths (one for each temperature) are used and that each bath is of sufficient size such that it is capable of accommodating numerous specimens at one time.

3.4 *Vacuum pump* capable of evacuating air from the vacuum desiccator to a negative pressure of at least 510mm Hg (⅔ atmospheres).

6.4 Remove the specimen from the vacuum desiccator, remove any water on its surface using a towel and determine its wet mass. Designate this as M_w. Determine the percent saturation (S) as follows:

$$S = \frac{M_w - M_d}{\frac{M_d}{G_{mb}} - \frac{M_d}{G_{mm}}} \times 100$$

where:

S = percent saturation,
M_d = mass of dry specimen, g,
M_w = mass of wet specimen, g,
G_{mb} = bulk specific gravity and
G_{mm} = maximum specific gravity

6.5 Place the specimen in a hot (60°C) water bath for 6 hours.

6.6 Remove the specimen from the hot bath and immediately place it in a cold (5°C) water bath for 16±1 hours.

6.7 Remove the specimen from the cold bath and immediately place it in a 20°C water bath (i.e., a water bath having a temperature equal to the stiffness test temperature) for 2 hours.

6.8 Remove the specimen from the 20°C water bath and determine the conditioned stiffness for the first conditioning cycle. Designate this as $ITSM_{C1}$. NOTE: Ensure that the temperature of the test specimen is equal to 20°C prior to performing the stiffness test.

6.9 Repeat Steps 6.5 through 6.7. Determine the conditioned stiffness of the specimen for the second conditioning cycle. Designate this as $ITSM_{C2}$.

6.10 Repeat Steps 6.5 through 6.7. Determine the conditioned stiffness of the specimen for the third conditioning cycle. Designate this as $ITSM_{C3}$.

6.11 The specimen could be subjected to further conditioning cycles (i.e., Steps 6.5 through 6.7) and tested to determine the stiffness after each cycle (designate the stiffnesses as $ITSM_{Ci}$; i = 4, 5, 6, ...).

6.12 Determine the stiffness ratio (ITSM Ratio) for the specimen as follows:

$$ITSM\ Ratio = \frac{ITSM_{Ci}}{ITSM_U};\ i = 1,\ 2,\ 3,\ ...$$

B Indirect Tensile Stiffness Modulus

INTRODUCTION

Various methods have been employed to measure the stiffness of bituminous mixtures. Conventional techniques have principally included uniaxial and triaxial compression and/or tension tests and flexural beam tests. However, in the late 1960s Hudson et al (1) developed an indirect tension test based on the Brazilian test (2) used to measure the tensile strength of concrete. In the early 1980s the test was accepted as a standard method by the American Society for Testing and Materials (3). Cooper and Brown (4) introduced the method to the UK with the development of the Nottingham Asphalt Tester which has subsequently gained widespread use in much of Europe and has become a British Standards Institution Draft for Development (5). This appendix presents a brief overview of the theory supporting the indirect tensile stiffness modulus (ITSM) test for bituminous mixtures, the principal factors affecting the results and advantages and disadvantages of the test.

DETERMINATION OF STIFFNESS BY INDIRECT TENSION

Theory

Hadley et al (6) and Anagnos and Kennedy (7) developed equations that permit the calculation of the tensile strength, tensile strain, modulus of elasticity and Poisson's ratio for a homogenous, isotropic, linear elastic cylinder subjected to a diametrically applied static "line" load. The equations were extended to allow calculation of the "instantaneous resilient Poisson's ratio" and the "instantaneous resilient modulus of elasticity" for a cylinder subjected to repeated loading (7) as follows:

$$\nu = \frac{\dfrac{\delta_v}{\delta_h} \cdot c_1 + c_2}{\dfrac{\delta_v}{\delta_h} \cdot c_3 + c_4} \qquad \textbf{B.1}$$

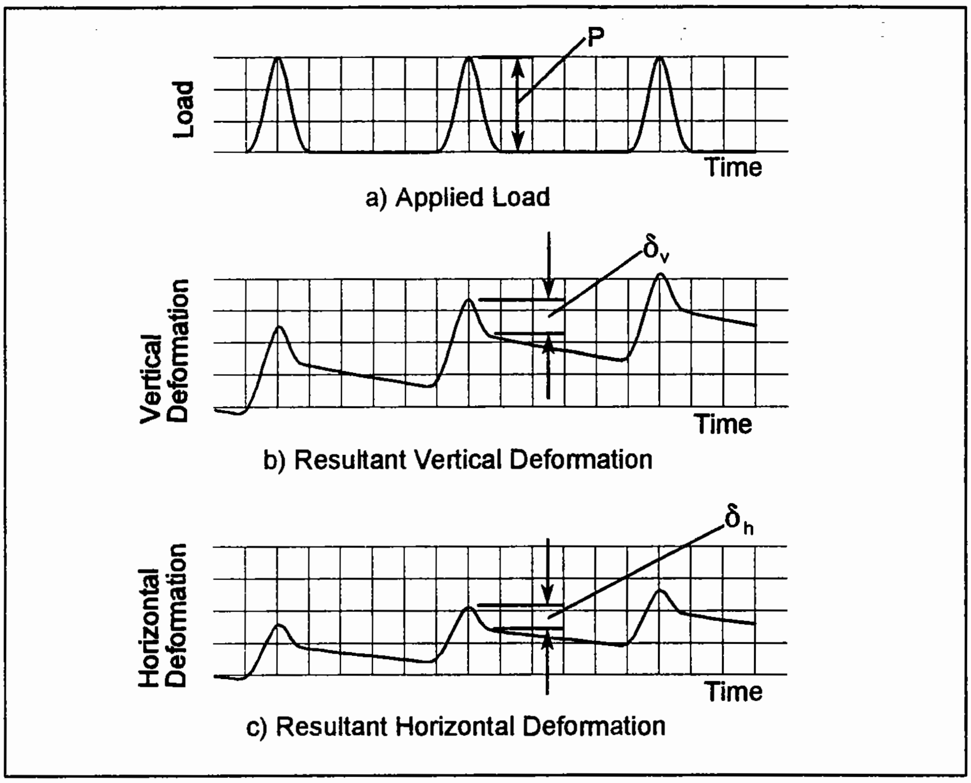

Figure B.1. Typical Load and Deformation Relationships for the Repeated Load Indirect Tensile Test.

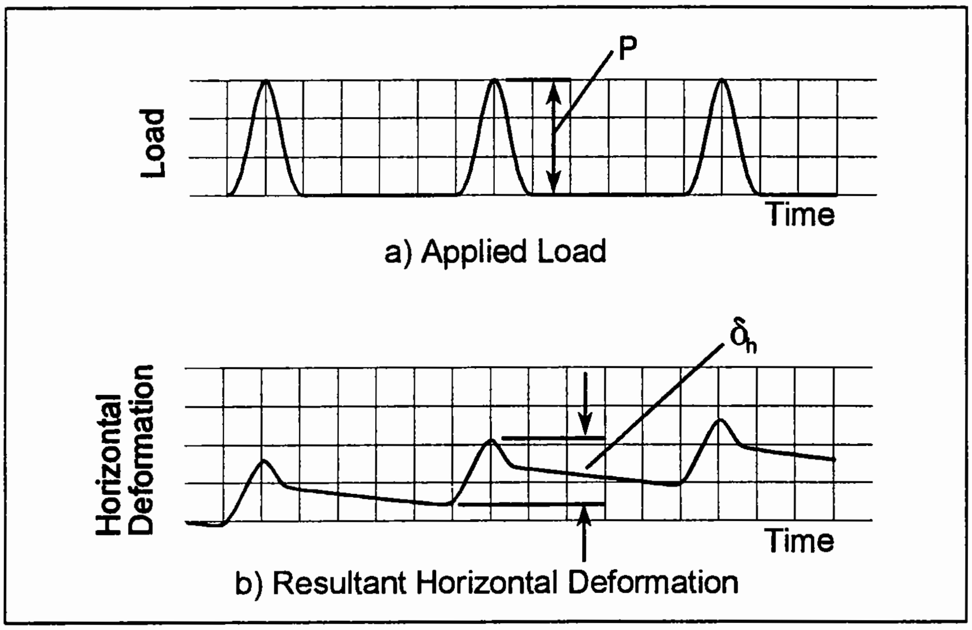

Figure B.2. Typical Load and Deformation Relationships for the Indirect Tensile Stiffness Modulus (ITSM) Test.

Table B.1. General Effect of the Principal Factors Affecting the ITSM.

Factor	General Effect on ITSM
Specimen temperature during test	High temperature ➔ low stiffness modulus Low temperature ➔ high stiffness modulus
Loading frequency	Low frequency ➔ low stiffness modulus High frequency ➔ high stiffness modulus
Stress amplitude	High stress ➔ low stiffness modulus Low stress ➔ high stiffness modulus
Poisson's ratio (assumed)	Low value ➔ low stiffness modulus High value ➔ high stiffness modulus
Bitumen grade (for a particular mixture type)	High penetration bitumen ➔ low stiffness modulus Low penetration bitumen ➔ high stiffness modulus
Bitumen content (for a particular mixture type)	Highest stiffness modulus is generally achieved at or very near the optimum binder content for compacted aggregate density (BS 598, 12)
Bitumen modifiers	Use of modified bitumen in mixtures can significantly increase or reduce the stiffness modulus of the mixture and the magnitude of the effect greatly depends on the type of modifier. It should be noted, however, that modifiers are generally used to improve characteristics of the mixture other than its stiffness modulus.
Void content (for a particular mixture type)	High air voids ➔ low stiffness modulus Low air voids ➔ high stiffness modulus (for some mixtures very low air voids can result in a reduction in stiffness modulus)

stress amplitude. The differences in stiffness modulus amongst the various lines are due to variations in void content.

Figure B.5 shows the variation in stiffness modulus for a range of assumed Poisson's ratios with all other variables being held constant. The plot indicates that a moderate error in the stiffness modulus occurs if the value of Poisson's ratio is assumed incorrectly. Although it would seem logical to assume different Poisson's ratios for different conditions (e.g., mixtures types, bitumen types, temperatures, etc.), there is currently insufficient information to make specific recommendations for appropriate values. Section 7.3 provides further details regarding Poisson's ratio and suggests that a value of 0.35 be used for all testing conditions.

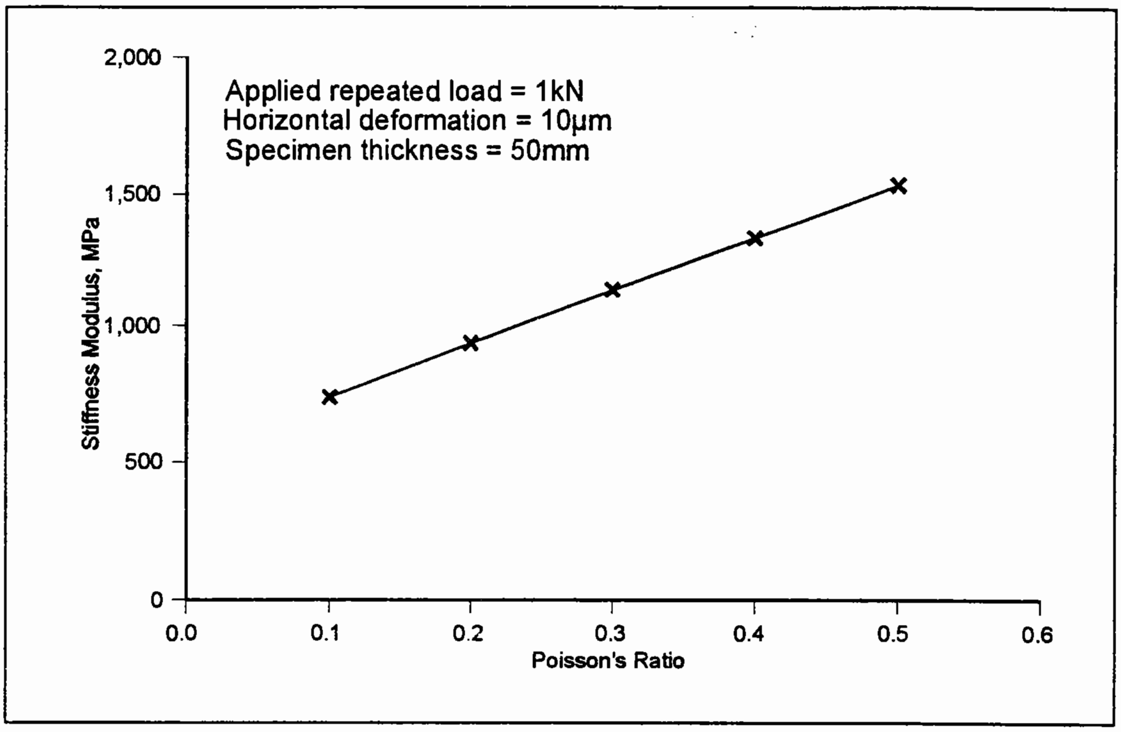

Figure B.5. Hypothetical Stiffness Values Showing Effect of Poisson's Ratio on the ITSM.

A variation in the void content of a particular mixture results in a variation in stiffness modulus. Figure B.6 shows typical ITSM test results for three dense macadams which indicate that the stiffness modulus decreases as the air void content increases.

The effect of bitumen content on the stiffness modulus of typical 30/14 HRA mixtures is shown in Figure B.7. The data indicates that the maximum stiffness modulus occurs very near to the binder content corresponding to the maximum compacted aggregate density.

ADVANTAGES AND DISADVANTAGES

Advantages

Indirect tension testing has several advantages over other methods (e.g., direct tension and/or compression or bending beam) to determine the stiffness of bituminous materials. The principal advantages include:

1) The test is relatively simple, quick to conduct (i.e., user-friendly) and effectively non-destructive when testing conditions ensure essentially elastic response.
2) Tests are conducted on moulded specimens or cores eliminating difficult specimen manufacture.
3) Tests can be conducted on "thin" specimens thereby allowing cores from surfacing layers, typically 50mm thick, to be tested.
4) A biaxial state of stress exists in the specimen during the test which may better represent field conditions than the stress conditions found in flexure tests.
5) The test can be effectively used for the purposes of comparing mixture variables such as constituent materials and volumetric proportions as well as changes due to the effects of ageing and water damage.
6) The test can, in principle, be used for the design of bituminous mixtures.
7) The equipment used for the test is relatively inexpensive and can be used for other types of tests such as fatigue and permanent deformation.

Disadvantages

The principal disadvantages of indirect tension testing of bituminous mixtures, relative to other methods, include:

1) The method relies on theoretical analysis using elastic theory. Consequently, tests need to be carried out in such a way as to make this assumption reasonable (e.g., moderate temperatures and reasonably fast loading times).
2) Although Poisson's ratio is necessary for the determination of the stiffness modulus in indirect tension testing, it cannot be accurately determined in such tests and must be assumed. For this reason, indirect tension tests may be less reliable than direct tension/compression or flexural tests.
3) The absence of stress reversal during testing allows the accumulation of plastic (permanent) deformation, particularly at high test temperatures.
4) The stiffness modulus cannot be accurately measured at relatively high temperatures (e.g., 40°C) owing to the large permanent deformations which occur during testing at such temperatures.

C Rheology Test Data

Dynamic shear rheometers (DSRs) can be used in a variety of ways to measure rheological properties of bitumens. Ordinarily, measurements in DSRs are conducted on neat bitumens sandwiched between metal platens (generally stainless steel and/or anodised aluminium) separated by a gap of 1 to 2mm which defines the thickness of bitumen under test. For example, the SHRP SUPERPAVE mixture design system (1) specifies minimum values for the loss modulus ($G^*\sin\delta$) and the complex modulus divided by the sine of the phase angle ($G^*/\sin\delta$) at a single frequency and temperature. The test frequency is 10 rad/s while the test temperature is selected according the climatic conditions in which the mixture will serve and ranges from 4 to 40°C for the $G^*\sin\delta$ specification and from 46 to 82°C for the $G^*/\sin\delta$ specification. Tests are carried out using a gap setting of 1mm if a 25mm diameter spindle is used or 2mm if an 8mm diameter spindle is used. Although use of the DSR in this way is useful for specification purposes, it does not utilise the full capabilities of the equipment.

Another use of dynamic shear rheometers is to measure the rheological properties of bitumens over a wide range of frequencies to determine the time (frequency) dependency of the modulus of bitumens. This is typically done at several temperatures such that master curves can be developed using time-temperature superposition which describe the thermorheologically simple linear viscoelastic behaviour of bitumen over a very wide range of frequencies. The reader is referred to Anderson et al (2) for construction of these master curves as well as various models developed to describe the curves.

The experimental work contained in this thesis utilised the capabilities of the DSR to measure the rheological properties of bitumens in contact with various substrate materials (principally mineral aggregates). Dynamic shear modulus tests were conducted at 25°C using a shear strain amplitude of 1% (controlled-strain) in a Bohlin Model DSR50 dynamic shear rheometer on the bitumens before and after accelerated ageing. Accelerated ageing consisted of exposure to air at 85°C in a forced-draft oven in the absence of light. All tests were conducted using the 8mm diameter parallel plate spindle with a gap setting of 25μm over a frequency range of 0.01 to 10Hz.

A typical output from a DSR test is shown in Figure C.1. The output provides values for the phase angle, dynamic viscosity, complex modulus (G*), storage modulus (G′), loss modulus (G″), shear strain and shear stress for each frequency of oscillation. The gap setting is shown incorrectly as 0.03mm (due to the software rounding 0.025 to 0.03). The shear stresses (τ) and shear strains (γ) are calculated as follows:

$$\tau = \frac{2T}{\pi r^3} \qquad \textbf{C.1}$$

and

$$\gamma = \frac{\theta r}{h} \qquad \textbf{C.2}$$

where:

τ = shear stress, Pa

T = applied torque, Nm

π = 3.14159265 to eight decimal places

r = radius of spindle, m

γ = shear strain, m/m

θ = deflection angle of the spindle, radians

h = bitumen thickness (gap setting), m

Although torque values are not provided in the summary output, they can be calculated from Equation C.1. For example, the maximum torques applied to the spindle (to apply the maximum shear stresses) for the data shown in Figure C.1

Table C.1. Summary of Rheology Test Results for Bitumen A on Aggregate A.

Sample AA001	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	83.69	17.10	1.08	88.30	70.30	4.42	81.02	151.00	9.59
0.02	83.51	16.90	2.13	83.29	68.50	8.67	76.56	141.00	18.30
0.05	80.25	16.20	5.18	78.48	60.80	19.50	70.44	119.00	39.70
0.1	85.83	13.40	8.45	73.58	56.10	36.70	68.71	101.00	67.80
0.2	79.77	13.40	17.00	72.83	49.30	64.80	63.80	82.40	115.00
0.5	69.43	9.43	31.60	70.50	41.10	137.00	61.62	61.80	221.00
1	76.72	10.90	70.70	67.69	34.20	232.00	58.96	48.10	352.00
2	77.96	9.97	128.00	64.59	28.00	390.00	55.62	35.80	545.00
5	72.84	8.63	284.00	59.13	20.20	739.00	51.63	23.60	947.00
10	68.98	7.71	519.00	55.56	15.20	1150.00	47.48	16.20	1380.00
Sample AA002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	85.47	16.30	1.03	75.81	151.00	9.78	76.79	157.00	10.10
0.02	84.07	15.00	1.90	59.76	88.90	12.90	74.34	143.00	18.70
0.05	82.60	14.00	4.43	63.66	73.30	25.70	70.82	114.00	37.80
0.1	80.83	13.20	8.39	63.71	60.10	42.10	66.78	100.00	68.30
0.2	80.60	12.80	16.30	66.11	49.70	68.30	65.24	83.50	116.00
0.5	81.04	11.70	37.30	65.55	39.00	135.00	62.90	63.20	223.00
1	77.39	10.60	68.30	63.96	31.50	220.00	59.54	49.50	361.00
2	77.08	9.82	127.00	62.84	25.80	364.00	55.76	37.00	562.00
5	73.46	8.38	275.00	60.15	19.20	694.00	51.36	24.20	973.00
10	70.76	7.23	481.00	56.70	14.50	1090.00	47.83	16.60	1410.00
Sample AA007	Unaged			Aged 48 hours			Aged 120 hours		
0.01	82.27	5.66	0.359	81.99	35.30	2.24	80.47	93.00	5.93
0.02	81.69	5.35	0.679	79.97	31.60	4.03	77.90	86.50	11.10
0.05	80.98	4.87	1.55	78.16	28.70	9.20	75.25	76.90	25.00
0.1	80.15	4.60	2.93	76.61	26.70	17.30	73.15	65.50	43.00
0.2	78.86	4.26	5.45	73.88	24.90	32.50	70.74	58.00	77.10
0.5	77.56	3.77	12.10	71.57	20.90	69.10	67.76	45.90	156.00
1	76.12	3.41	22.10	69.49	17.90	120.00	65.76	37.70	260.00
2	74.75	3.09	40.30	67.10	14.90	204.00	62.01	30.10	429.00
5	72.04	2.54	83.80	63.20	11.30	396.00	57.58	21.50	800.00
10	69.75	2.14	143.00	59.62	8.67	632.00	52.98	15.40	1210.00

Table C.3. Summary of Rheology Test Results for Bitumen A on Aggregate D.

Sample DA002	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	86.87	20.00	1.26	83.67	55.10	3.48	72.93	466.00	30.60
0.02	84.91	18.90	2.38	80.79	55.10	7.01	70.10	387.00	51.80
0.05	83.06	18.40	5.81	76.22	51.10	16.50	66.02	319.00	110.00
0.1	81.94	17.90	11.40	75.27	45.00	29.20	64.03	262.00	183.00
0.2	81.53	16.60	21.10	70.79	38.90	51.70	60.40	203.00	294.00
0.5	77.45	15.00	48.20	69.77	33.00	111.00	57.46	148.00	551.00
1	78.76	13.70	87.70	67.47	27.40	186.00	54.11	110.00	853.00
2	76.12	12.40	161.00	64.48	22.20	309.00	48.80	77.30	1290.00
5	73.57	10.60	346.00	60.72	16.60	598.00	43.76	45.50	2070.00
10	70.50	9.01	600.00	57.05	12.60	945.00	39.07	28.30	2820.00
Sample DA003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	71.46	18.30	1.21	85.10	50.80	3.20	74.75	316.00	20.60
0.02	68.29	15.00	2.03	79.99	50.80	6.49	70.98	279.00	37.20
0.05	74.15	12.30	4.01	77.12	45.60	14.70	64.51	223.00	77.50
0.1	75.01	11.20	7.30	75.27	41.80	27.20	64.55	179.00	125.00
0.2	76.95	10.40	13.40	72.79	35.60	46.80	60.58	138.00	200.00
0.5	76.97	9.35	30.10	69.29	30.10	101.00	58.31	98.80	365.00
1	76.31	8.61	55.70	67.36	25.20	172.00	54.61	74.00	571.00
2	75.37	7.79	101.00	66.04	20.90	288.00	51.80	53.80	861.00
5	73.93	6.66	218.00	61.64	15.60	557.00	46.70	33.20	1430.00
10	71.42	5.69	377.00	57.69	11.70	868.00	42.35	21.40	2000.00

Table C.5. Summary of Rheology Test Results for Bitumen A on "Aggregate" F.

Sample FA001	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	85.59	14.60	0.921	44.54	116.00	10.40	81.94	210.00	13.30
0.02	84.11	15.10	1.91	53.13	111.00	17.50	71.39	212.00	28.10
0.05	81.92	14.00	4.43	61.06	92.20	33.10	66.95	172.00	58.80
0.1	81.41	13.00	8.25	65.14	82.70	57.30	63.93	136.00	94.90
0.2	79.12	11.90	15.20	65.15	70.90	98.10	60.74	105.00	152.00
0.5	79.80	10.80	34.50	62.36	52.80	187.00	58.51	76.80	283.00
1	77.93	10.30	66.30	62.86	42.50	300.00	55.46	57.40	438.00
2	76.29	9.11	118.00	56.79	31.90	480.00	52.14	41.60	663.00
5	73.35	7.69	252.00	51.44	20.20	813.00	47.28	25.80	1100.00
10	71.25	6.72	446.00	48.48	13.80	1160.00	43.29	16.80	1540.00
Sample FA002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	87.45	15.20	0.959	84.14	65.20	4.12	78.14	257.00	16.50
0.02	84.15	15.40	1.94	84.31	41.90	5.29	73.67	227.00	29.70
0.05	82.32	14.60	4.62	75.30	54.90	17.80	66.85	183.00	62.50
0.1	80.73	13.80	8.79	73.99	50.30	32.90	62.94	146.00	103.00
0.2	79.88	13.20	16.80	71.65	44.50	59.00	60.13	115.00	167.00
0.5	79.19	11.70	37.50	69.46	36.80	124.00	56.25	79.00	298.00
1	79.61	10.80	68.90	66.80	30.40	208.00	51.11	56.20	454.00
2	76.22	9.56	124.00	64.41	25.10	350.00	49.99	42.00	688.00
5	72.82	8.10	266.00	60.04	18.40	667.00	46.12	26.20	1140.00
10	70.33	6.87	458.00	56.10	13.70	1040.00	42.53	16.90	1580.00
Sample FA003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	85.84	13.60	0.856	80.98	67.30	4.28	77.96	324.00	20.80
0.02	84.36	12.60	1.59	77.33	62.90	8.11	69.29	276.00	37.10
0.05	82.36	11.80	3.73	76.53	55.60	18.00	65.19	206.00	71.50
0.1	80.67	11.20	7.12	73.36	48.70	31.90	63.92	168.00	117.00
0.2	81.93	10.40	13.10	72.17	41.50	54.80	61.06	131.00	188.00
0.5	79.67	9.51	30.40	70.45	34.60	115.00	57.85	95.70	355.00
1	77.64	8.58	55.20	67.78	28.60	194.00	57.36	72.40	540.00
2	76.00	7.78	101.00	65.53	23.70	328.00	51.17	52.80	852.00
5	73.19	6.70	220.00	60.27	17.00	616.00	47.14	32.40	1390.00
10	71.22	5.74	381.00	56.58	12.80	960.00	43.11	21.10	1940.00

Table C.7. Summary of Rheology Test Results for Bitumen B on Aggregate C.

Sample CB001	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	71.59	390.00	25.80	58.90	1740.00	127.00	55.90	3120.00	237.00
0.02	65.97	334.00	45.90	56.02	1280.00	194.00	49.07	1980.00	329.00
0.05	64.89	264.00	91.70	51.32	824.00	332.00	47.03	1290.00	553.00
0.1	62.49	216.00	153.00	48.81	582.00	486.00	44.69	894.00	798.00
0.2	60.60	168.00	242.00	45.97	408.00	713.00	41.52	589.00	1120.00
0.5	57.87	119.00	440.00	42.21	243.00	1140.00	35.85	306.00	1640.00
1	55.53	89.00	678.00	38.82	157.00	1570.00	31.22	183.00	2220.00
2	48.22	60.10	1010.00	35.45	96.80	2100.00	29.98	107.00	2680.00
5	44.90	37.80	1680.00	31.57	47.70	2860.00	25.02	47.80	3550.00
10	40.29	24.00	2340.00	28.01	26.30	3520.00	22.32	25.50	4220.00
Sample CB002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	66.98	630.00	43.00	59.47	1410.00	103.00	57.66	3130.00	233.00
0.02	64.00	508.00	71.00	53.22	1070.00	167.00	51.87	2270.00	363.00
0.05	60.52	383.00	138.00	51.59	701.00	281.00	45.02	1330.00	592.00
0.1	59.46	301.00	220.00	49.02	537.00	447.00	43.00	964.00	888.00
0.2	57.15	244.00	365.00	46.61	368.00	636.00	39.00	627.00	1250.00
0.5	55.47	165.00	629.00	41.66	208.00	985.00	36.08	338.00	1800.00
1	49.84	115.00	945.00	37.40	129.00	1330.00	32.28	195.00	2300.00
2	47.40	82.10	1400.00	34.72	81.10	1790.00	28.02	107.00	2880.00
5	41.16	46.40	2210.00	31.63	41.90	2510.00	23.77	48.20	3760.00
10	36.17	27.70	2950.00	28.08	23.40	3120.00	21.17	25.00	4350.00
Sample CB008	Unaged			Aged 48 hours			Aged 120 hours		
0.01	73.59	281.00	18.40	61.38	2110.00	151.00	59.38	2170.00	159.00
0.02	67.85	235.00	31.90	55.47	1470.00	224.00	50.42	1510.00	246.00
0.05	65.37	184.00	63.60	51.89	972.00	388.00	47.81	952.00	404.00
0.1	62.23	151.00	107.00	48.00	687.00	581.00	45.40	665.00	587.00
0.2	61.20	120.00	172.00	45.75	486.00	853.00	42.65	449.00	834.00
0.5	57.39	84.30	314.00	41.41	284.00	1350.00	38.24	248.00	1260.00
1	55.37	64.00	489.00	37.83	174.00	1780.00	36.23	159.00	1700.00
2	51.61	47.50	761.00	34.14	107.00	2390.00	31.77	90.70	2160.00
5	47.31	29.30	1250.00	28.99	50.80	3290.00	26.95	41.90	2900.00
10	43.12	19.20	1760.00	25.40	27.30	3990.00	23.86	22.40	3480.00

Table C.9. Summary of Rheology Test Results for Bitumen B on Aggregate E.

Sample EB001	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	61.47	452.00	32.30	59.90	966.00	70.10	46.19	3690.00	321.00
0.02	59.18	334.00	48.90	58.19	741.00	110.00	43.21	2360.00	434.00
0.05	60.16	268.00	96.90	52.10	501.00	199.00	38.42	1330.00	673.00
0.1	59.83	217.00	158.00	50.10	361.00	296.00	37.41	870.00	900.00
0.2	57.99	162.00	240.00	48.15	266.00	449.00	34.06	523.00	1170.00
0.5	54.35	115.00	446.00	41.34	149.00	708.00	30.08	262.00	1640.00
1	51.99	85.60	682.00	41.86	102.00	962.00	28.62	154.00	2020.00
2	49.89	60.40	992.00	37.47	64.10	1320.00	25.50	85.40	2490.00
5	44.84	36.60	1630.00	34.18	34.00	1900.00	22.22	37.60	3130.00
10	40.72	23.30	2240.00	31.50	20.10	2420.00	20.62	20.40	3630.00
Sample EB003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	70.66	372.00	24.80	62.76	1050.00	74.40	53.63	3840.00	300.00
0.02	66.23	301.00	41.30	59.57	765.00	111.00	47.37	2560.00	438.00
0.05	62.58	227.00	80.50	51.16	468.00	189.00	41.27	1430.00	683.00
0.1	59.70	175.00	127.00	50.09	365.00	299.00	37.59	897.00	923.00
0.2	59.16	135.00	198.00	47.07	257.00	442.00	34.99	562.00	1230.00
0.5	55.95	96.10	364.00	42.30	150.00	699.00	31.79	288.00	1720.00
1	49.78	64.60	532.00	40.30	101.00	977.00	28.76	163.00	2130.00
2	49.54	49.10	810.00	37.60	64.10	1320.00	27.02	97.70	2700.00
5	44.35	29.10	1310.00	33.78	33.60	1900.00	23.21	42.20	3360.00
10	40.44	18.60	1800.00	30.75	19.40	2390.00	20.80	22.10	3900.00

Table C.11. Summary of Rheology Test Results for Bitumen C on Aggregate A.

Sample AC001	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	85.27	15.70	0.991	76.81	136.00	8.76	72.55	432.00	28.50
0.02	82.17	14.80	1.88	74.46	117.00	15.30	69.10	351.00	47.20
0.05	80.69	13.60	4.34	72.57	98.20	32.30	65.45	269.00	92.90
0.1	80.30	12.40	7.92	70.15	84.00	56.10	63.34	220.00	154.00
0.2	78.65	11.50	14.70	67.93	71.30	96.70	61.14	176.00	252.00
0.5	77.13	10.10	32.60	65.82	56.80	196.00	57.94	126.00	468.00
1	76.03	9.10	58.90	63.68	46.10	323.00	55.37	94.90	724.00
2	74.17	7.98	104.00	60.94	36.40	524.00	51.60	68.90	1100.00
5	71.79	6.61	219.00	55.92	25.30	960.00	46.32	41.90	1820.00
10	69.16	5.55	373.00	51.68	18.20	1460.00	41.52	26.60	2520.00
Sample AC002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	86.27	12.30	0.774	78.87	152.00	9.73	74.24	391.00	25.50
0.02	84.36	11.90	1.50	74.85	126.00	16.40	68.58	339.00	45.80
0.05	82.50	11.30	3.58	72.74	116.00	38.10	65.79	252.00	87.00
0.1	80.49	10.20	6.53	70.87	95.70	63.70	64.70	207.00	144.00
0.2	79.80	9.50	12.10	68.44	82.50	111.00	61.98	168.00	240.00
0.5	79.10	8.71	27.90	66.22	64.60	222.00	58.69	121.00	446.00
1	77.36	7.89	50.80	64.60	53.80	374.00	56.05	92.20	698.00
2	75.36	7.00	90.90	61.41	41.80	598.00	52.40	67.70	1070.00
5	73.28	6.02	198.00	56.26	29.20	1100.00	46.56	41.60	1800.00
10	71.00	5.17	344.00	51.94	21.00	1670.00	41.73	26.80	2530.00
Sample AC003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	85.19	26.10	1.64	75.08	171.00	11.10	73.50	373.00	24.50
0.02	82.49	22.60	2.86	73.55	164.00	21.50	69.10	303.00	40.80
0.05	80.84	21.30	6.77	70.61	137.00	45.70	65.94	246.00	84.60
0.1	79.95	20.10	12.80	69.55	110.00	73.70	64.47	192.00	134.00
0.2	77.83	18.00	23.10	68.12	89.80	122.00	62.00	152.00	216.00
0.5	76.70	15.90	51.20	65.18	69.20	240.00	58.28	108.00	398.00
1	75.76	14.20	92.00	62.99	55.80	393.00	55.97	82.00	622.00
2	73.78	12.50	164.00	60.35	44.00	637.00	52.63	60.00	949.00
5	70.70	10.40	346.00	55.37	30.10	1150.00	47.42	37.20	1590.00
10	67.77	8.66	588.00	51.01	21.30	1720.00	42.98	24.20	2230.00

Table C.12. Summary of Rheology Test Results for Bitumen C on Aggregate C.

Sample CC002	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	86.10	16.70	1.05	72.34	161.00	10.60	64.21	341.00	23.80
0.02	84.55	16.10	2.03	72.18	146.00	19.20	64.06	299.00	41.80
0.05	83.20	15.20	4.80	69.92	125.00	41.80	63.08	220.00	77.40
0.1	81.70	14.10	8.95	70.09	107.00	71.30	62.65	179.00	126.00
0.2	80.12	12.80	16.30	67.97	89.50	121.00	61.24	140.00	201.00
0.5	78.35	11.40	36.50	65.80	69.10	238.00	58.11	100.00	371.00
1	76.15	10.40	67.40	63.49	56.40	396.00	56.28	76.30	576.00
2	75.09	9.48	123.00	60.35	44.50	644.00	52.37	55.30	877.00
5	72.93	8.20	269.00	55.61	30.70	1170.00	48.13	35.20	1480.00
10	70.04	7.01	469.00	50.68	21.60	1760.00	43.56	23.00	2090.00
Sample CC003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	85.54	11.50	0.727	75.29	174.00	11.30	69.94	508.00	34.00
0.02	84.44	11.20	1.41	73.52	159.00	20.80	68.58	367.00	49.50
0.05	81.67	10.60	3.36	70.89	130.00	43.10	63.93	257.00	89.70
0.1	80.58	9.75	6.21	69.16	108.00	72.70	62.30	209.00	148.00
0.2	79.17	9.12	11.70	66.69	90.60	124.00	61.39	168.00	240.00
0.5	77.70	7.99	25.70	64.54	73.00	254.00	57.69	117.00	436.00
1	77.97	7.49	48.10	62.53	58.40	414.00	53.93	87.00	677.00
2	76.02	6.72	87.10	59.63	46.50	677.00	51.17	62.60	1010.00
5	72.95	5.56	183.00	54.56	31.50	1220.00	46.44	38.20	1660.00
10	70.87	4.77	317.00	50.10	22.10	1810.00	41.96	24.50	2300.00
Sample CC007	Unaged			Aged 48 hours			Aged 120 hours		
0.01	83.74	20.70	1.31	69.82	126.00	8.43	63.85	600.00	42.00
0.02	83.15	20.00	2.53	74.17	98.10	12.80	54.10	430.00	66.70
0.05	81.58	18.70	5.94	72.29	84.50	27.90	58.49	294.00	108.00
0.1	79.82	17.40	11.10	70.12	70.60	47.20	58.14	231.00	171.00
0.2	79.65	16.60	21.30	68.78	59.50	80.20	58.13	181.00	267.00
0.5	77.55	14.50	46.70	66.10	46.00	158.00	56.88	126.00	471.00
1	76.78	13.00	84.10	64.28	37.50	261.00	55.42	95.10	726.00
2	74.73	11.60	151.00	61.68	30.00	428.00	52.02	68.40	1090.00
5	72.19	9.75	322.00	57.91	21.60	800.00	46.15	41.30	1800.00
10	69.36	8.23	553.00	53.82	15.70	1220.00	41.20	26.10	2490.00

Table C.14. Summary of Rheology Test Results for Bitumen C on Aggregate E.

Sample EC001	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	85.69	19.80	1.25	77.05	130.00	8.40	70.91	363.00	24.10
0.02	83.30	19.10	2.41	74.85	117.00	15.30	69.45	312.00	41.80
0.05	81.37	17.60	5.58	73.25	100.00	32.80	65.29	247.00	85.30
0.1	78.82	16.20	10.40	70.82	84.60	56.30	64.34	199.00	139.00
0.2	79.80	15.40	19.60	69.05	72.40	97.40	62.48	162.00	229.00
0.5	77.81	13.90	44.80	66.98	56.60	193.00	59.13	118.00	430.00
1	76.03	12.50	81.20	64.12	46.00	322.00	55.78	90.40	687.00
2	74.54	11.20	146.00	60.81	36.40	524.00	52.42	65.90	1050.00
5	71.80	9.40	311.00	57.07	25.90	971.00	47.23	40.80	1740.00
10	68.85	7.92	533.00	52.66	18.70	1480.00	42.46	26.30	2450.00
Sample EC002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	86.30	10.70	0.671	77.67	126.00	8.10	63.27	538.00	37.90
0.02	85.04	9.90	1.25	77.30	109.00	14.10	61.88	409.00	58.20
0.05	83.04	9.54	3.02	73.73	97.40	31.90	63.81	326.00	114.00
0.1	81.87	9.18	5.82	70.58	82.40	54.90	62.33	260.00	184.00
0.2	80.11	8.54	10.90	69.32	69.70	93.60	60.36	213.00	307.00
0.5	77.67	7.84	25.20	67.26	55.00	187.00	56.86	147.00	553.00
1	76.01	7.14	46.30	65.49	45.40	313.00	53.96	111.00	859.00
2	76.26	6.52	84.30	61.55	35.70	511.00	50.66	80.70	1310.00
5	73.71	5.57	182.00	56.91	25.50	955.00	44.41	47.90	2150.00
10	71.56	4.80	318.00	52.62	18.30	1450.00	38.77	29.20	2930.00
Sample EC003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	85.73	17.40	1.10	77.92	97.80	6.28	69.65	274.00	18.30
0.02	85.05	15.90	2.01	76.03	87.00	11.30	67.91	255.00	34.50
0.05	82.86	16.10	5.09	74.00	76.00	24.80	65.73	191.00	65.70
0.1	81.14	15.30	9.74	71.10	65.10	43.30	65.38	159.00	110.00
0.2	79.83	14.40	18.30	70.48	57.30	76.50	63.40	126.00	178.00
0.5	78.29	12.50	40.00	67.37	44.90	153.00	60.75	93.20	336.00
1	76.38	11.20	72.30	65.40	37.10	256.00	57.43	71.90	536.00
2	75.82	10.10	131.00	63.36	29.90	421.00	53.96	53.40	830.00
5	72.70	8.46	278.00	58.76	21.40	787.00	49.04	34.20	1420.00
10	70.31	7.16	477.00	55.02	15.90	1220.00	43.98	22.30	2020.00

Table C.16. Summary of Rheology Test Results for Bitumen D on Aggregate A.

Sample AD002	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	55.07	165.00	12.70	57.03	201.00	15.00	60.56	391.00	28.20
0.02	53.76	117.00	18.20	56.65	159.00	23.90	57.64	304.00	45.30
0.05	58.86	89.70	32.90	60.11	120.00	43.30	58.08	216.00	80.00
0.1	60.85	72.30	52.00	61.50	98.30	70.30	60.69	185.00	133.00
0.2	62.41	60.40	85.60	62.29	80.70	115.00	60.67	148.00	213.00
0.5	64.10	47.20	165.00	61.09	61.30	220.00	59.74	110.00	400.00
1	63.89	38.60	270.00	60.20	49.50	358.00	58.24	85.50	632.00
2	63.36	31.90	449.00	58.96	39.30	577.00	53.82	63.80	994.00
5	59.58	23.80	868.00	55.15	27.70	1060.00	48.89	41.00	1710.00
10	55.53	17.90	1360.00	50.89	19.70	1590.00	43.64	26.70	2430.00
Sample AD003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	39.25	267.00	26.50	56.47	321.00	24.20	51.95	698.00	55.70
0.02	46.82	200.00	34.50	56.04	219.00	33.10	48.52	492.00	82.50
0.05	43.79	121.00	54.80	58.20	162.00	60.00	52.14	337.00	134.00
0.1	50.70	94.40	76.70	59.27	129.00	94.00	53.71	272.00	212.00
0.2	53.55	74.60	117.00	60.88	104.00	150.00	55.27	210.00	321.00
0.5	55.32	53.40	204.00	61.17	79.80	286.00	55.14	148.00	566.00
1	57.37	41.00	306.00	60.16	63.00	457.00	54.05	113.00	875.00
2	58.42	32.30	477.00	58.17	49.60	734.00	51.26	82.50	1330.00
5	57.71	23.20	862.00	54.12	33.90	1320.00	45.44	50.10	2210.00
10	55.04	17.40	1330.00	48.85	23.50	1960.00	39.97	31.30	3060.00

Table C.18. Summary of Rheology Test Results for Bitumen D on Aggregate D.

Sample DD003	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	60.99	67.00	4.82	56.17	175.00	13.20	58.19	302.00	22.30
0.02	56.94	46.90	7.03	55.83	135.00	20.50	56.17	229.00	34.70
0.05	59.93	36.10	13.10	59.81	103.00	37.40	59.60	175.00	63.80
0.1	62.33	30.30	21.50	61.97	84.60	60.30	60.27	145.00	105.00
0.2	64.42	25.20	35.10	62.03	71.60	102.00	60.96	115.00	165.00
0.5	66.49	20.30	69.70	63.21	54.90	193.00	60.27	85.70	310.00
1	67.07	17.10	117.00	62.07	44.00	313.00	57.61	66.60	495.00
2	67.35	14.60	199.00	60.69	35.50	511.00	55.62	51.00	777.00
5	65.45	11.40	393.00	56.51	25.00	942.00	50.66	33.40	1350.00
10	62.86	9.16	647.00	52.67	18.20	1440.00	45.72	22.30	1950.00
Sample DD004	Unaged			Aged 48 hours			Aged 120 hours		
0.01	54.03	91.00	7.07	53.13	199.00	15.60	62.08	280.00	19.90
0.02	53.40	59.60	9.32	53.46	148.00	23.20	57.75	204.00	30.40
0.05	57.05	45.30	16.90	55.65	106.00	40.20	61.13	157.00	56.20
0.1	60.26	37.10	26.80	58.76	84.80	62.30	59.97	128.00	93.00
0.2	62.88	30.70	43.30	58.59	67.20	98.90	61.54	104.00	149.00
0.5	64.88	24.30	84.20	60.97	53.10	191.00	61.50	79.90	286.00
1	66.04	20.20	139.00	60.66	42.80	309.00	60.19	63.60	461.00
2	65.94	16.90	232.00	59.82	33.90	493.00	57.28	48.90	730.00
5	64.68	13.00	453.00	56.56	24.20	912.00	53.01	33.20	1310.00
10	62.59	10.50	746.00	52.51	17.70	1400.00	48.43	23.00	1930.00

Table C.20. Summary of Rheology Test Results for Bitumen D on "Aggregate" F.

Sample FD003	Unaged			Aged 48 hours			Aged 120 hours		
Freq,Hz	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa	δ,°	V,kPa·s	G*,kPa
0.01	58.07	72.30	5.36	58.26	222.00	16.40	59.01	547.00	40.10
0.02	57.90	56.90	8.43	58.98	172.00	25.20	59.62	400.00	58.20
0.05	62.07	46.30	16.50	59.36	132.00	48.30	59.44	289.00	105.00
0.1	63.48	38.00	26.70	61.60	108.00	77.10	59.03	225.00	165.00
0.2	65.46	32.10	44.30	61.54	86.40	124.00	59.30	182.00	266.00
0.5	65.73	25.60	88.10	61.63	67.00	239.00	58.32	134.00	496.00
1	68.48	22.00	148.00	61.53	52.50	375.00	55.96	105.00	797.00
2	66.98	18.60	254.00	59.00	42.00	615.00	52.90	76.30	1200.00
5	64.78	14.60	506.00	54.81	29.00	1110.00	46.84	47.50	2040.00
10	61.80	11.60	829.00	50.42	20.60	1680.00	41.40	30.20	2870.00