Durability of Bituminous Paving Mixtures

by

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Thesis submitted to the University of Nottingham
for the degree of Doctor of Philosophy.

October, 1995

To Michelle

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 remains 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

Acknowledgements

The research work reported in this thesis formed part of the Bitutest project led by the University of Nottingham and funded by the Department of Transport / Engineering and Physical Sciences Research Council LINK Programme. Industrial support was provided by Cooper Research Technology Ltd., Esso Petroleum Company Ltd., Foster Yeoman Ltd., Mobil Oil Company Ltd., Nynas Bitumen, Shell Bitumen, SWK Pavement Engineering Ltd., Tarmac Roadstone Ltd. and Wimpey Environmental Ltd. In addition, 14 Highway Authorities carried out collaborative work on the project. I would like to acknowledge all this support together with that of the Rees Jeffreys Road Fund, the County Surveyors Society and, in particular, the Worshipful Company of Paviors. I would like to express my gratitude to my colleagues John M Read, J Michael Gibb, Keith Cooper, Professor PS Pell and, especially, Professor SF Brown for their support during this project and throughout my research work at the University of Nottingham.

I would like especially to thank my wife Michelle for her unending support whilst I prepared this thesis and, indeed, throughout my studies at the University.

Table of Contents

Table of Contents (Continued)

Table of Contents (Continued)

List of Figures

Page

List of Figures (Continued)

List of Figures (Continued)

Page

List of Tables

List of Tables (Continued)

Page

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

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*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.

worldwide consumption of bitumen (2). In 1994, over fifty million tonnes of bitumen was used for building roads and, not surprisingly, most of this went into roads in the United States and in Europe. The annual consumption of bitumen in the UK has been considerably more modest, hovering around two million tonnes per year over the last two decades (1), but is nonetheless a sizeable quantity.

1.2 PROBLEM STATEMENT

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. Bitumens are used to bind together aggregates of various mineralogical compositions and graded to various size distributions resulting in mixtures which vary considerably in terms of density, binder content and bitumen-aggregate combinations—the permutations are astronomical given the diversity of the compositions of bitumens and mineral aggregates alone. Each of these factors can significantly influence the performance characteristics of the mixture which are largely dictated by the interaction between the bitumen and the aggregate. Stated another way, the ability of the bitumen to maintain adequate adhesion and waterproofing characteristics as well as resistance to detrimental environmental factors (i.e., the ability of the bitumen to provide good durability) is not determined solely by the bitumen but must also consider mixture variables; in particular, mineral aggregate type, aggregate gradation, binder content and void content (which is related to aggregate gradation and density and may be roughly indicative of mixture permeability). Thus, compatibility of the bitumen and the aggregate, the volumetric proportions of binder and air voids and the permeability of the mixture are all important factors when considering the durability of bituminous paving mixtures.

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 remains an enigma.

The recently completed (1993) Strategic Highway Research Program (SHRP) in the United States was one such research effort that studied, amongst many other things, age hardening and water damage in bituminous mixtures. A total of $50 million was spent over a five year period to define chemical and physical properties of bitumens and their relationship to pavement performance. The project was principally concerned with the development of tests conducted on neat bitumens that could predict mixture performance but a substantial portion—$10.5 million—was concerned with the development of mixture tests. Numerous "innovative" products have come from this program (4, 5) of which those of present interest include:

- a conceptual model (i.e., explanation) of bitumen;
- an accelerated ageing procedure performed on neat bitumens;
- accelerated ageing procedures performed on loose and compacted bituminous mixtures and
- a procedure to induce and evaluate damage due to water on compacted bituminous mixtures.

These, as well as some of the other test methods related to bitumen and/or mixture performance, are discussed where appropriate throughout this thesis.

The Bitutest project carried out at the University of Nottingham and completed in August, 1995 was similar in concept to the portion of the SHRP program concerned with mixture tests in that tests were developed to assess mixture performance. However, the approach adopted for the Bitutest project was to develop simple tests, unlike some of the complex (and expensive) mixture tests developed for the SHRP asphalt program. As part of the Bitutest project test methods and practices for durability were also developed. For ageing, these were modelled after the methods developed for the SHRP program whereas the method developed to assess damage due to water was partially modelled after the method developed for SHRP and partially after a method developed prior to the SHRP asphalt program.

The development of the durability tests and practices for the Bitutest project were this Author's responsibility. Hence, a substantial portion of this thesis is dedicated to the evaluation of the tests developed for this project.

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

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2 Literature Review

2.1 INTRODUCTION

This chapter summarises the principal findings from a review of selected literature concerning the durability of bituminous mixtures. References were obtained from a search conducted by the library at the Transport Research Laboratory, this Author's own literature search and from the literature itself. The vast majority of references were obtained from literature from the United States as most of the research in this area has been reported there—relatively little has been reported in the UK.

2.1.1 Problem Statement

Highway engineers attach considerable importance to the durability of bituminous paving mixtures; and rightly so, as the costs of maintenance and rehabilitation of pavement structures that do not survive their design life can be substantial. While the principal failure mechanisms resulting from traffic loading are cracking and permanent deformation, adverse environmental effects can accelerate the deterioration process. The durability of bituminous mixtures is therefore not only important to the engineer but also to the road user, for it is the road user who must ultimately pay for the construction and maintenance of roads. Many factors affect the durability of bituminous mixtures. However, assuming that a pavement layer is constructed according to specifications, it is generally agreed that the two primary factors that affect the durability of the mixture are embrittlement of the bitumen due to age hardening and damage due to moisture[1].

It is well known that bitumen becomes stiffer (increases in viscosity) during the mixing and construction process of bituminous pavements as well as while the

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[1]The terms *moisture* and *water* are used interchangeably throughout this chapter and are intended to mean liquid water which appears to affect the mixture in a different way from that of *water vapour*.

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.

result in the loss of material—severe stripping can deteriorate the bituminous mixture to a virtually cohesionless base material (4).

2.1.2 Definition of Durability

A product which is *durable* is one which is able to exist for a long period of time without significant deterioration. The factors which affect the durability of bituminous mixtures, under this definition, would include all factors which contribute to deterioration. However, the highway industry generally restricts the term durability to those effects which are related to the environment; namely moisture and ageing. For example, Whiteoak (5) states that, "Durability can be defined as the ability to maintain satisfactory rheology, cohesion and adhesion in long-term service." The Asphalt Institute Manual Series No.2 (6), however, refers only to water when discussing the durability of bituminous mixtures. Terrel and Al-Swailmi (3) state that, "Environmental factors such as temperature, air, and water can have a profound effect on the durability of asphalt concrete mixtures." In addition, Terrel and Shute (7) indicate that, "Environmental factors, traffic and time are the factors which need to be accounted for in the development of test procedures to simulate the field. Environmental factors include; moisture from precipitation or groundwater sources, temperature fluctuations (including freeze-thaw conditions) as well as [ageing] of the asphalt. The effect of traffic could also be considered as an external influence or environment." Further, Terrel and Al-Swailmi (3) showed that traffic loading increases stripping and concluded that repeated loading (i.e., simulation of traffic loading) is a very important variable to be included in water conditioning protocols. Similarly, in an earlier study, Lottman (8) found that heavy traffic volume appeared to increase the rate of damage due to moisture more effectively than climatic extremes of precipitation and temperature.

The above lack of consensus regarding the factors which influence the durability of bituminous paving materials makes an accurate definition difficult. Clearly, there is a general consensus that water and ageing affect durability but uncertainty as to whether traffic loading should be included. Lottman (9), Tunnicliff and Root (10), and Terrel and Al-Swailmi (3) have incorporated the effects of temperature variation 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.

2.2.1 Source of Bitumen

Most bitumens are the product of the distillation of crude oil, although some are products of solvent precipitation processes. In either process the components of the crude oil having the greatest molecular weight become concentrated in the bitumen. It is generally agreed that crude oil originated from the sedimentation of large quantities of organic and vegetable matter, together with mud and rock fragments, on the ocean floor which were converted into hydrocarbons by heat from the earth's crust and pressure applied by the upper layers of sediment, possibly aided by bacteria and radioactive bombardment (5). It is not known how long the process took but it has been reported to be on the order of millions of years (5, 11, 12). Due to the nature with which crude oil originated and the large number of such deposits throughout parts of the world, the physical and chemical properties of the crude oils vary widely as do the properties of bitumens produced from them.

2.2.2 Elemental Composition of Bitumen

Bitumen is comprised of a complex mixture of organic molecules which vary widely in composition. The molecules are comprised primarily of hydrogen and carbon, referred to as hydrocarbons, but most contain one or more heteroatoms (nitrogen, sulphur, and oxygen) and trace amounts of metals, primarily vanadium, nickel and iron. Table 2.1 lists the elemental composition of several typical paving grade bitumens, each from a different source and having widely varying physical characteristics. Note that the concentration of heteroatoms, although a minor component, can vary widely depending on the source of the bitumen (i.e., the crude oil). Petersen (11) notes, "Because the heteroatoms impart functionality and polarity to the molecules, their presence may make a disproportionately large contribution to the differences in physical properties among [bitumens] from different sources."

Although elemental composition is important to note, it provides little information regarding how the atoms are assembled into molecules or what types of molecular structures are present in the bitumen, knowledge of which is necessary for a fundamental understanding of how composition affects physical properties and chemical reactivity (11).

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

2.2.3 Molecular Structure

The organic molecules comprising bitumens vary widely in composition from nonpolar, saturated hydrocarbons to highly polar, highly condensed ring systems (11). The way in which the elements are incorporated into molecules and the type of molecular structure present is far more important than the total amount of each element present in bitumen. Because of the way the source of bitumen was derived from living organisms, it is not surprising that the molecular structure of the components of bitumen are highly diverse. A full discussion of the various types of organic compounds found in bitumens is well beyond the scope of this thesis, but it is important to note the ways in which carbon atoms are linked to one another.

Figure 2.1 illustrates the three principal types of molecules found in bitumen. The carbon atoms in the aliphatics, or paraffinics, are linked in straight or branched chains. In the naphthenics, or cyclics, they are linked in simple or complex (condensed) saturated rings, where "saturated" means that the highest possible hydrogen to carbon ratio is present. Aromatics are materials characterised by the presence of one or more especially stable six-atom rings (e.g., benzene, toluene, etc.).

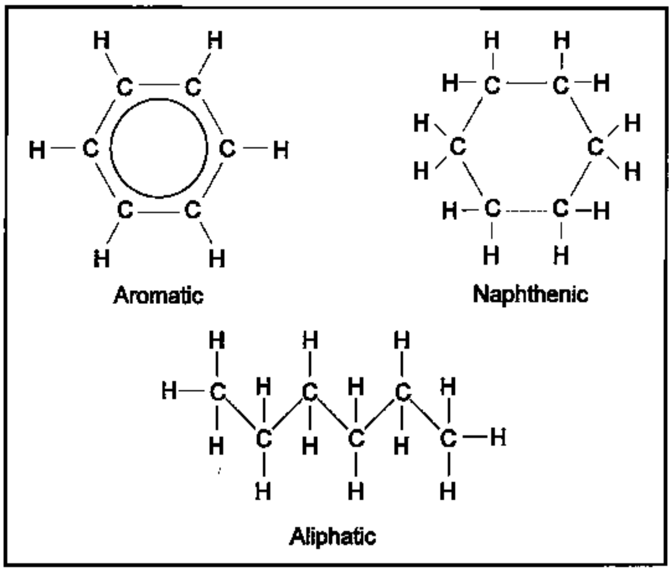

Figure 2.1 Types of Molecules Found in Bitumen.

The physical and chemical behaviour of bitumens are affected by the various ways in which these compounds interact with one another. The molecules are held together through chemical bonds which are relatively weak and can be broken by application of heat and/or shear loading. For example, when bitumen is heated, the intermolecular associations are destroyed, which gives the molecules mobility and results in a mixture that has the ability to flow freely. When the bitumen cools, the bonds are 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 are comprised of saturates, naphthene aromatics and polar aromatics. The saturate fraction is a viscous oil lacking polar chemical functional groups. The molecules of this fraction are nonpolar and may contain saturated normal and branched-chain (i.e., aliphatic) hydrocarbons, saturated cyclic hydrocarbons and, in addition to sulphur, possibly a small amount of mono-ring aromatic hydrocarbons. The naphthene aromatic fraction is a viscous liquid which constitutes the major proportion of the dispersion medium for the asphaltenes. This fraction may contain condensed nonaromatic and aromatic ring systems and possibly sulphur and the heteroatoms oxygen and nitrogen. The polar aromatic fraction, comprised of highly condensed aromatic ring systems and functional groups containing heteroatoms, serve as the peptisers or dispersing agents for the asphaltenes. This fraction is highly polar giving it strong adhesion characteristics.

Each of the components of bitumen is comprised of many different chemical compounds that coexist in neat bitumen as a homogenous mixture which is made possible by the interaction of the various components with one another to form a balanced and compatible system. It is the balance of the components which gives bitumen its unique viscoelastic properties. Imbalance or incompatibility amongst the components, as sometimes manifested by component phase separation, leads to undesirable properties (11).

2.2.5 Functionality and Polarity

As previously mentioned, the heteroatoms—nitrogen, sulphur and oxygen—impart functionality and polarity to the molecules present in bitumens. Although present in small quantities, the heteroatoms significantly affect the physical properties and performance characteristics of bitumens. Functionality refers to the way in which molecules in bitumen interact with each other as well as with the molecules and/or surfaces of other materials (e.g., aggregate). Polarity refers to the way in which the electrochemical forces in the molecules are imbalanced, producing a dipole. Polar compounds form associations which give bitumen its elastic properties. These compounds coexist with nonpolar compounds which, together, give bitumen its viscous properties.

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 hydrogen 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.

functions in the same molecule), and nonpolar (neutral) components demonstrated the existence of the solvent and dispersed moieties as postulated by the model. The researchers found the nonpolar components to consist of aliphatic, naphthenic and aromatic hydrocarbons (Figure 2.1) as well as substantial amounts of organosulphur compounds. The polar fraction was found to consist of predominately amphoterics, which are believed to contain condensed aromatic ring units capable of forming associations through π-π interactions as well as other acid-base interactions. These multifunctional molecules were shown to have a profound effect on the physical properties of bitumens and are considered to be the principal viscosity-enhancing components of bitumens.

Size exclusion chromatography (SEC), which was used to separate bitumens into fractions comprised of associating and non-associating components according to molecular size, was also used to validate the microstructural model. It was shown by this method that large units, much larger than at the molecular level, did exist and could be isolated. The amphoterics, as determined by ion exchange chromatography, were found to compose most of the associating components as determined by SEC and, thus, complemented the IEC results. Bitumens having relatively few associating components were found to differ in rheological properties from those having large amounts of associating components. Although the amount of the associating component would be expected to correlate with asphaltene content, which provides an indicator of total polarity, there did not always exist a strong correlation.

The results of the research carried out by the A-002A research team confirmed that the essential features of the microstructural model were valid. Petersen et al (18) indicates that, by using the model, it should be possible to predict important physical properties of bitumens from specific chemical properties and vice versa. However, they concede that no global chemical variable was found to be a good predictor of physical properties.

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).

Table 2.2. Effects Which May Reduce the Binding Properties of Bitumen (1).

Effects	Influenced by					Occurs		Ways to Retard
	Time	Heat	Oxygen	Sun-light	Beta & Gamma Rays	At Surface	In Mass	In General, Selected, Source and Process
1. Oxidation (in dark)	✓	✓	✓			✓		1) Inert atmoshere 2) Free radical inhibitors
2. Photooxidation (direct light)	✓	✓	✓	✓		✓		1) Protection from light 2) Inert atmosphere 3) Free radical inhibitors
3. Volatilisation	✓	✓				✓	✓	Protection from heat
4. Photooxidation (reflected light)	✓	✓	✓	✓		✓		1) Protection from light 2) Inert atmosphere 3) Free radical inhibitors
5. Photo chemical (direct light)	✓	✓		✓		✓		1) Protection from light 2) Additives?
6. Photo chemical (reflected light)	✓	✓		✓		✓	✓	1) Protection from light 2) Additives?
7. Polymerisation	✓	✓				✓	✓	Free radical inhibitors
8. Development of internal structure (Ageing, Thixotropy)	✓					✓	✓	1) Add dispersing agents 2) Change source and processing of bitumen
9. Exudation of oil (Syneresis)	✓	✓				✓		Reduce paraffinic content
10. Changes by nuclear energy	✓	✓			✓	✓	✓	
11. Action by water	✓	✓	✓	✓		✓		Change source and processing
12. Absorption by solid	✓	✓				✓	✓	Improve dispersion of bitumen
13. Adsorption of components at solid surface	✓	✓				✓		
14. Chemical reactions or catalytic effects at interface	✓	✓				✓	✓	
15. Microbiological deterioration	✓	✓	✓			✓	✓	Add fungistatic and bacteriostatic agents

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.

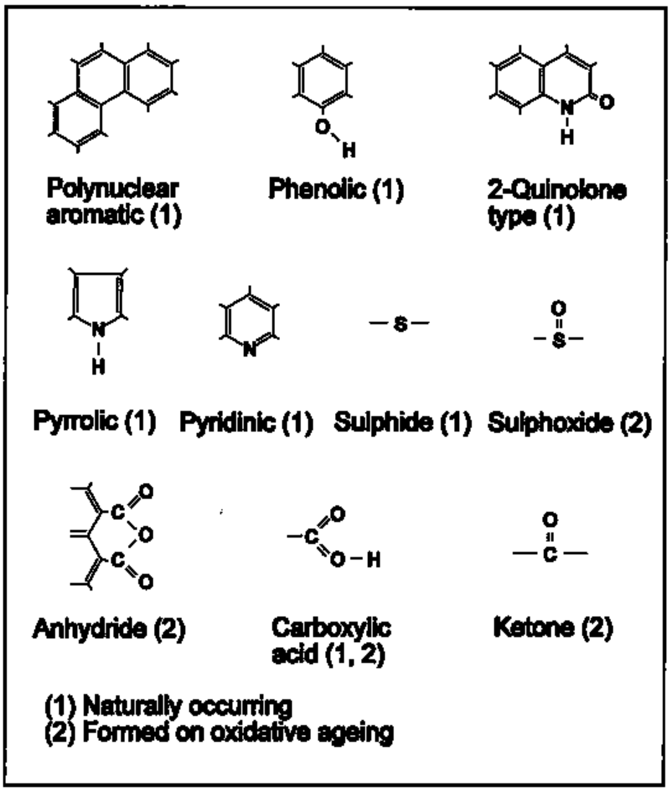

Figure 2.2. Chemical Functionalities in Bitumen Molecules Normally Present or Formed on Oxidative Ageing (11).

Table 2.3. Chemical Functional Groups Formed in Bitumens During Oxidative Ageing (20).

Bitumen	Concentration (moles per litre)				Average Hardening Index[b]
	Ketone	Anhydride	Carboxylic Acid[a]	Sulphoxide	
B-2959	0.50	0.014	0.008	0.30	38.0
B-3036	0.55	0.015	0.005	0.29	27.0
B-3051	0.58	0.020	0.009	0.29	132.0
B-3602	0.77	0.043	0.005	0.18	30.0

Note: Column oxidation, 130°C, 24 hours, 15μm film
[a]Naturally occuring acids have been subtracted from reported value.
[b]Ratio of viscosity after oxidative ageing to viscosity before oxidative ageing.

Table 2.4. Carbonyl Functional Groups Formed in Wilmington Bitumen Fractions During Column Oxidation (21).

Fraction	Concentration (moles per litre)		
	Ketone	Anhydride	Carboxylic Acid
Saturate	0.045	0.010	Trace
Aromatic	0.32	0.017	–[a]
Polar Aromatic	1.48	0.088	–[a]
Asphaltene	1.82	0.080	ND[b]
Whole Bitumen	1.02	0.052	0.007

[a]Some acids lost on alumina column during component fractionation.
[b]Not determined.

Although the products of oxidation are an important factor in age hardening, ageing is not directly related to concentrations of oxidation products (18). For example, the non-associating components in the solvent moiety may be very effective in dispersing weakly associated polar species formed by oxidation resulting in a highly oxidised bitumen that does not show a large change in stiffness. Conversely, a small change in stiffness can result from formation of strongly associated polar species that are dispersed by non-associating components in a solvent moiety with poor dispersing capacity. Thus, the degree of stiffening is the result of the total associating polarity 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-aggregate interface may, under certain situations, change the properties of the bitumen enough to affect its durability in service.

Petersen (11) also recognizes that environmental factors, particularly water, can seriously affect the performance and durability of bituminous paving materials. However, although damage due to water may be related to bitumen composition and adsorption of bitumen components onto aggregate surfaces, it is primarily an interfacial phenomenon.

Consequences of Age Hardening

Age hardening of the binder in bituminous mixtures is the result of compositional changes causing an increase in viscosity in the bitumen. Figure 2.3 illustrates the increase in viscosity over time for several different bitumens while in service (23). Similar findings have been reported by other researchers (e.g., 24-28). Note that the viscosities of the bitumens increased roughly two orders of magnitude during nearly 10 years of service. It should be noted, however, that the steric hardening (molecular structuring) component may not be represented in the data shown as the viscosities were determined on recovered bitumen from the mixtures which probably destroyed the effect and, therefore, the values shown are probably somewhat lower than that of the in situ bitumens.

Excessive age hardening can result in a brittle bitumen—one with significantly reduced flow capabilities—which contributes to various forms of cracking in the bituminous mixture. Cracking generally occurs in the form of fatigue, thermal, or reflective cracking. Fatigue cracking is the result of an accumulation of damage, arising from repeated or fluctuating stresses (i.e., traffic loading), which eventually leads to fracture. Thermal cracking is the result of thermally-induced tensile stresses which exceed the tensile strength of the bitumen. Thermal cracking can occur as a result of the mixture temperature falling below some limiting temperature and/or as the result of an accumulation of permanent tensile strain arising from repeated or fluctuating thermal stresses. Reflective cracking occurs in mixtures which overlay existing roadways that are cracked. Cracks in the overlay appear directly above

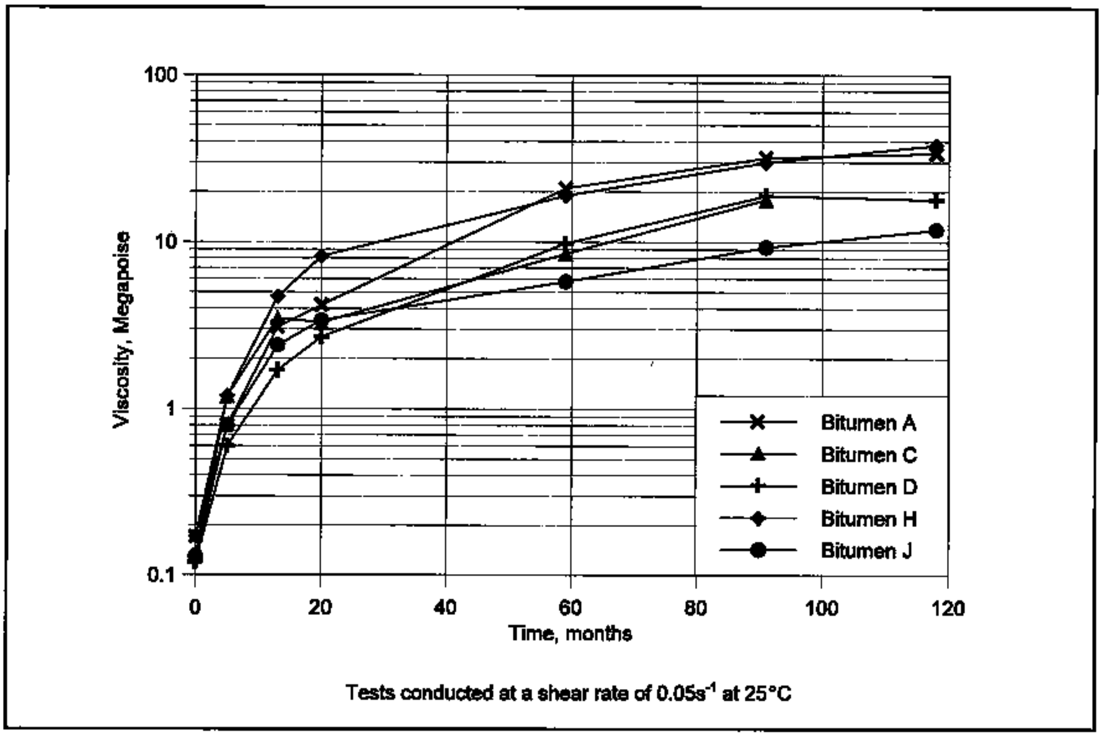

Figure 2.3. Viscosity Change of Several Bitumens During Service in Pavements (23).

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.

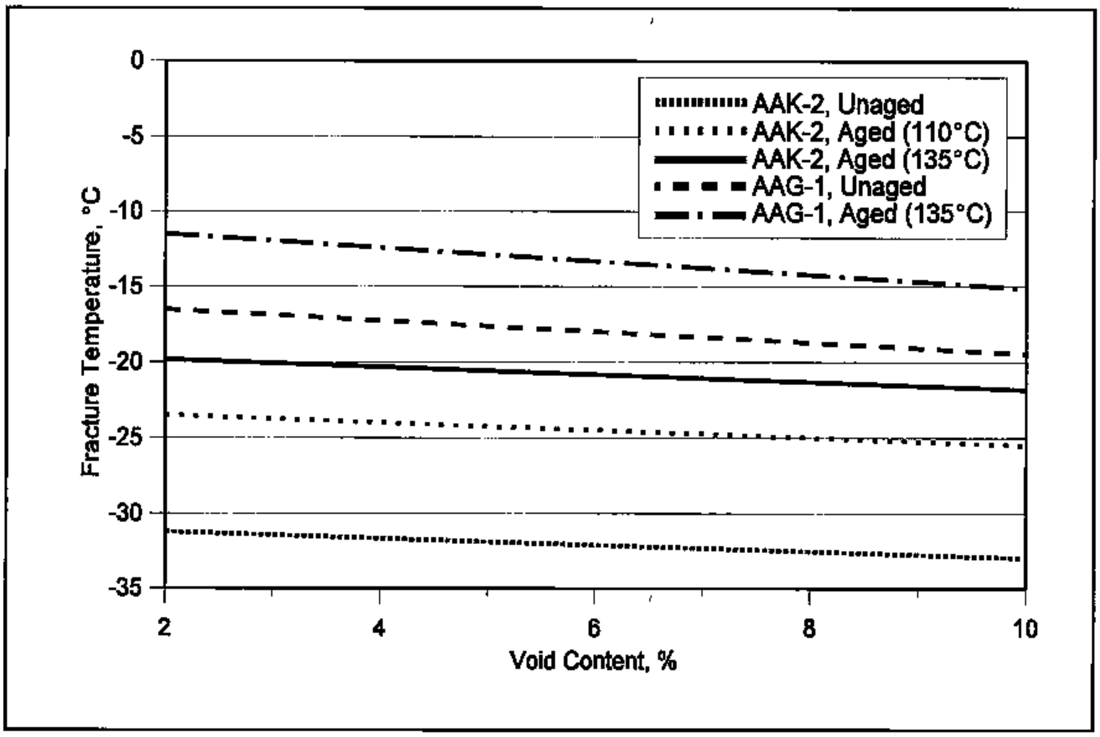

Figure 2.4. Effect of Age Hardening on Fracture Temperature (29).

2.3.2 Factors Affecting Water Sensitivity

It has long been recognised that moisture can degrade the structural integrity of bituminous mixtures. Although many factors contribute to the degradation of bituminous mixtures, moisture appears to play a major role. In general, water can reduce the stiffness or strength of a bitumen-aggregate mixture and/or cause the bond between bitumen and aggregate to fail, both potentially resulting in significant distress to the bituminous pavement. The mechanisms of damage due to moisture are not clearly understood. However, many researchers have investigated moisture sensitivity of bitumens and bituminous mixtures and have provided significant advances towards a better understanding of the mechanisms of moisture damage.

Mechanisms of Moisture Damage

It is generally agreed that moisture can degrade the integrity of bituminous mixtures in two ways: 1) by causing a reduction in the cohesive strength and stiffness of the mixture, characterised by a softening of the mixture and 2) by causing failure of the adhesion (or bond) between bitumen and aggregate, referred to as stripping (2, 7).

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 film. Failures can be cohesive or interfacial and can occur in either the bitumen or the aggregate.

Their work also showed that the adsorptive behaviour of bitumen and bitumen model components on aggregates is highly specific and particularly influenced by the aggregate surface chemistry; the chemistry of the bitumen has less influence. The polar functional groups of bitumen adhere to active sites (i.e., those sites that contain metals or charged species) on the aggregate surface through electrostatic, dipole-dipole or Van der Waals interactions. The polar functional groups (see Figure 2.1) most strongly adsorbed on aggregates such as granite, limestone, greywacke and gravel included sulphoxides, carboxylic acids, nitrogen bases and phenols, whereas less polar species such as pyrroles and ketones and nonpolar species were found to be much less adsorbed and not competitive for the aggregate surface. Interestingly, the two most polar species (i.e., sulphoxides and carboxylic acids) were readily desorbed in substantial amounts of water whereas the nitrogen bases and phenols were found to be most resistive to aqueous desorption.

Surfaces rich in alkaline earth metals (e.g., calcium and magnesium) were less likely to be susceptible to adhesive debonding than were surfaces rich in alkali elements (e.g., sodium and potassium). In earlier work Scott (33) suggested that the susceptibility of siliceous aggregates to stripping may be associated with the presence of water soluble cations and aluminosiclicates where the mechanism of water stripping is probably 1) the dissolution of water soluble salts, 2) the dissolution of silica resulting from the high pH environment generated by solubilisation of the alkaline earth cations, 3) electrostatic repulsion between the negatively charged aggregate and anionic components of the bitumen at the interface and 4) dissolution of soaps formed between acid anions on the bitumen surface and alkali metal cations on the aggregate surface. Thus, it can be seen that the adhesion and debonding characteristics of a bitumen-aggregate system cannot be determined solely by the generic aggregate type but must be determined by the physical and chemical nature of the specific surface with which the bitumen comes in contact.

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 mixtures. Welborn (35) concluded that although thin film oven tests (which are widely accepted methods for measuring the potential hardening of bitumen during plant mixing and widely used in research endeavours) predict the properties of the bitumen at the time of construction, they do not provide adequate information regarding the change in properties during service in the pavement. However, as will become evident, thin film ovens continue to be used in attempts to predict bitumen durability. Consequently, a review of such tests is warranted.

Bell (14) stated that, "Compared to research on asphalt cement, there has been little research on the aging of asphalt mixtures, and, to date, there is no standard test." In addition, in recent research work for the Strategic Highway Research Program in the United States, Bell has found that there exists a significant interaction between bitumen and aggregate with regard to age hardening of bituminous mixtures. This is not an entirely new finding as other researchers (e.g., 1, 11, 35) have indicated the importance of the effect of the aggregate on the ageing of the binder in bituminous mixtures. For this reason, a review of tests on mixtures is also warranted.

Binder Tests for Ageing

Numerous attempts have been made by researchers to correlate accelerated laboratory-ageing of neat bitumen with field performance. Many of the research endeavours utilised thin film ovens to age the bitumen in an accelerated manner. The **Thin Film Oven Test** (TFOT) was introduced by Lewis and Welborn (36). In this test the residue of a 50ml sample of bitumen, which had been placed on a flat, 140mm diameter container such that it was 3.2mm thick and heated for 5 hours at 163 °C, was tested for penetration, ductility and softening point. Although the test was adopted by the American Society for Testing and Materials (ASTM) in 1969 (ASTM D1754; 37) as a test method to evaluate bitumen durability, particularly from the standpoint of hardening during plant mixing, numerous significant modifications to the test have been developed since its inception. A few minor modifications to the TFOT have been developed as well: Edler et al (38) used a film thickness of 100μm and an exposure time of 24 hours while Griffin et al (39) reported on the **Shell Microfilm Test** which ages a 5μm thick film of bitumen for 2 hours at 107 °C. The bitumen is 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:

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

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.

Kemp and Predoehl (42) developed the **Tilt-Oven Durability Test** (TODT). The TODT is an adaptation of the RTFOT whereby the oven is tilted 1.06° higher at the front to prevent bitumen migration from the bottles. In addition, the test is conducted for 168 hours at 113 °C. Penetration, viscosity and ductility tests are conducted on the residue.

Recognising that although thin film oven tests can adequately measure the relative hardening characteristics of bitumens during the mixing process but fall short of accurately predicting long-term hardening in the field, several researchers have combined thin film oven ageing with oxidative ageing. Bell (14) reports work by Lee (43) on the development of the **Iowa Durability Test** (IDT). This test subjects the original bitumen being evaluated to the Thin Film Oven Test (i.e., a 3.2mm film heated for 5 hours at 163 °C) followed by pressure oxidation treatment to the residue whereby the residue is placed in a vessel which is pressurised to 2.07MPa using pure oxygen and heated to 65 °C for up to 1000 hours. In this work Lee found that ageing the original bitumen in this manner followed a hyperbolic relationship similar to that which actually occurred in the nine bitumens he evaluated over a five year period of field ageing.

Bell (14) also reports work carried out by Edler et al (38) which utilised a similar approach to Lee (43) in a study to evaluate procedures to retard oxidative hardening of bituminous mixtures in South Africa. In this work the original bitumen was subjected to extended rolling thin film oven ageing (8 hours) followed by oxidation at a pressure of 2.07MPa and a temperature of 65 °C for 96 hours. Bell (14) notes the procedure produced hyperbolic curves and resulted in ageing indices similar to that reported by Petersen (44). In addition, they found that although the addition of lime significantly increased the viscosity of the unaged binder, it also significantly retarded hardening of the bitumen.

Petersen et al (44) developed the **Thin Film Accelerated Ageing Test** (TFAAT) which is a modification to the RMFO such that it provided a 4g sample of bitumen, a sample size which provided a sufficient quantity of bitumen for further testing as 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 in pavements. He also notes that a temperature of 85 °C has been recommended by Bell and Sosnovske (47) and Robertson et al (48).

The SHRP A-002A research team developed a method "to rapidly simulate in the laboratory oxidative age-hardening as it occurs in the field within a pavement" (49). The method, referred to as **TFO-PAV**, involves oxidation in the RTFOT (ASTM D2872; 37) or the TFOT (ASTM D1754; 37) to simulate the age hardening which occurs during plant mixing followed by oxidation of the residue in a pressurised ageing vessel (PAV) to simulate age-hardening which occurs in the field (50, 51). The residue from either thin film oven (TFO) is conditioned in the PAV for 20 hours at a pressure of 2.1MPa and at temperatures between 90 and 110 °C, the particular ageing temperature being dependent on the climatic region where the binder will be put into service and is selected from the SHRP performance-graded binder specification (51).

Mixture Tests for Ageing

Conducting tests on bitumen-aggregate mixtures in an attempt to predict the durability characteristics of the mixture is not a new approach. Dow (34) proposed a durability test whereby the recovered bitumen from aged and unaged mixtures were tested in the penetrometer to determine the change in consistency due to ageing. In this test, the aged mixture was heated in an oven at 149 °C for 30 minutes prior to recovering the bitumen. Welborn (35) notes that there was no evidence that this method was used in specifications, but that it did give some indication of the relative durability of bitumens supplied at the time. The development of a standard method for recovering bitumen from bituminous mixtures (52) led to several subsequent studies of bitumen-aggregate mixtures. Welborn (35) and Bell (14) provide excellent summaries of these studies. Bell (14) identifies four categories of mixture tests: 1) extended heating procedures; 2) oxidation tests; 3) ultraviolet/infrared treatment and 4) steric hardening. The following discussion under these headings is based largely on Bell's work (14).

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 indicate loss of volatiles. Finally, beams were cut from the slabs and the shrinkage during the ageing test determined.

Von Quintas et al (55) have published the findings from the second phase of the study to develop an Asphalt Aggregate Mixture Analysis System (AAMAS). They investigated the use of forced-draft oven ageing to simulate "production hardening;" that is, hardening due to short-term ageing. The laboratory method involved heating loose mixtures for periods of 8, 16, 24 and 36 hours in an oven at a temperature of 135°C. They compared the recovered versus initial penetration and viscosity ratios for bitumens used in each of five projects, for both field and laboratory ageing. For two projects they obtained similar levels of ageing in the laboratory as that which occurred in the field. Bell (14) notes that there is considerable scatter in the laboratory data and that Von Quintas and his co-workers discounted the possibility of using the TFOT or RTFOT to age the bitumen first, and then prepare laboratory mixtures, because this would be time consuming.

Von Quintas et al (55) also investigated "long-term environmental ageing" using a forced-draft oven as follows:

1) Six compacted specimens were placed in an oven for 48 hours at 60°C.
2) Three specimens were removed and the temperature of the oven was increased to 107°C to age the remaining three specimens for 120 hours.

Pressure oxidation treatment was also investigated, with three compacted specimens conditioned for 120 to 240 hours at a temperature of 60°C and a pressure of 0.7MPa. Von Quintas and his co-workers found that indirect tensile strengths were higher for oven-aged mixtures and failure strains were lower for pressure oxidised mixtures implying that the oven ageing procedure was more severe. They also presented data of initial and recovered properties of the bitumen which indicated that oven ageing was more severe for one project but less severe for the other two. Von Quintas and his co-workers recommended that oven ageing be used rather than pressure oxidation. Bell (14) points out that, due to the limited and somewhat questionable data, further research was needed before selection of an ageing method. He also points out that the 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.

He offers that confinement may be desirable or that it may be necessary to use a lower temperature. The fatigue data (performed in the controlled stress mode) indicated that increased ageing time resulted in increased cycles to failure with the more poorly compacted mixtures showing longer fatigue lives. Bell (14) explains that, because the tests were carried out at a fixed level of applied tensile stress, the stiffer mixtures (as a result of ageing) experienced lower strain levels during the tests. He notes that a different trend would be expected if a fixed tensile strain were used.

Ultraviolet/Infrared Treatment. Hveem et al (40) presented a comprehensive description of various tests and specifications for paving grade bitumens. Among them was an infrared weathering test where a mixture of Ottawa sand and bitumen (5 to 7μm thick) was tested in a "semi-compacted state." The infrared radiation was controlled to give a constant mass temperature of 60°C while a constant flow of air at 41°C was maintained across the specimen. They describe a calibration procedure to determine the number of hours required in the weathering test to correspond to field ageing based on a shot abrasion test used to evaluate the aged mixture. Bell (14) notes that the authors "state with some confidence" that 1000 hours of exposure in the weathering machine is approximately equivalent to 5 years of field ageing.

Hugo and Kennedy (54) evaluated the effect of ultraviolet (UV) radiation on laboratory-prepared specimens as well as on those obtained from newly constructed pavements. They utilised two approaches: 1) a procedure similar to that used by Traxler (1) which used 54 hours of UV exposure and 2) use of the Atlas weatherometer for a period of 14 days. Bell (14) notes that the levels of ageing obtained through these tests were very small relative to similar tests conducted on neat bitumen.

Bell (14) indicates that Tia et al (59) conducted an extensive study which included development of ageing methods using heat and ultraviolet light. They found that similar levels of ageing were obtained in mixture samples aged by either oven ageing or UV light. They recommended that an improved method be developed incorporating both methods of ageing with the operating temperature being 60°C.

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.

Bell and Sosnovske (47) and Bell et al (60) describe methods to age mixtures so as to simulate both short-term and long-term ageing; that is, ageing which occurs during construction and ageing which occurs whilst the pavement is in service, respectively. Short-term oven ageing (STOA) involves curing the bitumen-aggregate mixture in a forced-draft oven for 4 hours at 135°C while it is in a loose state (i.e., after mixing but prior to compaction). Two alternatives were developed to simulate long-term ageing of bituminous mixtures: 1) long-term oven ageing (LTOA) in a forced-draft oven and 2) low-pressure oxidation (LPO) by passing heated oxygen through a specimen fitted in a modified triaxial cell. Both methods are carried out on compacted specimens which have been short-term aged prior to compaction.

Bell and Sosnovske (61) evaluated various combinations of time and temperature for the two long-term ageing procedures; namely, LTOA at 85°C for 5 days, LTOA at 100°C for 2 days and LPO at 60°C or 85°C for 5 days. They concluded that the long-term ageing methods produced somewhat different rankings of ageing susceptibility amongst the 32 laboratory-prepared mixtures they tested compared with the short-term ageing procedure as well as with each other. They partially attribute the differences to variability in the materials, ageing procedures and testing. The low-pressure oxidation method caused the most ageing and least variability in ageing susceptibility rankings relative to short-term ageing. Based on their findings they recommended that loose mixtures should be short-term aged for 4 hours at 135°C followed by either long-term oven ageing or low pressure oxidation of the compacted mixture for 120 hours at 85°C. They indicate that, for the long-term ageing procedures, a temperature of 100°C for a duration of 48 hours could be used but warn that such a high temperature may cause damage to specimens.

Earlier, Potschka (62) described a simple test apparatus which can be used to age uncompacted bituminous mixtures thus allowing the investigation of the hardening of bitumen including the effect of aggregate. The apparatus consists of an insulated pot which is electrically heated and thermostatically controlled. An uncompacted bituminous mixture placed in the pot rests on a perforated metal sheet which is covered with filter paper. Synthetic air (consisting of 20.5% oxygen and 79.5%

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

damage is generally made by a ratio of conditioned to unconditioned strength or stiffness (e.g., indirect tensile strength or indirect tensile stiffness), where "unconditioned" refers to the as-cored or as-manufactured properties of the compacted mixture and "conditioned" refers to the properties after the compacted mixture has been subjected to some sort of treatment intended to simulate in-service conditions of the pavement.

Terrel and Al-Swailmi (3) note that none of the identified tests have emerged as acceptable over a wide range of conditions and materials and describe a method developed under the A-003A contract for the SHRP asphalt program. Additionally, under the A-003B contract for the SHRP asphalt program, Curtis et al (32) describe a method they developed to be used as a screening test to determine whether a particular bitumen-aggregate mixture is likely to be sensitive to damage due to water.

Whiteoak (5) identifies six categories of adhesion tests as follows:

1) Static Immersion Tests;
2) Dynamic Immersion Tests;
3) Chemical Immersion Tests;
4) Immersion Mechanical Tests;
5) Immersion Trafficking Tests and
6) Coating Tests.

Whiteoak (5) notes that, although the various tests can be used to compare different combinations of bitumen and aggregate, little information is available to correlate test data with performance in practice. Additionally, he notes that the various tests differ in the type of specimen used, the conditions under which the sample is tested and the method by which stripping is assessed.

Although the water sensitivity tests developed to date have some shortcomings with regard to prediction of long-term pavement performance, it is fruitful to review the methods to ascertain what has been learned from the methods as well as to determine 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.

Whiteoak (5) also describes **chemical immersion tests** whereby aggregate coated with bitumen is boiled in solutions containing various concentrations of sodium carbonate. The concentration of the sodium carbonate solution in which stripping is first observed is used as a measure of adhesiveness. Whiteoak notes that the artificial conditions of the test make it of doubtful value in predicting road performance.

Curtis and her co-workers (32) developed the **Net Adsorption Test** for SHRP to be used as a screening procedure for selecting bitumens and aggregates as well as to determine the effectiveness of antistripping additives. The test involves adsorption of bitumen dissolved in toluene onto the aggregate followed by aqueous desorption of the bitumen. The adsorption phase is carried out for 6 hours and adsorption is measured indirectly by virtue of the decrease in bitumen concentration in the toluene-bitumen solution. After the adsorption phase, a prescribed quantity of water is introduced into the system and bitumen is desorbed from the coated aggregate, the quantity of which is determined by the increase in bitumen in solution. The amount of bitumen which remains on the aggregate after aqueous desorption is termed the net adsorption.

Tests on Compacted Mixtures

Numerous tests on compacted mixtures, either prepared in the laboratory or cored from existing pavements, have been developed in an attempt to assess the moisture susceptibility of the mixtures. These methods are summarised in the following paragraphs.

The **Texas Freeze-Thaw Pedestal Test** (FTPT) attempts to simulate viscosity changes in bituminous mixtures which have been in service for five years. In this test small specimens (41mm in diameter by 19mm in height) are fabricated from a single size aggregate (100% passing the 0.85mm sieve and retained on the 0.5mm sieve). After fabrication the specimen is cured at 23 °C for 3 days then placed on a pedestal which acts as a fulcrum. The arrangement is placed in a water bottle and subjected to thermal cycling until the specimen is observed to have cracked. Mixtures which crack within 10 thermal cycles are deemed to be moisture susceptible while those 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, aggregate gradation, bitumen content and void content. Four of these specimens are tested according to the standard Marshall method yielding a standard stability value. The remaining four specimens are vacuum treated under water at a temperature between 0 and 1 °C, stored in a water bath at 60 °C for 48 hours and tested for Marshall stability. The retained Marshall stability is then determined as the ratio of the Marshall stability of the specimens which were conditioned to the standard (unconditioned) Marshall stability.

Whiteoak (5) also describes the **Immersion Wheel Tracking Test** (IWTT) which simulates the effect of traffic whilst the bituminous mixture is immersed in water. In this test time to failure is determined for bituminous mixtures immersed in water at 40 °C and subjected to 20kg wheel loading reciprocating at 25 cycles per minute where failure is indicated by a sudden and significant increase in plastic deformation of the specimen. Whiteoak notes a study which indicated that good correlation exists between stripping failures on heavily trafficked roads and the behaviour of similar materials in the immersion wheel tracking test. The immersion wheel tracking test was shown to have excellent correlation with the Net Adsorption Test (described earlier) for the four aggregates evaluated in the comparison (13). However, Whiteoak (5) cited another study incorporating seventeen aggregates which showed wide variability in failure times.

Lottman (9) describes the method, commonly referred to as the **Lottman procedure**, he developed for the prediction of moisture damage in dense-graded bituminous mixtures. The method consists of obtaining conditioned to unconditioned ratios of indirect tensile strength and stiffness where the conditioned specimens are either subjected to vacuum saturation alone or to vacuum saturation followed by freeze-plus-warm-water soak, more commonly referred to as freeze-thaw. Vacuum saturation consists of submerging the specimens in distilled water in a vacuum desiccator, applying a partial vacuum (660mm Hg) for 30 minutes, after which the specimens are left submerged for an additional 30 minutes at atmospheric pressure. Freeze-thaw consists of tightly wrapping the vacuum-saturated specimens in plastic wrap, placing them in heavy-duty plastic bags (each containing ≈3ml of distilled 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.

Terrel and Al-Swailmi (3) describe the **Environmental Conditioning System** (ECS), a system developed to evaluate the sensitivity of bituminous mixtures to moisture-induced damage. The system is comprised of three major components: 1) a fluid conditioning system; 2) a loading system and 3) an environmental conditioning cabinet. The fluid conditioning system is used to wet the test specimen as well as determine air and water permeability of the compacted mixture. It also contains facilities to monitor pH of the distilled water passing through the specimen as well as specimen and water temperatures. The loading system is comprised of an electro-pneumatic closed-loop servo system and a modified triaxial cell which also serves as the load frame. The triaxial cell/load frame is housed within the environmental cabinet which is capable of a wide range of temperatures (-20 to 100 °C) and humidity levels (up to 95% relative humidity). The specimen (102mm in diameter by 102mm in height) is tested in a triaxial configuration where no confining stress (i.e., $\sigma_2 = \sigma_3 = 0$). The environmental cabinet is capable of heating and cooling the specimen from a temperature of 25 °C to 100 °C and −20 °C, respectively, within 2 hours.

The test method reported by Terrel and Al-Swailmi (3) is summarised in Table 2.5. Note that both air and water permeability are measured and that the specimen is tested in a triaxial configuration (with no confining stress). It is interesting to note that Lottman (63) reports to have evaluated repeated-load triaxial tests (like that used in the ECS) but found that the resultant data for laboratory specimens did not correlate well with pavement cores and, thus, the test was not pursued further.

2.5 LINKING TEST METHODS TO FIELD PERFORMANCE

Several studies have been conducted to evaluate the long-term durability of bituminous pavements. A majority of the studies reviewed investigated only the ageing characteristics of bitumens in relation to pavement durability. This section reviews the significant studies which attempted to link durability tests to field performance.

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

(Period 1) and completed in March 1955 (Period 2). Periodic inspections and coring was carried out over a period of several years (March 1955 to April 1964) to investigate the following factors:

1) Surface surveys and crack records;
2) Deflection properties;
3) Properties of pavement cores removed at various time intervals and
4) Changes in original properties of the various bitumens during mixing and pavement service life.

Zube and Skog (23) provide the following conclusions:

1) Observations and test results confirm the fact that bitumens manufactured by different methods and from different crude oil sources, although placed under virtually identical conditions, exhibit varying degrees of hardening during the mixing process.
2) The initial void content and the rate of change in void content during service life, together with the bitumen content, appear to be dominant factors in the hardening rate of the various bitumens. However, the rate of hardening under equivalent weathering and pavement conditions is also influenced by the bitumen source.
3) Of the various durability tests evaluated, results from the TFOT and hardening in the mixer show an excellent correlation and the Shell Microfilm Test appears to be the best test for predicting the durability properties of the bitumen.

Vallerga and Halstead (22) describe an extensive 30-month field and laboratory study on paving-grade bitumens subjected to 11 to 13 years of service in 53 highway pavements located throughout the United States. The specific objectives of the study were to:

1) Measure changes that occurred in the physical and chemical properties of bitumen, after 11 to 13 years in service in pavements, from a sufficient variety of sources to be representative of national [US] bitumen production;

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.

The major conclusions drawn from this study included:

1) High average air temperature (thermal oxidation) was the most significant factor affecting the rate and amount of bitumen hardening.
2) Void content also contributed to the rate of oxidation with higher percentages being more detrimental.
3) Aggregate porosity was found to have a significant effect on hardening of the bitumen in hot climatic regions and was more significant with more volatile bitumen.
4) The following were identified as factors that would improve bitumen durability:
   a) Adherence to compaction specifications to reduce voids;
   b) The selective use of bitumens that are most suited to the quality of aggregate available;
   c) Avoidance of use of absorptive aggregate, if possible, in hot climates;
   d) Use of the softest grade of bitumen consistent with mixture curing and stability constraints and
   e) Insulation of the pavement with a cover such as a reflective chip seal, especially in hot climates.

Kandhal and Koehler (26) report the findings from three bitumen durability projects undertaken by the Pennsylvania Department of Transportation on 16 dense-graded bituminous pavements. The pavements were periodically cored to determine the percentage of air voids and rheological properties of the aged bitumens. Tests on the bitumen included penetration at 25°C, viscosity at 60°C, ductility at 15.6°C and at 50mm/minute and shear susceptibility at 25°C. In this study they found:

1) Changes in percentage air voids and bitumen properties, such as viscosity and shear susceptibility, were found to follow the hyperbolic model suggested by Brown et al (64) and Lee (43).
2) Low temperature ductility was found to be an important factor in pavement performance; lower ductility values were associated with a higher incidence of load-associated longitudinal cracking.

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 were mixtures obtained at site and brought into the laboratory, where they were reheated to 110 °C and compacted in a kneading compactor prior to being tested.

Subsequent to this preliminary study a more comprehensive effort was undertaken to include a greater number of field sites as well as to validate the combined ageing procedures. The field sites represented areas throughout the United States as well as one from southeastern France and were aged up to 19 years. The results of this effort showed that short-term oven ageing (i.e., ageing the loose mixture for 4 hours at 135 °C) plus long-term oven ageing for 2 days at 85 °C or 1 day at 100 °C represented the amount of ageing that could be expected for "young" mixtures (0 to 3 years) in the field. Extending the durations to 4 to 8 days at 85 °C or 2 to 4 days at 100 °C appeared to be representative of "older" mixtures (greater than 3 years) in the field but conservative for some mixtures. They recommend that long-term ageing be carried out at 85 °C citing that 100 °C may cause damage to specimens and result in unreliable data.

2.5.2 Water Sensitivity

Although numerous investigations have been carried out with regard to the water sensitivity of bituminous mixtures, particularly the evaluation of additives, only two studies which address long-term durability were found. These were Lottman's work (4, 9, 63) and validation of the Environmental Conditioning System (66) and are discussed below.

Lottman (4, 9, 63) conducted a study involving eight dense-graded bituminous pavements representing a variety of mixtures in various climatic regions in the United States that were evaluated periodically for approximately five years. Aggregates which had a history of moisture damage were generally chosen for inclusion in the bituminous mixture. The test sections of the pavements were selected such that they were at an elevation of 305m.

Immediately after construction the pavements were cored and tested using the Lottman procedure (9) to determine the initial tensile strengths and stiffnesses as well 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 strength) and stripping appear to correspond to the maximum achievable levels of moisture damage in pavements greater than five years old. Lottman (4) notes the vacuum saturation plus freeze-thaw portion of the test had been previously correlated with damage in pavements 3 to 12 years old (8).

Lottman (4) sums up by inferring that dense-graded mixtures that give high long-term ratios will withstand the rigors of a reasonable moisture-damage mechanism while those with low long-term ratios are prone to stripping and will not provide long-term field service.

Efforts to validate the ECS, described by Allen and Terrel (66), involved comparison of ECS test results on mixtures prepared with the original materials from 12 field sites with results from stiffness modulus tests carried out on field cores from the field sites. The ECS test results were also compared with those from rutting tests conducted in the Laboratoires des Ponts et Chausées (LCPC) rutting tester as well as the Elf (Hamburg) Wheel Track Tester. The 12 field sites were located throughout the United States and all were only a few years old (the oldest pavement was constructed in 1989).

Allen and Terrel (66) showed that, although the ECS was able to discriminate between mixtures that performed well and those that performed poorly with regard to water sensitivity, the ECS procedure caused more damage than that indicated by field cores in 8 of the 12 mixtures they evaluated. They indicate that the confounding factors of ageing and variation in the environment conditions among field sites, as well as the relatively short time the mixtures were in place, are probable reasons for the lack of correlation. The ECS was shown to correlate reasonably well with rutting tests conducted in the LCPC rutting tester on mixtures that had been water damaged by a procedure similar to that used for the ECS. A limited number of tests conducted in the Elf wheel tracker showed that a reasonable correlation existed between it and the LCPC rutting tester.

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 currently exists no test method for water sensitivity which has been shown to accurately predict long-term field performance of bituminous pavements.

Embrittlement of the binder due to ageing in bituminous mixtures can also adversely affect bituminous mixtures. Distress in bituminous mixtures due to age-hardening is usually manifested in cracking as a result of a reduction in the flow properties of the binder. Age-hardening of the binder is the result of compositional changes in the bitumen, principally the atmospheric oxidation of certain components of the bitumen which form highly polar and strongly interacting chemical functional groups containing oxygen (e.g., ketones, sulphoxides and, to a lesser extent, carboxylic acids). These species influence the total associating polarity 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. This results in increased viscosity and reduced penetration of the bitumen and, thus, a reduction in the ability of the bitumen to flow under the influence of thermal- and traffic-induced stresses and strains when incorporated in a pavement. Confounding the issue is that there exists clear evidence that the aggregate plays a significant role in the way bitumen hardens over time and that steric hardening (molecular structuring) may contribute to reduced durability characteristics.

Several tests have been developed in an attempt to predict the hardening characteristics of bitumens in bituminous pavements. Thin film oven tests have been shown to adequately predict the hardening of bitumen during plant-mixing and, in many instances, to predict hardening of the bitumen in pavements. However, the correlations of these tests with field performance are generally limited to specific materials (e.g., a limited number of crude oils and aggregate types), specific conditions (e.g., voids less than 4%) and times the pavement is in service (e.g., 11 to 13 years). Tests on mixtures have also 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. It is interesting to note that, although it is recognised that the aggregate is quite important with regard to age hardening of bituminous mixtures, the TFO-PAV test, a thin film oven and pressure oxidation technique performed on the 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.

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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 following sections describe an investigation of the effect of oven storage on the material properties (stiffness modulus) of bituminous mixtures. The outcome of the study led to the development of the *Standard Practice for Laboratory Preparation of Compacted Bituminous Mixtures* (Appendix A); one of two protocols developed for ageing of bituminous mixtures, this one for short-term ageing.

3.2 MATERIALS INVESTIGATED

Investigations were carried out on two typical UK mixture types obtained from six different sources (contractors) providing information from six different mixtures. The mixtures investigated included three continuously-graded base course mixtures, of which two were dense bitumen macadams (DBMs) and one was a heavy duty macadam (HDM), and three gap-graded wearing course mixtures, all of which were hot rolled asphalts (HRAs). Materials from the six sources were obtained prior to mixing at the plant as well as prior to paving at the site. Thus, virgin materials were obtained for laboratory mixing to investigate the effect of laboratory curing periods, and plant-mixed materials were obtained for the purpose of determining the duration of curing period in the laboratory which best simulates actual construction practices. All test specimens were compacted using the percentage refusal density method (1) and all evaluations were based on the indirect tensile stiffness modulus (Appendix B) as determined in the Nottingham Asphalt Tester (NAT) in accordance with BS DD213 (2).

3.3 EXPERIMENT DESIGN

3.3.1 Laboratory Samples

The experiment design for the investigation to determine whether or not oven storage affects the material properties (stiffness modulus) of bituminous mixtures was as follows:

- Number of contractors: 6
- Number of mixtures: 1 per contractor
- Number of ageing periods: 5
- Number of specimens per ageing period: 3
- $6 \times 1 \times 5 \times 3 = 90$ test specimens

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 which test specimens were compacted without having been oven aged. Following compaction, the specimens were tested for density and stiffness modulus.

3.4.2 Field Samples

Plant-mixed materials, made from the same ingredients as those used for the laboratory specimens, were obtained immediately prior to paving in the field (at the screws of the paver). These materials were then compacted on site in accordance with the percentage refusal density method. At least six specimens were compacted, taken to the University, and tested for density and stiffness modulus.

3.5 ANALYSIS

The methodology for analysis of the results was originally to take the average stiffness modulus of the field samples and plot this on a stiffness modulus-versus-storage period plot for the laboratory specimens. From this plot a laboratory storage period, which would simulate the amount of ageing that occurred in the plant, could be determined from the intersection of the field stiffness modulus value and the stiffness modulus-versus-storage period curve. This method was used for the gap-graded (HRA) mixtures but not for the continuously-graded (HDM and DBM) mixtures as the void contents of the field samples for these mixtures varied signficantly, thus providing a range of stiffness modulus values. To overcome this, a regression of stiffness modulus versus void content was performed and the stiffness modulus at the average void content of the laboratory specimens was determined. Thus, modulus values at similar void contents were compared.

3.6 TEST RESULTS

3.6.1 Continuously-Graded Mixtures

The continuously-graded mixtures utilised in this study included a 20mm DBM base course mixture with 200 pen bitumen, a 28mm DBM base course mixture with 200 pen bitumen, and a 28mm HDM base course mixture with 50 pen bitumen. All three mixtures were from batch plants. Table 3.1 and Figures 3.1 and 3.2 summarise the results for the 20mm DBM mixture. The results indicate that oven storage is not necessary to simulate the amount of ageing that occurred in the plant (see Figure 3.2).

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				

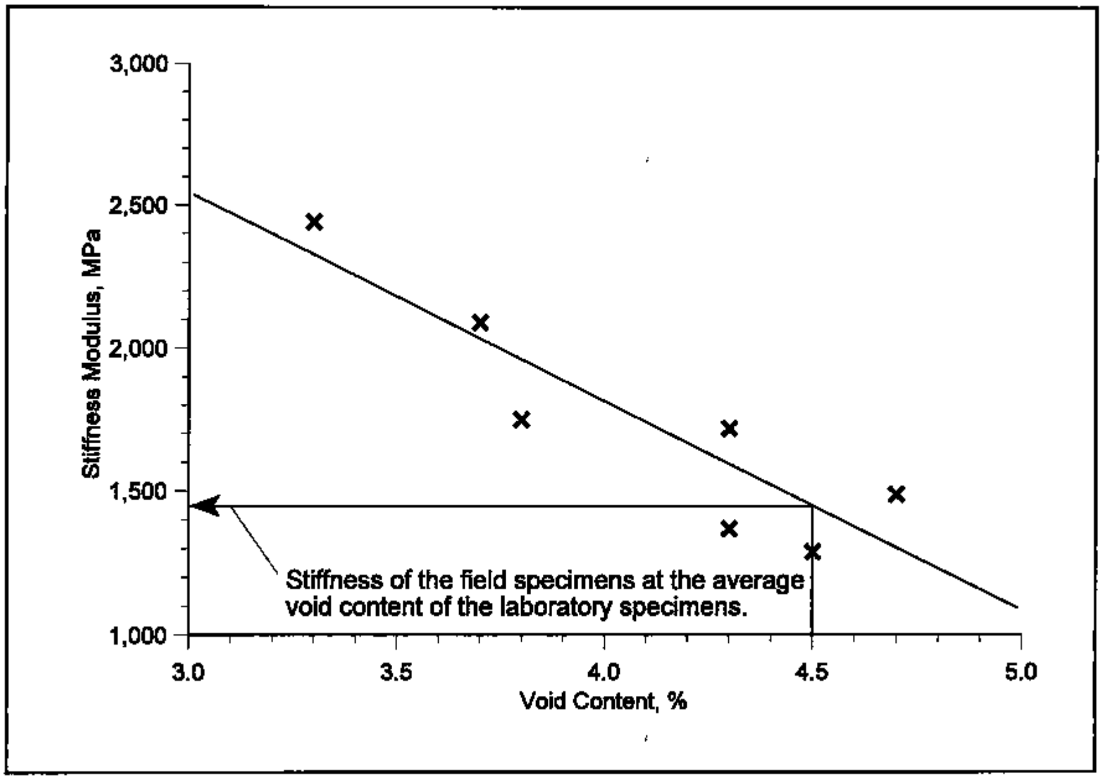

Figure 3.1. Test Results for the 20mm DBM Field Specimens.

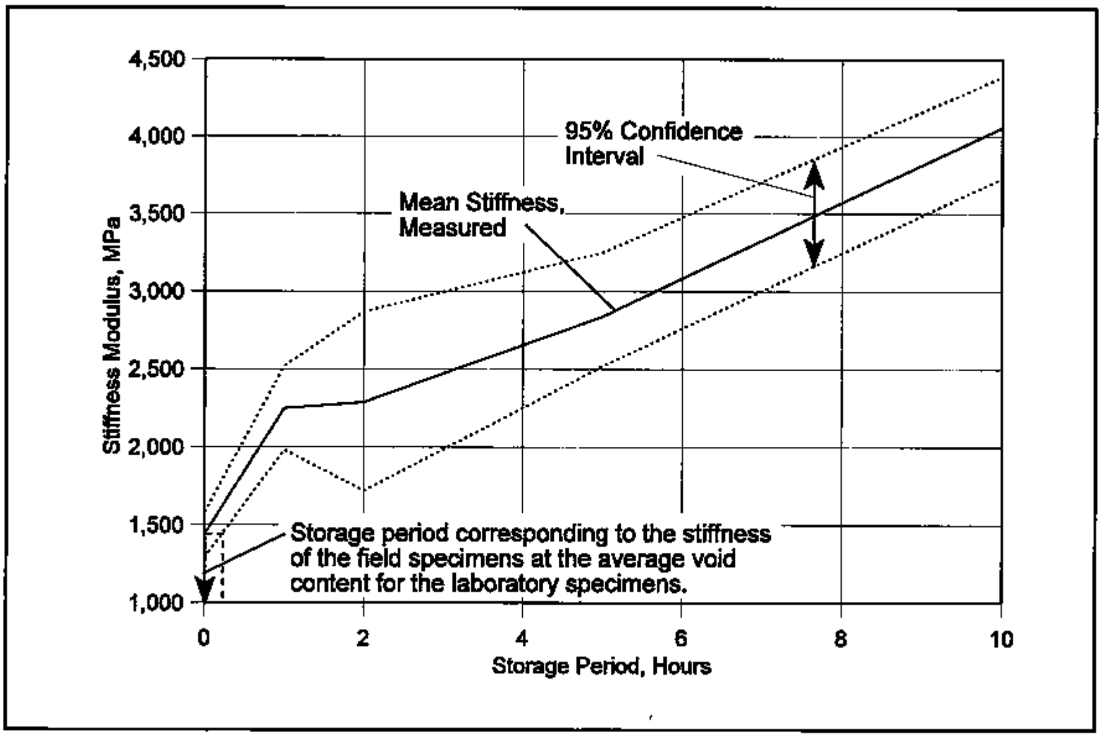

Figure 3.2. Test Results for the 20mm DBM Laboratory Specimens.

Table 3.2 and Figures 3.3 and 3.4 summarise the results for the 28mm DBM mixture. These results indicate that a storage period of just over 2 hours provided a stiffness equivalent to that obtained for the plant-mixed materials at an equivalent void content. In addition, when consideration is given to the variability of the test results, as indicated by the 95% confidence interval, it can be seen that a storage period between 1.9 and 2.5 hours is reliably predicted. Table 3.3 and Figures 3.5 and 3.6 summarise the results for the 28mm HDM mixture. These results also indicate that a storage period of just over 2 hours provides a stiffness equivalent to that obtained for the plant-mixed materials at an equivalent void content. There is, however, considerable variability in the test results for the laboratory prepared specimens (see Table 3.3 and Figure 3.6). This variability was largely due to the inability of the NAT to apply large enough loads to induce sufficiently large deformations in the specimens that had been stored for 5 and 10 hours such that the deformations could be made reliably. The results indicate a storage period between 1.3 and 5 hours, which is too imprecise for practical use although it embraces the more specific periods determined for the other DBM mixtures.

Table 3.2. Summary of Results for the 28mm 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	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				

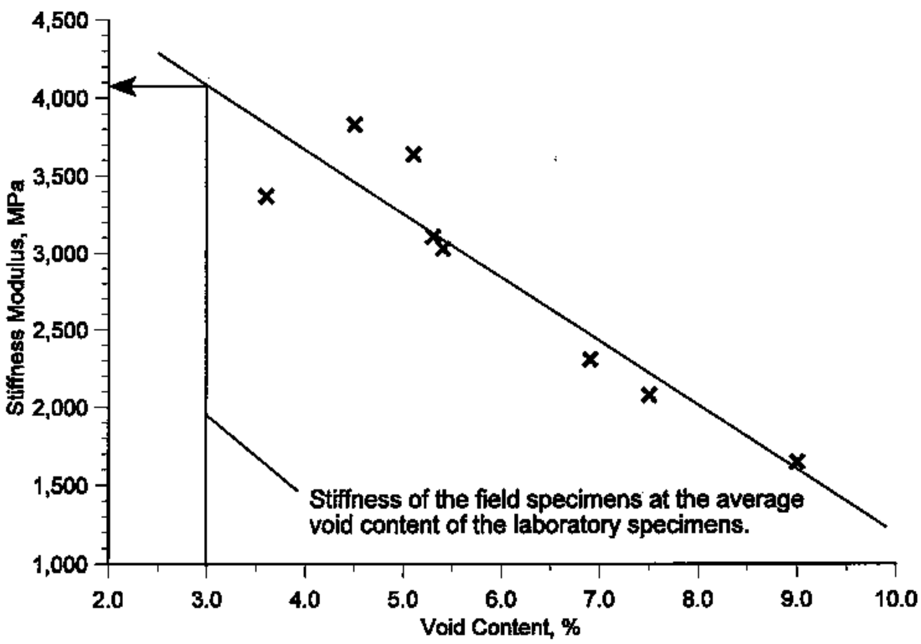

Figure 3.3. Test Results for the 28mm DBM Field Specimens.

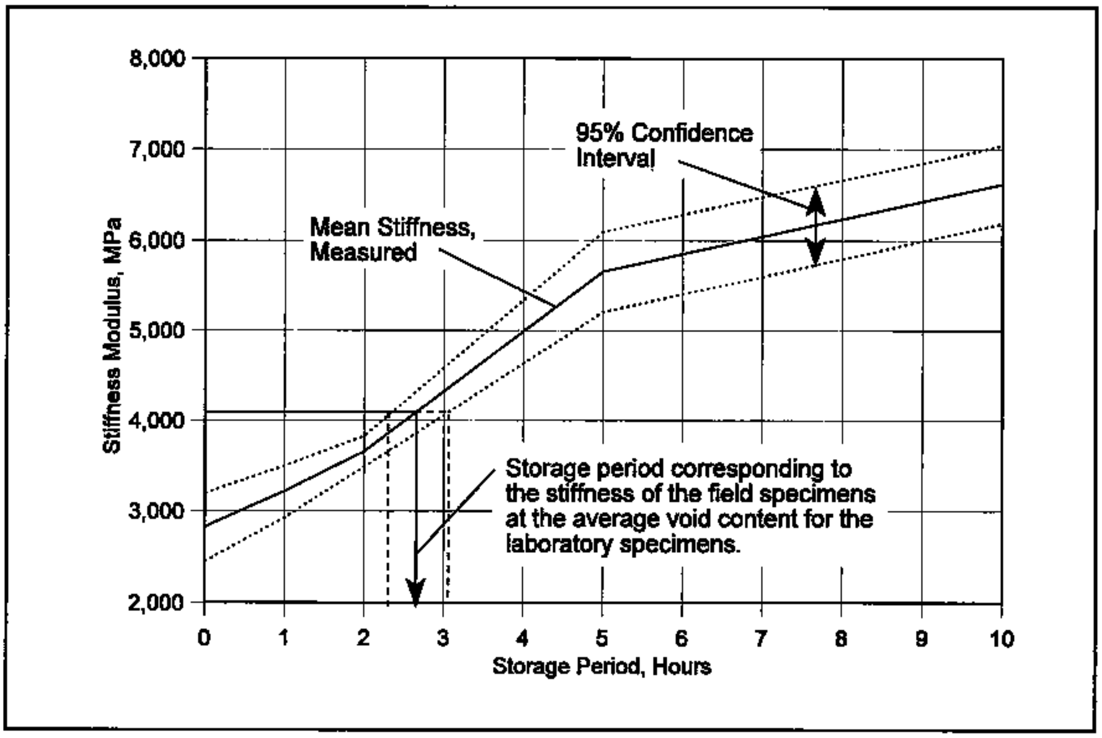

Figure 3.4. Test Results for the 28mm DBM Laboratory Specimens.

Table 3.3. Summary of Results for the 28mm HDM Mixture.

Field Specimens			Laboratory Specimens			
					Stiffness (MPa)	
Sample ID	Void Content (%)	Stiffness (MPa)	Storage Period (Hours)	Average Void Content (%)	Mean	95% Confidence Interval
F1	3.4	9340	0	2.6	7080	6780 - 7370
F2	5.9	7520	1	4.2	7770	7440 - 8100
F3	4.3	9090	2	5.0	8310	7570 - 9050
F4	5.6	6990	5	5.1	9880	8320 - 11450
F5	6.4	7610	10	6.1	23100	15400 - 30860
F6	5.2	8140				
F7	6.4	7400				
F8	8.9	7110				
F9	7.0	6210				

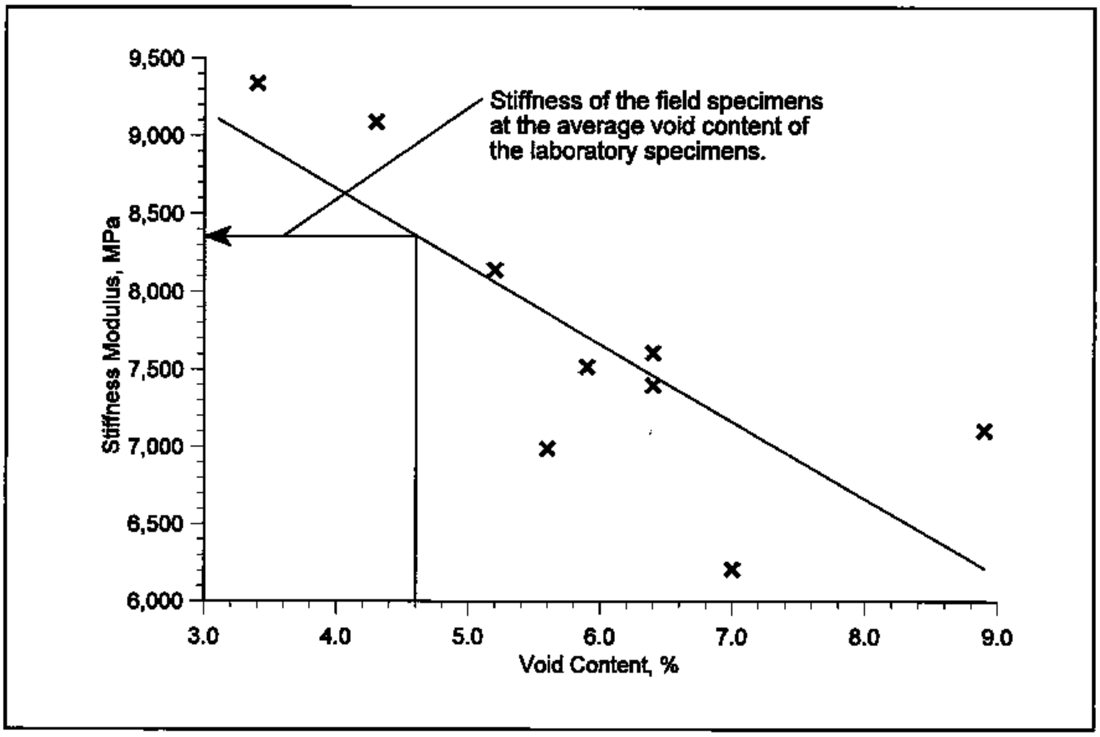

Figure 3.5. Test Results for the 28mm HDM Field Specimens.

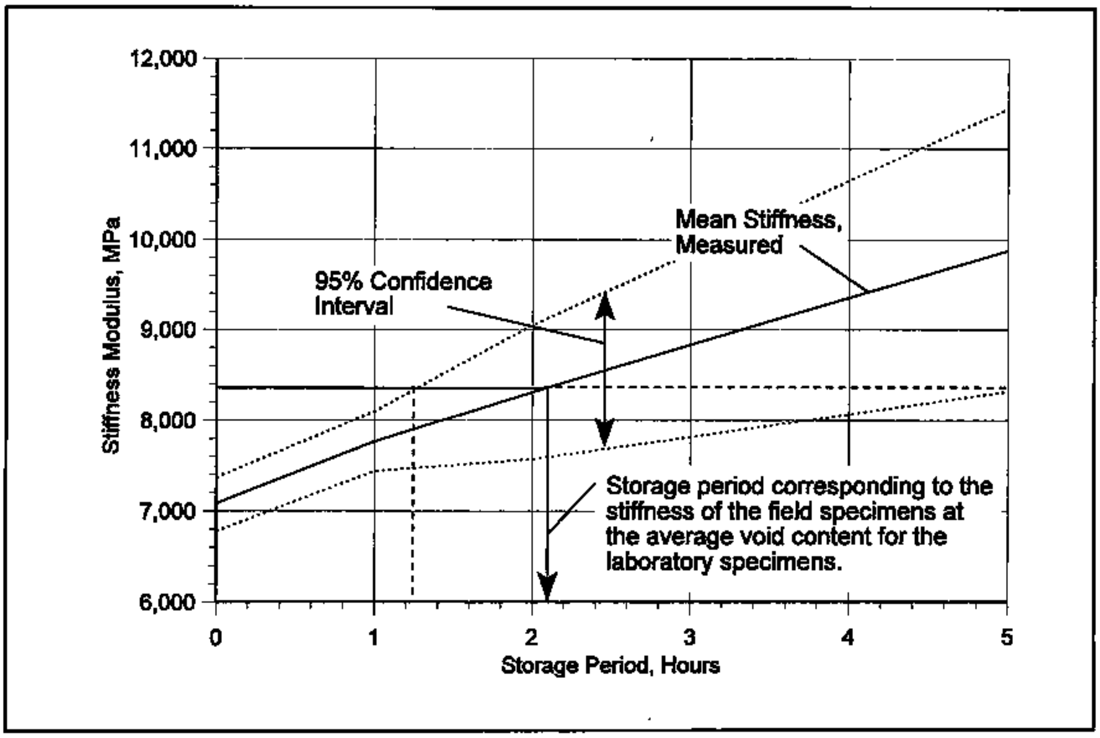

Figure 3.6. Tests Results for the 28mm HDM Laboratory Specimens.

3.6.2 Gap-Graded Mixtures

The gap-graded mixtures utilised in this study included a 30/10 HRA and two 30/14 HRA wearing course mixtures, all of which contained 50 pen bitumen. One of the two 30/14 mixtures was from a batch plant, whereas the other was from a drum mixer plant. The results for the 30/10 HRA wearing course are summarised in Table 3.4 and Figure 3.7 and indicate that oven storage is not necessary to simulate the ageing which occurred in the plant. The results for the 30/14 wearing course mixture from the batch plant are summarised in Table 3.5 and Figure 3.8. These results also indicate that oven storage is not necessary to simulate the ageing which occurred in the plant. The results for the 30/14 wearing course mixture from the drum mixer plant are summarised in Table 3.6 and Figure 3.9 and again, indicate that oven storage is not necessary.

3.7 DISCUSSION OF RESULTS

The test results, in all cases, indicated that oven storage of the loose mixture at 135°C significantly affected the stiffness modulus of the compacted mixture. For example, storage of the mixtures for 1 hour resulted in an increase in stiffness moduli of between 10 and 56% for the continuously-graded mixtures and between 14 and 41% for the gap-graded mixtures relative to the stiffness moduli of the mixtures that had not been stored (i.e., 0 hours). The large differences in the magnitude of stiffness increase amongst the various mixtures is probably due to differences in temperature susceptibilities of the bitumens as well as differences in the mitigating effects of aggregates on the ageing of bitumens. Nevertheless, the data clearly indicate that the oven storage period is an important factor when preparing representative bituminous mixture specimens in the laboratory.

The test results comparing the stiffness moduli of the specimens mixed in a plant and compacted on site with those of the laboratory prepared specimens clearly indicate that some oven ageing of the loose mixture was warranted for continuously-graded mixtures but that none was necessary for gap-graded mixtures. This difference is considered to be caused by factors such as bitumen content of the mixture, bitumen film thickness and void content of the compacted mixture.

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	Mean stiffness for the field samples = 1770 MPa with a standard deviation of 72.5MPa and a coefficient of variation of 4.1%			
F7	3.5	1760				
F8	3.2	1880				

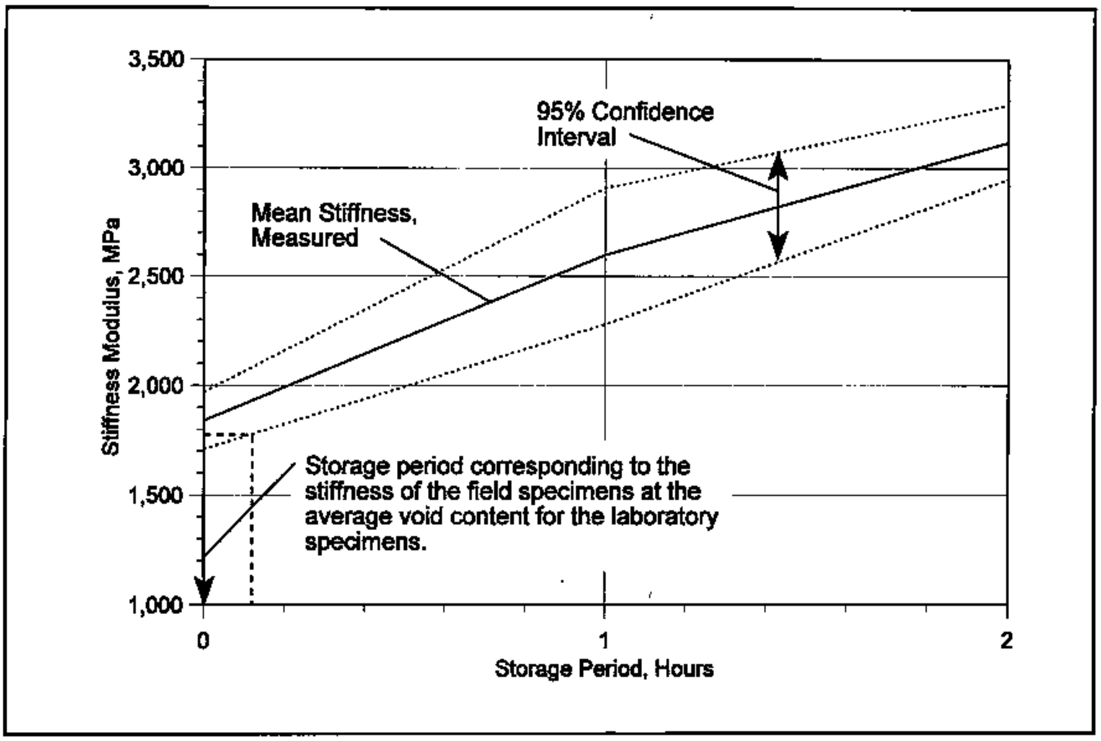

Figure 3.7. Test Results for the 30/10 HRA Laboratory Specimens.

Table 3.5. Summary of Results for the 30/14 HRA Wearing Course Materials (Batch 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	1.9	2020	0	2.5	2170	2070 - 2270
F2	2.1	2070	1	2.9	2840	2570 - 3100
F3	2.1	1790	2	3.3	3220	3040 - 3400
F4	2.0	2230				
F5	2.5	1870				
F6	1.8	2200				
F7	2.7	2970				
F8	3.8	2300				
F9	2.3	2310				

Mean stiffness for the field samples = 2194MPa with a standard deviation of 343MPa and a coefficient of variation of 15.6%.

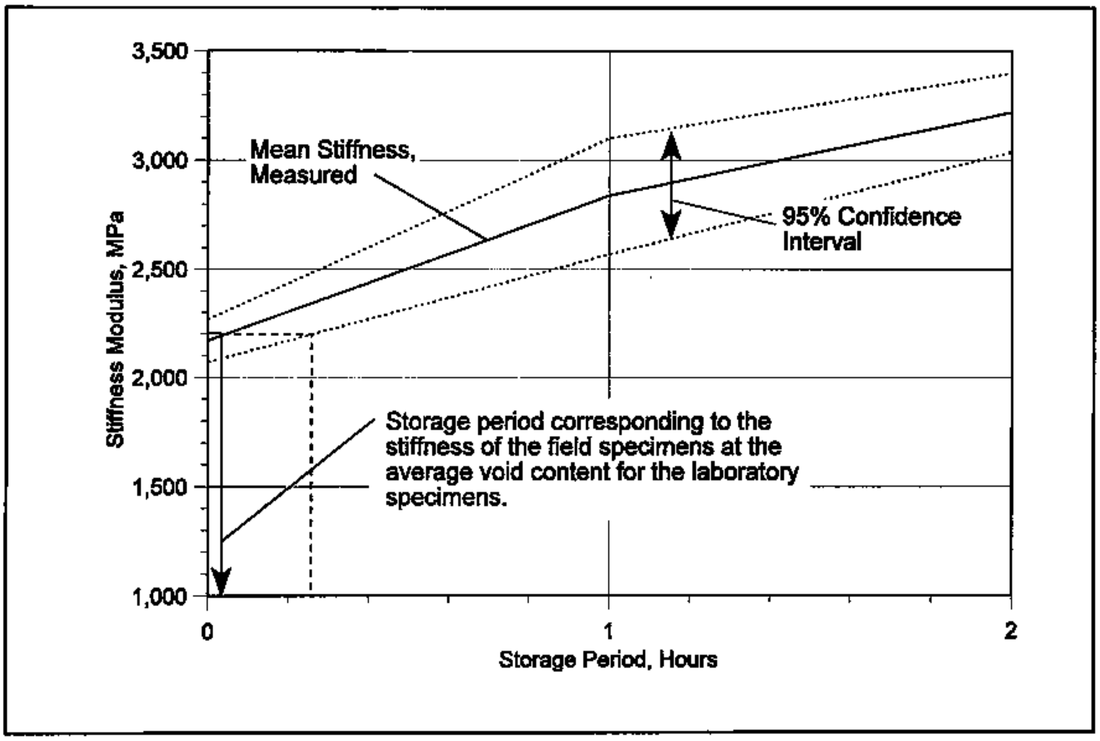

Figure 3.8. Test Results for the 30/14 HRA Laboratory Specimens from the Batch Plant.

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

Field Specimens			Laboratory Specimens			
					Stiffness (MPa)	
Sample ID	Void Content (%)	Stiffness (MPa)	Storage Period (Hours)	Average Void Content (%)	Mean	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%.

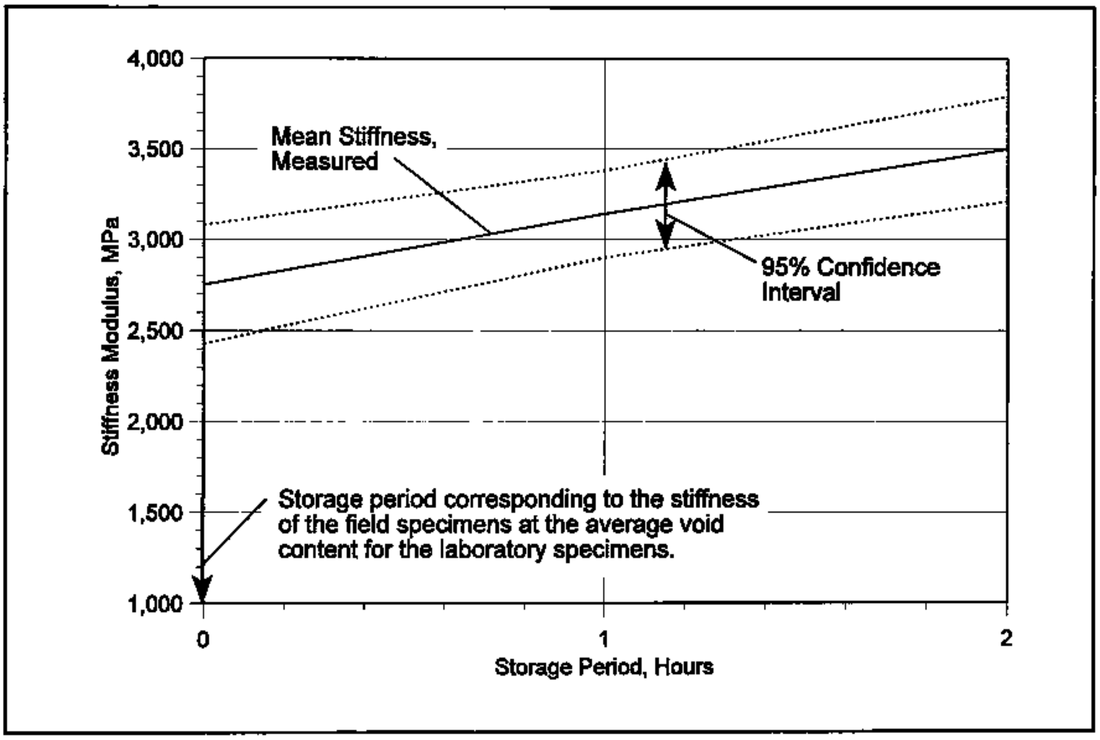

Figure 3.9. Test Results for the 30/14 HRA Laboratory Specimens from the Drum Mixer Plant.

3.8 CONCLUSIONS

Based on the results reported above, the following conclusions appear to be warranted:

1) Storage of loose mixtures at 135 °C prior to compaction significantly affected the stiffness moduli of the compacted mixtures. It was shown that as little as 1 hour of exposure resulted in stiffness increases of 10 to 56%.
2) Due to the sensitivity of loose mixtures to storage in ovens at elevated temperatures prior to compaction, it can be concluded that a standardised procedure is necessary for the preparation of uniform and representative laboratory test specimens.
3) The investigation provided strong evidence to show that storing the loose continuously-graded mixtures in a laboratory oven for a period of around 2 hours at 135 °C was representative of the ageing which occurred during the mixing, storage and transport of the mixtures in actual construction practice.
4) The investigation provided strong evidence to show that storing the loose gap-graded mixtures in an oven was not necessary when fabricating test specimens in the laboratory to be representative of mixtures produced in batch plants. However, compared with field-compacted specimens from drum mixer plants, laboratory-mixed materials with no ageing resulted in specimens having stiffness moduli far greater than those of the field specimens.

3.9 REFERENCES

1. "The Percentage Refusal Density Test," TRRL Contractor Report No.1, Transport and Road Research Laboratory, Department of Transport, 1987.

2. "Method for the determination of the indirect tensile stiffness modulus of bituminous materials," DD 213, British Standards Institution, 1993.

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 ageing in the laboratory must, therefore, compress many years of time into a few hours or days. In other words, for a simulative method to be practical, it must be completed in a reasonably short period and it must provide an accurate and reproducible estimate of the changes in the properties of the binder that approximate changes in the same properties during many years of service.

Findings from the literature review indicate that the majority of tests developed to simulate long-term ageing attempt to do so through accelerated ageing of the neat bitumen. This is usually accomplished through elevated temperatures, high pressures, thin films or in oxygen-rich environments and may incorporate a combination of several or all of these treatments. Although most of these tests are attractive in that they are carried out in a short time frame, none take into account the effect of mineral aggregates, which have been shown to influence the ageing characteristics of bitumens (see Chapter 5).

It is the Author's opinion that a test carried out in the laboratory should reproduce, as close as possible, the in situ conditions it attempts to simulate. Thus, a laboratory test which attempts to simulate long-term ageing of bituminous mixtures should be carried out on mixtures, not just neat bitumens, thereby including the influence of mineral aggregates. Although several tests have been developed for mixtures, the long-term ageing test developed for the Strategic Highway Research Program (1) appeared, at the time the literature review was first written (December, 1993), to be an appropriate method. Thus, based largely on this method, the *Standard Practice for Long-Term Oven Ageing of Compacted Bituminous Mixtures* (Appendix A) was adapted to UK mixtures for use on the Bitutest project. The following section presents a summary of the work carried out to evaluate the efficacy of the method.

4.2.2 Evaluation of Long-Term Ageing Protocol

Mixtures Evaluated

Evaluation of the long-term oven ageing procedure concentrated primarily on hot rolled asphalt (HRA) wearing course mixtures as it was presumed that this layer would be the most affected by age hardening. Mixture parameters such as binder 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

Evaluation Methodology

The basic methodology for evaluating the ageing procedure was to carry out the procedure on mixtures representative of properly constructed pavement materials as well as on mixtures not constructed to specification (i.e., with a lean binder content and/or high void content). Thus, tests were carried to determine if the procedure was sensitive to differences in volumetric proportions of binder and air voids, both of which are known to influence the ageing susceptibility of bituminous mixtures. Evaluation of the efficacy of the procedure was based on the magnitude of the increase in stiffness modulus of the mixture as represented by the ratio of the stiffness modulus after ageing to the stiffness modulus before ageing, referred to as the stiffness modulus ratio or ageing index. Ageing of the mixtures consisted of exposure to 85 °C for 120 hours in a forced-draft oven in the absence of light and stiffness modulus tests were carried out in accordance with BS DD 213 (3).

Test Results

A summary of the stiffness modulus ratios for all of the mixtures tested is given in Figure 4.1. The data indicate that the accelerated ageing procedure has a minimal effect on hot rolled asphalt mixtures representative of properly constructed pavement materials but a fairly significant effect on those not constructed to specification. For example, the data indicate that the average stiffness for the three 30/14 hot rolled asphalt mixtures with a design binder content (i.e., Mixtures 1, 2 and 3) increased by less than 25% even though one of these (Mixture 3) had a fairly high void content of 4.8%, whereas the stiffness increase for the 30/14 HRA mixture with a lean binder content (Mixture 4) was about 50%. For the two 30/14 HRA mixtures with equivalent air voids (Mixtures 3 and 4), the mixture with the lean binder content (Mixture 4) was affected more by the accelerated ageing procedure.

The ageing procedure clearly affected the dense bitumen macadams more than it did the hot rolled asphalts; for the DBM mixtures, the stiffness modulus increased by a minimum of about 80%. The increase in stiffness modulus appears to have been influenced by the mineral aggregates in the mixtures. For example, Mixture 5, which contained a gritstone aggregate, asphalt sand and limestone filler, increased in

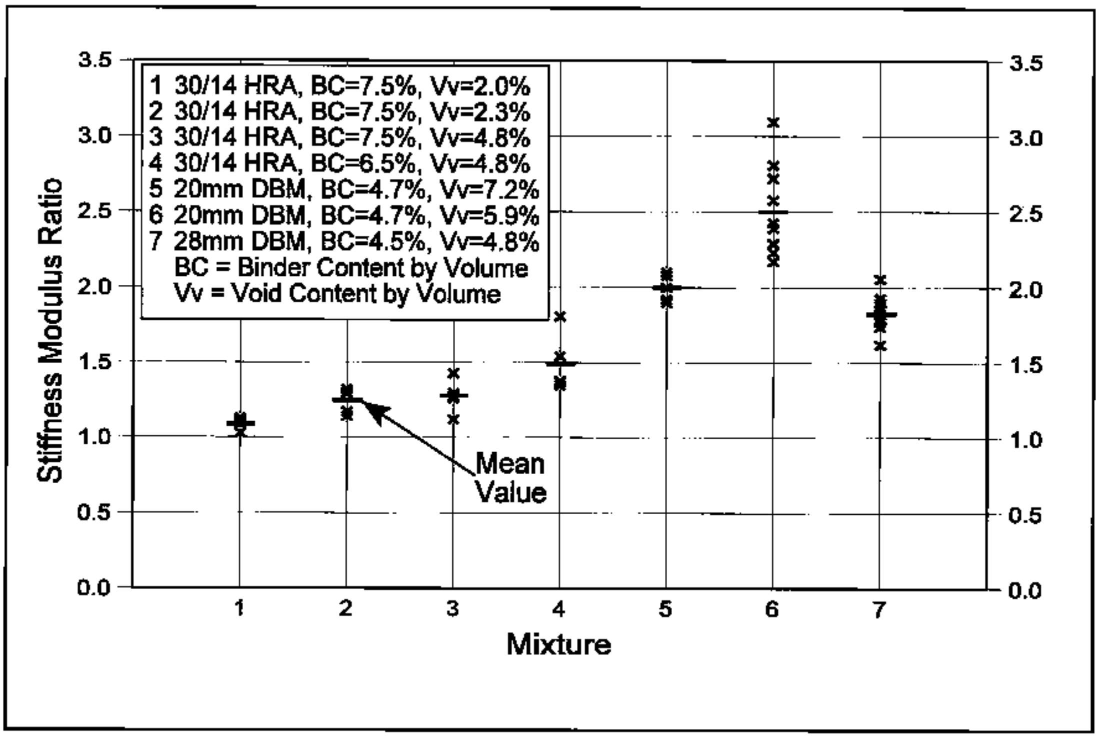

Figure 4.1. Summary of Stiffness Modulus Ratios for the Mixtures Used to Evaluate the Long-Term Ageing Protocol.

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 mixtures that are representative of pavement materials not constructed to specification (i.e., those with a lean binder content and/or high air voids). Therefore, the method appears sufficiently sensitive to differences in volumetric proportions of binder and air voids in the mixture to be confidently used for comparative purposes (e.g., for end-product specification testing). Although the method has yet to be validated on typical UK mixtures, Bell et al (4) found that, for typical US mixtures, 4 days of oven ageing at 85 °C was representative of approximately 15 years of field ageing in climates not dissimilar to those found in the UK.

4.3 WATER SENSITIVITY

4.3.1 Development of Water Sensitivity Protocol

Water sensitivity is concerned with the susceptibility of bituminous mixtures to damage due to moisture—water and moisture are used synonymously here to mean water in its liquid phase, as opposed to water vapour or moisture vapour. Unlike long-term ageing, which is manifested in progressive stiffening of the binder over a long period, water damage can occur almost immediately after construction and is usually manifested in a decrease in mixture stiffness. Nevertheless, the criteria for an appropriate test method are the same. That is, the test must be able to be completed in a reasonably short period and it must provide an accurate and reproducible estimate of the changes in the properties of the mixture that approximate changes in the same properties of the mixture in situ.

Numerous tests have been developed in an attempt to meet this goal but none, as yet, has been universally accepted as a suitable method. However, several methods, some of which have enjoyed widespread use, appear to reasonably satisfy the above criteria. In particular, the Lottman method (5) and one of its variations, the Tunnicliff-Root method (6), and the method developed for SHRP (7) all deserve consideration. Although each of these methods have particular strengths, none has been able to accurately predict mixture performance. The *Test Method for Measurement of the Water Sensitivity of Compacted Bituminous Mixtures* (see Appendix A), referred to herein as the water sensitivity protocol, combines the strengths of the above methods 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

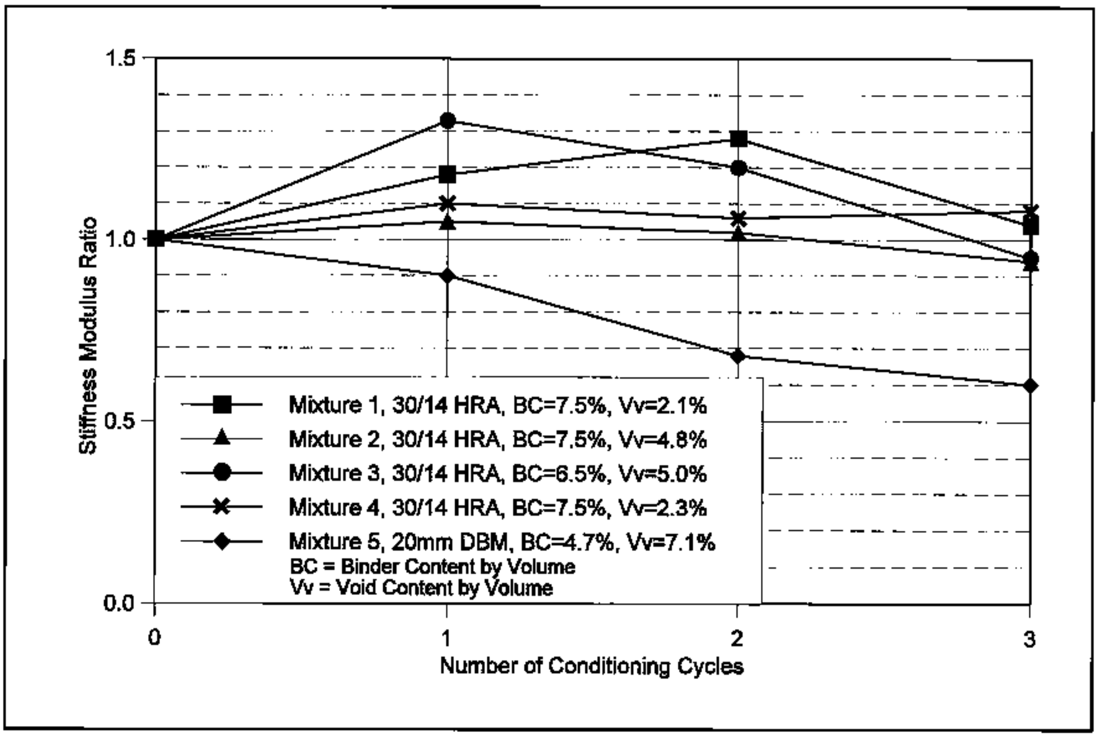

Figure 4.2. Summary of Results for the Mixtures Used to Initially Evaluate the Efficacy of the Water Sensitivity Protocol.

mixtures had relatively high air voids (Mixtures 2 and 3) and one of these had a binder content 1% less than design (Mixture 3). However, it would appear that the onset of moisture damage occurred after an initial increase in stiffness modulus for three of the 30/14 HRA mixtures. It is evident from these results that the procedure clearly induced damage in the DBM mixture.

The apparent onset of moisture damage to three of the four 30/14 HRA mixtures, as shown in Figure 4.2, suggests that the mixtures had not been subjected to enough conditioning cycles to induce noticeable moisture damage. This prompted investigation into how the number of conditioning cycles and the duration of the cycles affects the performance of the mixture. Fifteen 30/14 HRA cores from the same slab having a low binder content (1% less than design) were divided into three groups such that the average void content for each group was approximately equivalent. All specimens were tested for stiffness prior to being subjected to partial vacuum saturation, after which the groups were subjected to the water damage regimes indicated in Figure 4.3. Group 1, which formed the control group, was

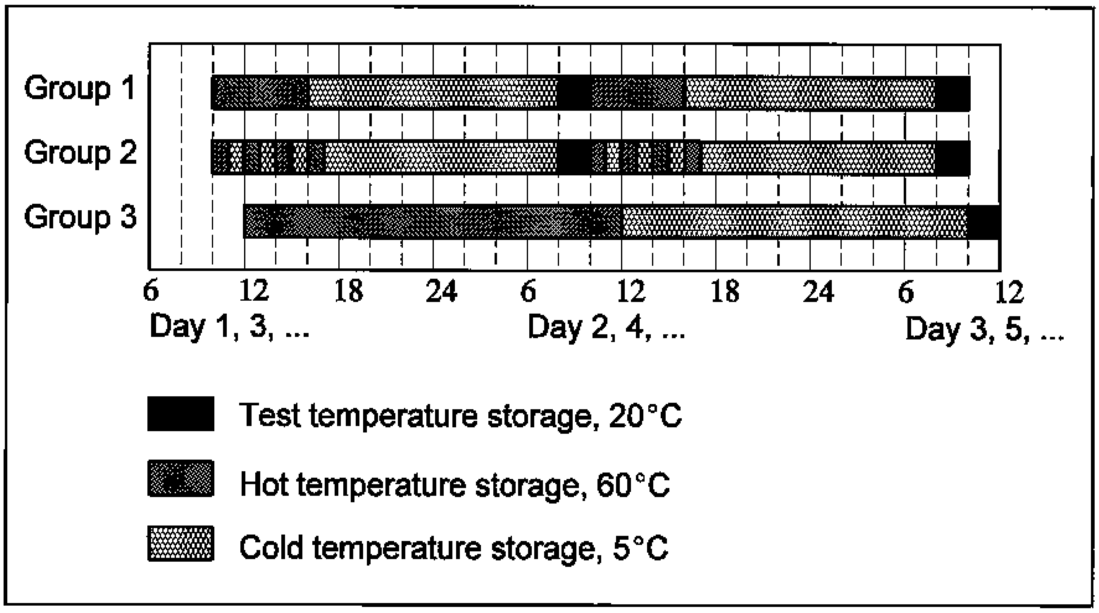

Figure 4.3. Moisture Damage Regimes for the Three 30/14 HRA Mixtures Used to Evaluate the Effects of Thermal Cycling.

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

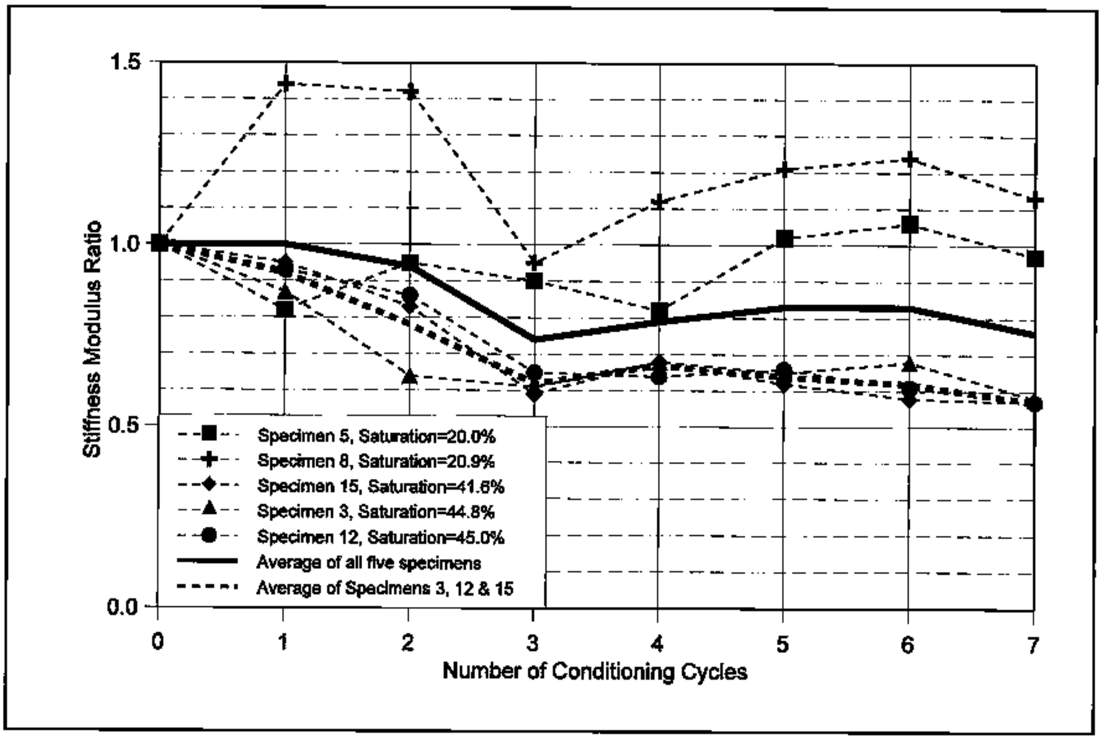

Figure 4.4. Summary of Test Results for the Specimens from Group 1 (Control Group) Used to Evaluate the Effects of Thermal Cycling.

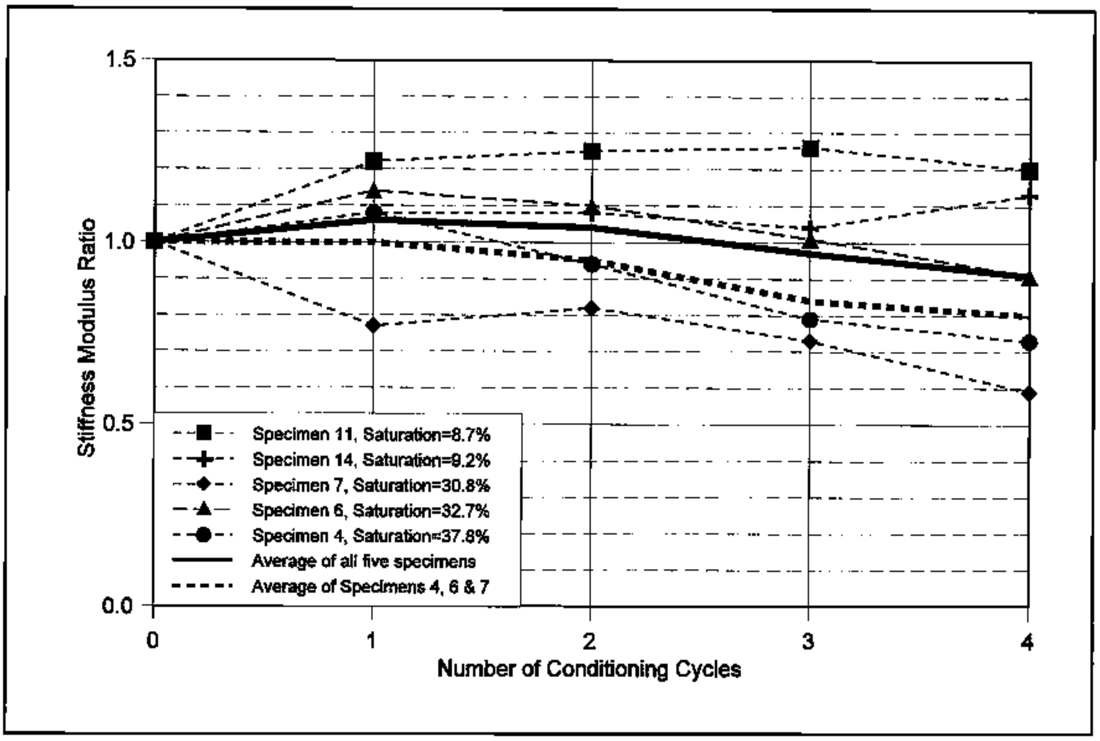

Figure 4.5. Summary of Test Results for the Specimens from Group 2 Used to Evaluate the Effects of Thermal Cycling.

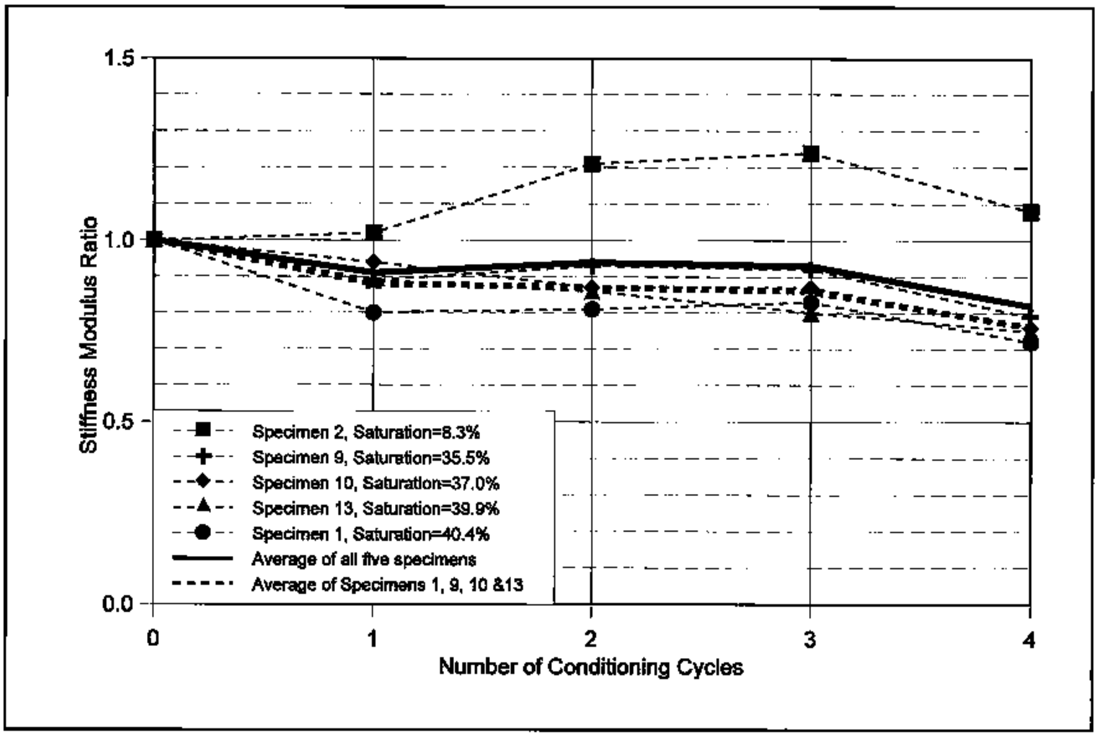

Figure 4.6. Summary of Test Results for the Specimens from Group 3 Used to Evaluate the Effects of Thermal Cycling.

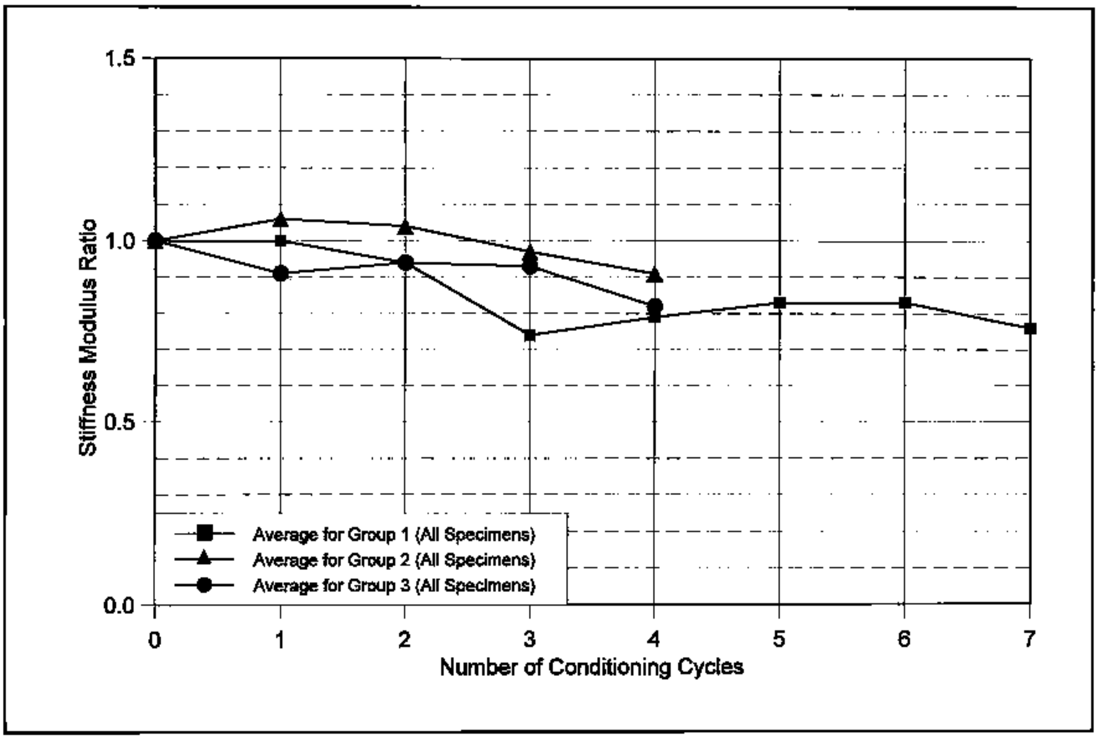

Figure 4.7. Summary of Test Results for the Three Groups of 30/14 HRA Test Specimens Used to Evaluate the Effects of Thermal Cycling.

further moisture damage. However, the results do pose further questions, particularly whether or not the degree of saturation is the dominant factor in causing a decrease in the stiffness of the mixture.

To investigate the importance of the degree of saturation (defined as the volume of void space filled with water, expressed as a percentage), two sets of nominally the same 30/14 HRA mixture, with different void contents, were subjected to different levels and durations of partial vacuum. One set, the control group, was subjected to the water damage regime indicated by the water sensitivity protocol, which calls for a partial vacuum of 510mm Hg for 30 minutes followed by thermal cycling. The other set formed the experimental group. It was also subjected to the water damage regime as indicated by the water sensitivity protocol, except that a much higher partial vacuum (650mm Hg) for a much longer period (60 minutes) was used prior to thermal cycling. This resulted in the two groups of specimens having significantly different initial degrees of saturation as shown in Table 4.2.

Table 4.2. Summary of Specimen Void Contents and Initial Degrees of Saturation for the HRA Mixture Used to Investigate the Importance of the Degree of Saturation.

Group	Partial Vacuum Level & Duration	Sample ID	Void Content (%)	Initial Degree of Saturation (%)
Control	510mm Hg (≈670 mbar) for 30 minutes	BH1-4	1.2	10.9
		BH1-8	2.9	21.9
		BH1-10	2.4	23.2
		BH1-12	1.8	8.9
		BH1-15	3.4	16.7
Experimental	650mm Hg (≈855 mbar) for 60 minutes	BH2-1	5.8	73.2
		BH2-6	7.5	66.2
		BH2-7	7.2	64.6
		BH2-9	7.8	67.9
		BH2-14	5.9	63.6

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.

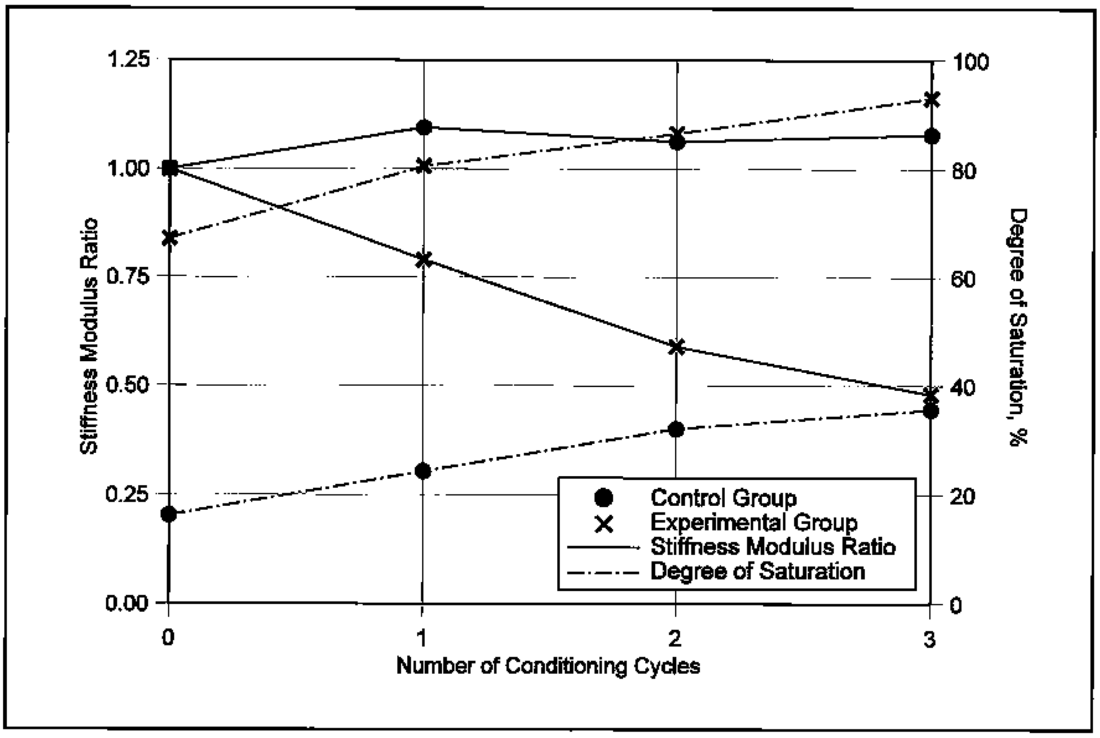

Figure 4.8. Summary of Results for Tests Investigating the Importance of the Degree of Saturation.

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 the disbonding of the bitumen mortar from the stones at the surface of the specimen. Disbonding occurred in both groups but occurred sooner in the experimental group.

Aside from evaluating how the degree of saturation affected moisture damage of HRA mixtures, evaluation of moisture damage to a dense bitumen macadam was also carried out. Two sets of five specimens from the same slab of a 20mm DBM were tested under two conditioning regimes. The sets were grouped such that they had nominally the same average void contents. One set, the control group, was tested in accordance with the water sensitivity protocol. It was initially intended that the second group, the experimental group, be vacuum saturated at the same partial vacuum level as the control group (i.e., 510mm Hg) except for a longer period (60 minutes). However, as indicated in Table 4.3, a longer duration of partial vacuum did not result in an increase in degree of saturation relative to the control group. It was, therefore, decided to subject the experimental group to a conditioning regime similar to that prescribed for the immersion wheel tracking test (8) with the intention of attempting to correlate the two methods should this initial trial indicate the possibility of so doing. The conditioning regime entailed submersion of the specimens for 120 hours in a water bath at 60°C, followed by 24 hours in a water bath at 5°C and, finally, 2 hours in a water bath at 20°C.

Figure 4.9 shows the results of tests for the control group while Figure 4.10 shows the results of tests for the experimental group. As indicated in Figure 4.9, the average degree of saturation for the control group increased by about 10% while the average stiffness modulus decreased by about 40% over the three conditioning cycles. The data shown in Figure 4.10 indicates that the conditioning regime similar to that for the immersion wheel tracking test (IWTT) induced substantial damage to the mixture. The data indicates that the degree of saturation increased by over 45% while the stiffness modulus decreased by over 60%. The severity of damage induced by this conditioning regime, relative to that induced by the water sensitivity protocol, was believed to be too dissimilar to warrant further attempts at correlating the two test methods.

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

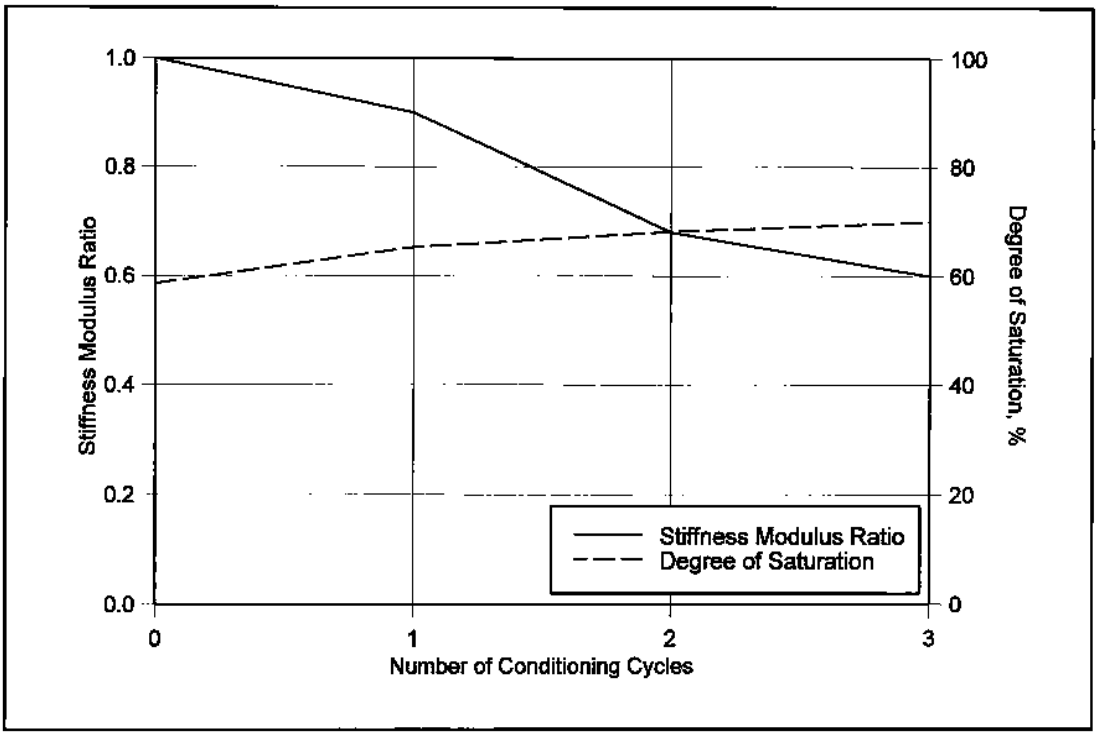

Figure 4.9. Results for the Specimens from the Control Group for Comparison With the Specimens Subjected to the IWTT Conditioning Regime.

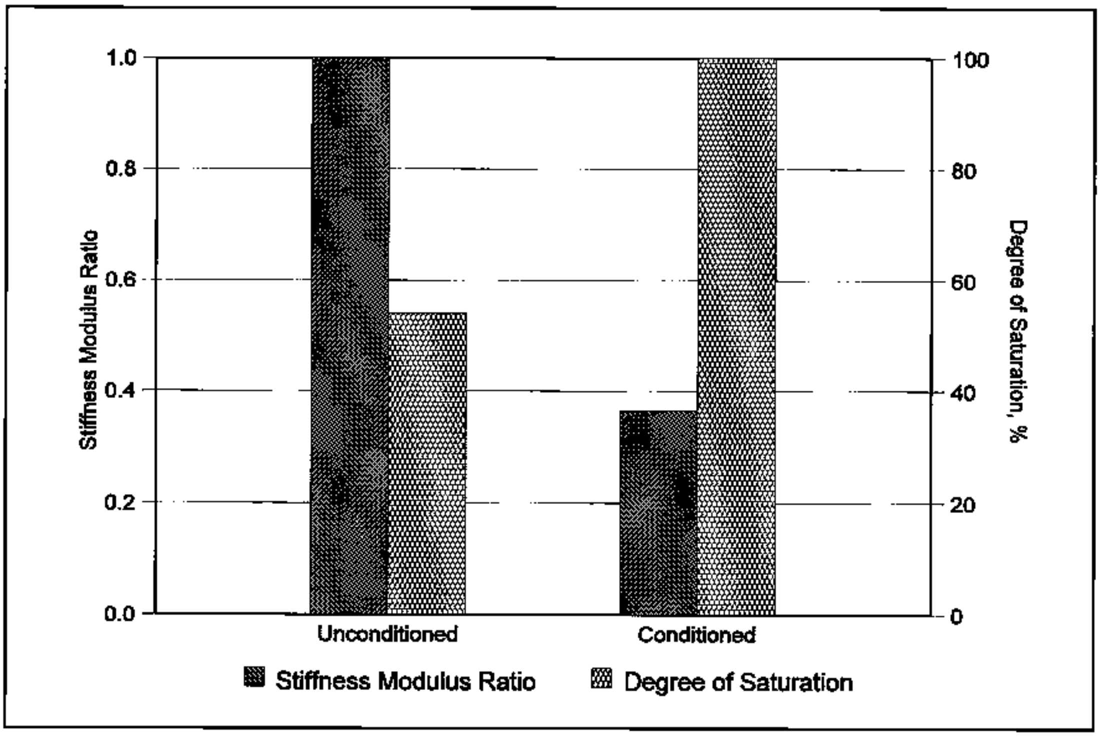

Figure 4.10. Results of Specimens from the Experimental Group Subjected to the IWTT Conditioning Regime.

The evaluations presented above indicate that the partial vacuum ("wetting") and conditioning (thermal cycling) regime as stated in the water sensitivity protocol appears to be reasonable in that, for mixtures that are typically impermeable (e.g., hot rolled asphalt mixtures), the wetting and thermal conditioning regime induced little or no moisture damage, as should be the case. However, for a typically permeable mixture (e.g., dense bitumen macadam), the wetting and thermal conditioning regime induced moderate moisture damage. The results also indicate that moisture damage can be induced in mixtures which are considered durable provided that the wetting and/or conditioning regime is severe (i.e., high initial saturation level and/or long submersion in hot water). Although the procedure appears to induce reasonable degrees of moisture damage to bituminous mixtures, it requires approximately five working days to complete. Furthermore, when several specimens are tested (e.g., more than about five) at the same time, it is difficult to complete all of the required tasks in a normal working day. Bearing in mind these points, an investigation was carried out to determine a simpler, but equally effective, method for inducing water damage in mixture specimens from that specified by the protocol.

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 4.4. Summary of Volumetric Properties and Degrees of Saturation for the Mixtures with Design 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	10	2.283	2.386	4.3	4.4	22.4	26.3
	11	2.275	2.386	4.6		25.0	
	12	2.275	2.386	4.6		29.1	
	17	2.284	2.386	4.3		27.2	
	23	2.284	2.386	4.3		27.3	
	24	2.283	2.386	4.3		27.0	
II	13	2.278	2.386	4.5	4.5	21.4	22.1
	14	2.278	2.386	4.5		21.3	
	18	2.279	2.386	4.5		17.3	
	25	2.278	2.386	4.5		25.7	
	26	2.278	2.386	4.5		21.4	
	27	2.278	2.386	4.5		25.6	
III	15	2.281	2.386	4.4	4.5	29.0	23.4
	16	2.278	2.386	4.5		17.4	
	19	2.275	2.386	4.6		21.2	
	20	2.280	2.386	4.4		25.4	
	21	2.277	2.386	4.6		22.0	
	22	2.277	2.386	4.6		25.6	

The results of tests, expressed as a ratio of conditioned to unconditioned stiffness modulus, are shown graphically in Figures 4.11 and 4.12. The data indicate that, for the mixture with a design binder content, an equivalent amount of damage to that induced by the water senstivity protocol occurred after one conditioning cycle for the Group III specimens and after three conditioning cycles for the Group II specimens (Figure 4.11). It is interesting to note that although all three groups for this mixture had approximately equivalent initial saturation levels, the results for Group III

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.

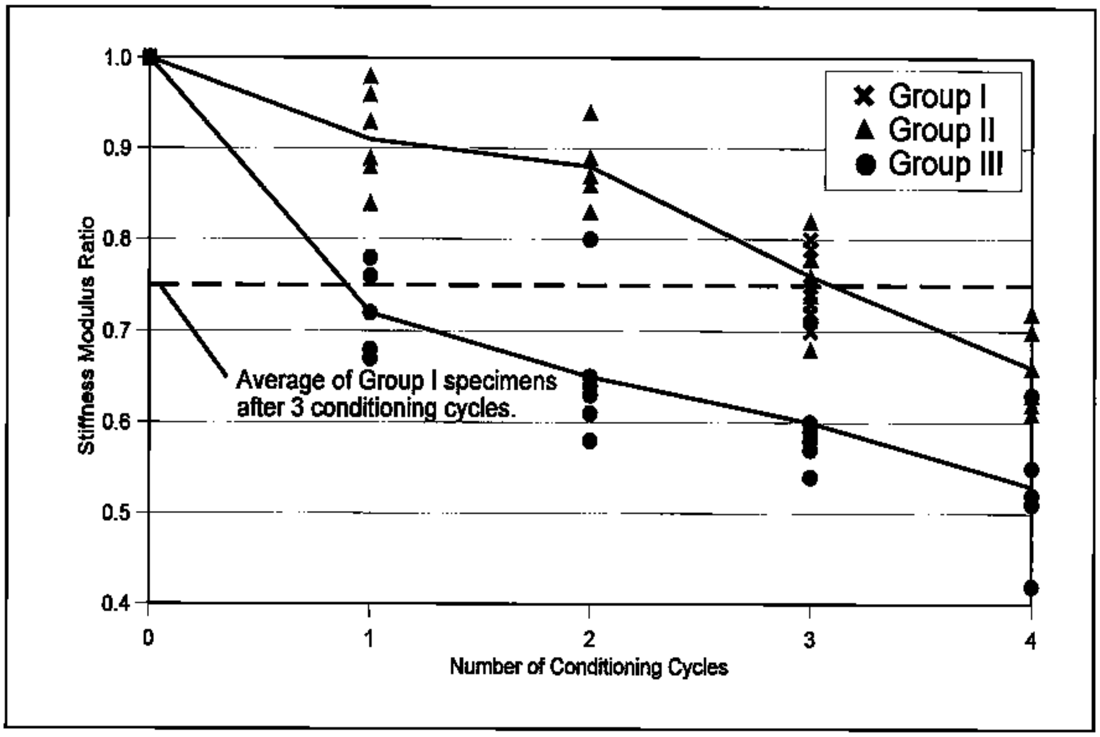

Figure 4.11. Results for the Mixture with Design Binder Content Used to Evaluate the Simpler Test Method.

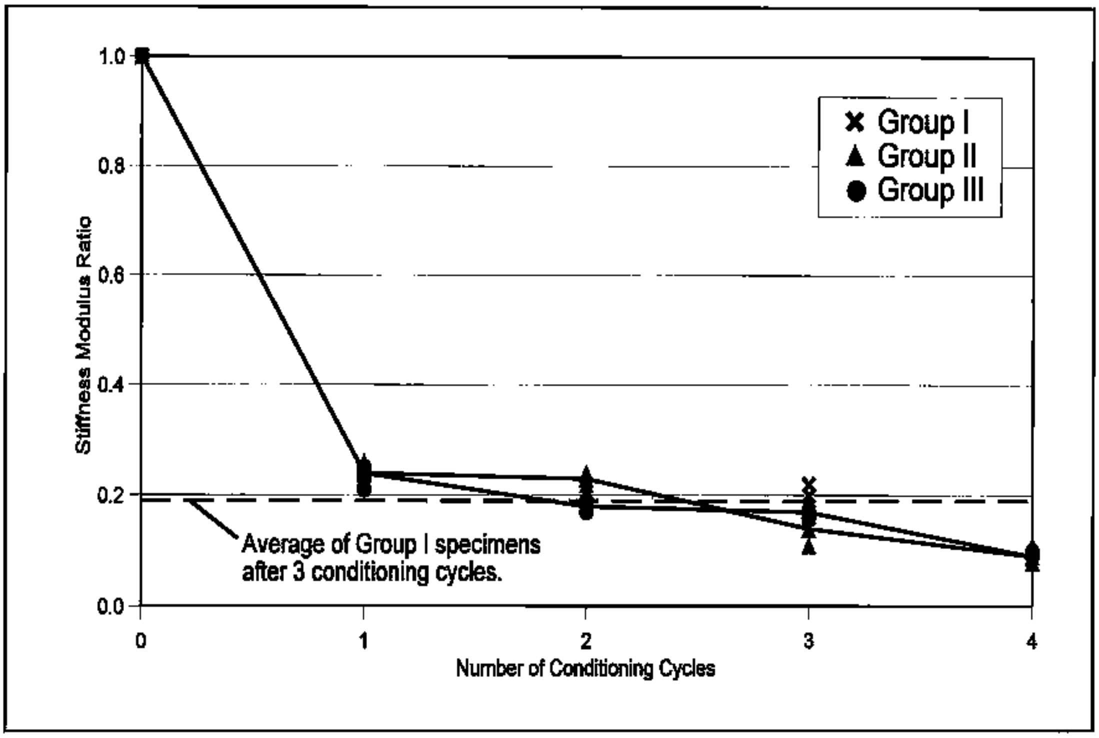

Figure 4.12. Results for the Mixture with Low Binder Content Used to Evaluate the Simpler Test Method.

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 clearly cause damage to mixtures with low binder contents and/or high void contents but not much damage to those representative of properly constructed mixtures. Measuring the indirect tensile stiffness modulus before and after inducing moisture damage appears to be an appropriate means of assessing the magnitude of damage subjected to the specimen in that it appears sufficiently sensitive to variations in volumetric proportions in mixtures. This supports what Lottman found over a decade ago; that loss of bond due to water damage appears to be more readily measured by tensile-type tests (9). Although the water sensitivity protocol has yet to be validated using in-service pavements, the protocol appears to be an adequate tool for comparative purposes (e.g., end-product specification testing).

4.4 REFERENCES

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5 Rheology of Bitumen in Contact With Mineral Aggregate

5.1 INTRODUCTION

Findings from the literature review (Chapter 2) indicate that the physical characteristics of bitumens are affected by the mineral aggregate with which they come into contact (1-5). The findings also indicate that the age hardening of the binder in bituminous mixtures is influenced by both the bitumen and the mineral aggregate (6, 7). Many of these findings, however, are based on studies where measurement of the effects of mineral aggregate were carried out on bitumens obtained from bitumen-aggregate mixtures through solvent extraction, a process which has been found to have a significant effect on the properties of the recovered bitumen (7). Nevertheless, the findings appear sufficiently valid to warrant further investigation of the effects of mineral aggregates on the rheological properties of bitumens.

This chapter describes a novel way in which a dynamic shear rheometer (DSR) was used to measure the rheological properties of bitumen before and after accelerated ageing while in contact with mineral aggregate. The tests were conducted at a very thin gap setting (25µm) in an attempt to measure the ageing effects very near the bitumen-aggregate interface. The experimental programme involved testing three straight-run bitumens and one polymer-modified bitumen coated on five base materials, one of which was stainless steel used as a control or "standard." Evidence is provided to show that mineral aggregates can significantly affect the rheological characteristics and ageing susceptibilities of bitumens.

5.2 DYNAMIC SHEAR RHEOMETRY

Bitumens are a thermoplastic material. At very low temperatures bitumens behave like glass in that they are very elastic but quite brittle. At very high temperatures bitumens behave like a fluid in that they possess viscosity, or the ability to flow when 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.

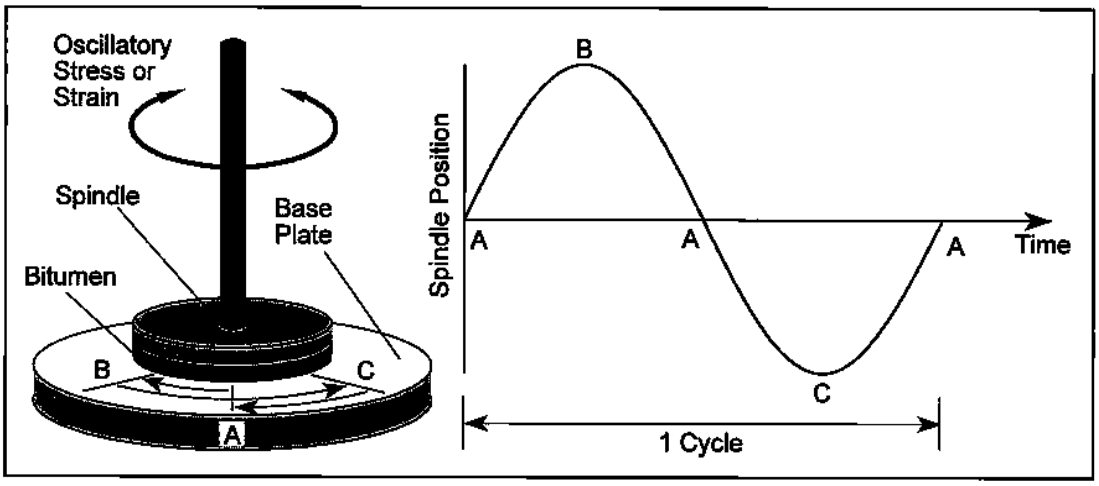
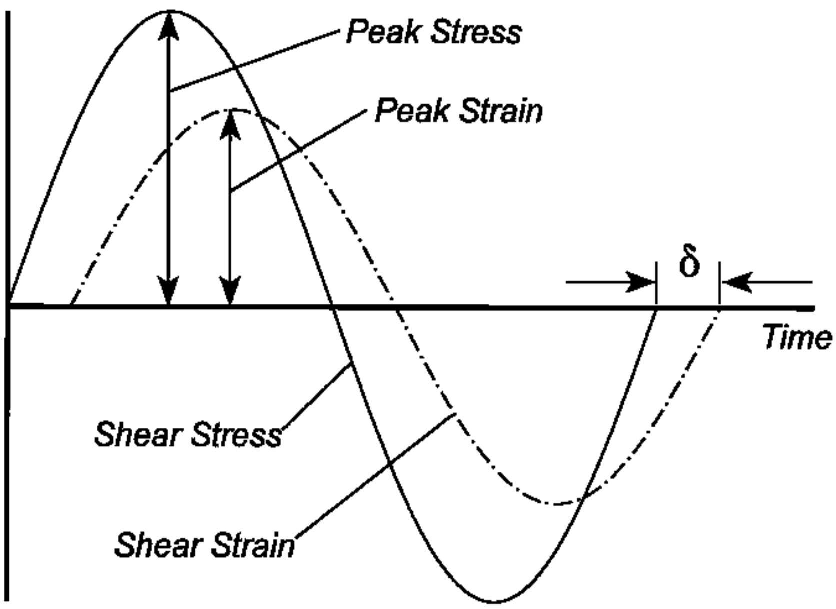


Figure 5.1. Principles of Operation of Torsional-Type Dynamic Shear Rheometers.

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



$$|G^*| = \frac{\textit{Peak Stress}}{\textit{Peak Strain}} = \textit{Complex Shear Modulus}$$

$$G' = |G^*| \cos \delta = \textit{Storage (Elastic) Modulus}$$

$$G'' = |G^*| \sin \delta = \textit{Loss (Viscous) Modulus}$$

$$\tan \delta = \frac{G''}{G'} = \textit{Loss Tangent}$$

$$|\eta^*| = \frac{|G^*|}{\omega} = \textit{Dynamic Viscosity}$$

where:
δ = phase angle
ω = angular frequency

Figure 5.2. Definitions of Moduli Obtained from Dynamic Shear Rheometry Tests.

behaviour. That is, the vertical axis represents completely viscous or fluid-like behaviour such as when bitumen is at very high temperature whereas the horizontal axis represents completely elastic or glass-like behaviour such as when bitumen is at very low temperatures. It should be noted that, at moderate to low temperatures, viscous behaviour can be realised through long-term or very slow shear loading and, at moderate to high temperatures, elastic behaviour can be realised through short-term 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.

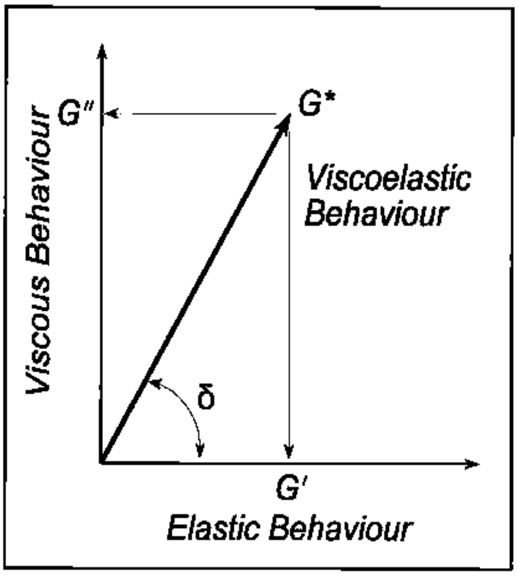


Figure 5.3. Viscoelastic Behaviour of Bitumen.

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

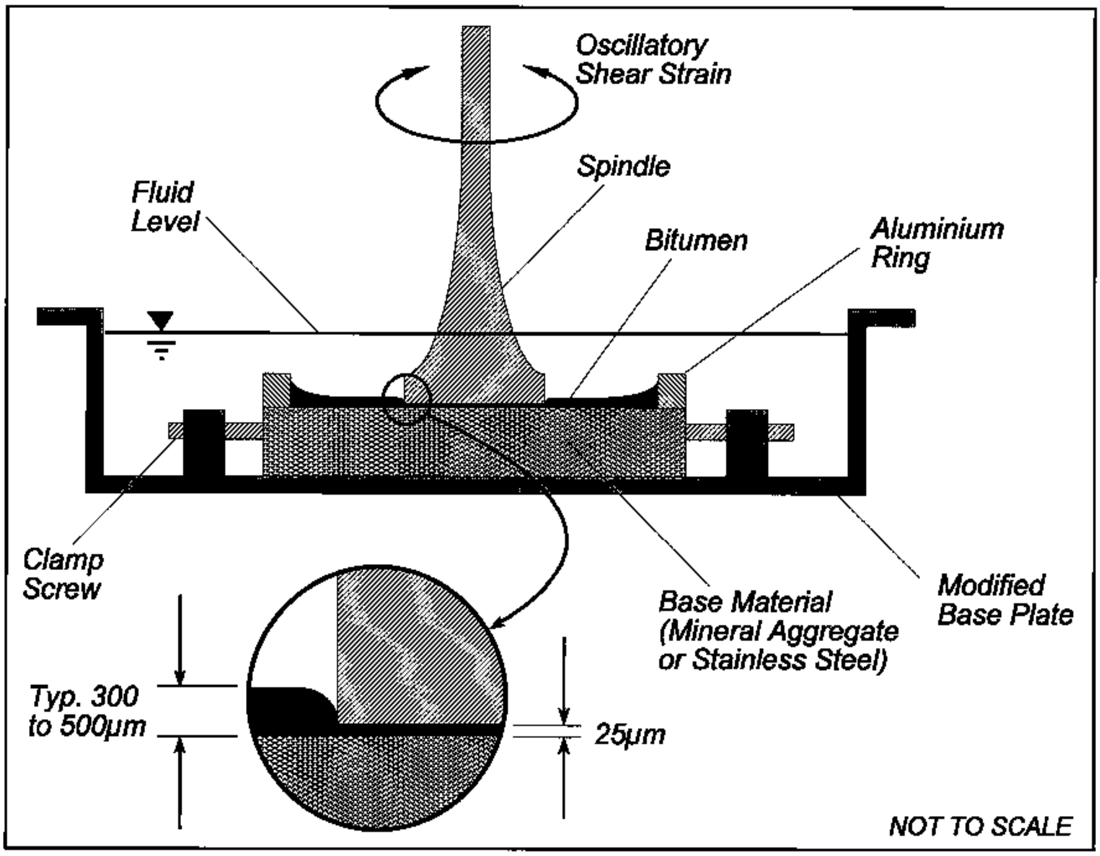


Figure 5.4. Experimental Arrangement Developed for Use in the Dynamic Shear Rheometer.

arrangement shown in Figure 5.4 indicates that, in addition to the usual test parameters associated with ordinary DSR testing (i.e., temperature, frequency, stress or strain amplitude, etc.), tests were conducted at very thin gap settings and bitumen was not trimmed from around the perimeter of the spindle. The need to remove the discs after establishing the zero gap setting (see below) and between tests provided a further potential source of error. The following section describes the efforts undertaken to overcome these problems as well as to develop a set of test conditions which allowed reliable measurements to be made.

5.3.2 Effect of Test Conditions on Measured Properties

Several specific specimen preparation and test conditions were carefully evaluated prior to embarking on the full test programme involving the various bitumens and aggregate types. These included the effects of temperature, strain amplitude, frequency of oscillation, immersion of the spindle in the bitumen and the repeatability 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.

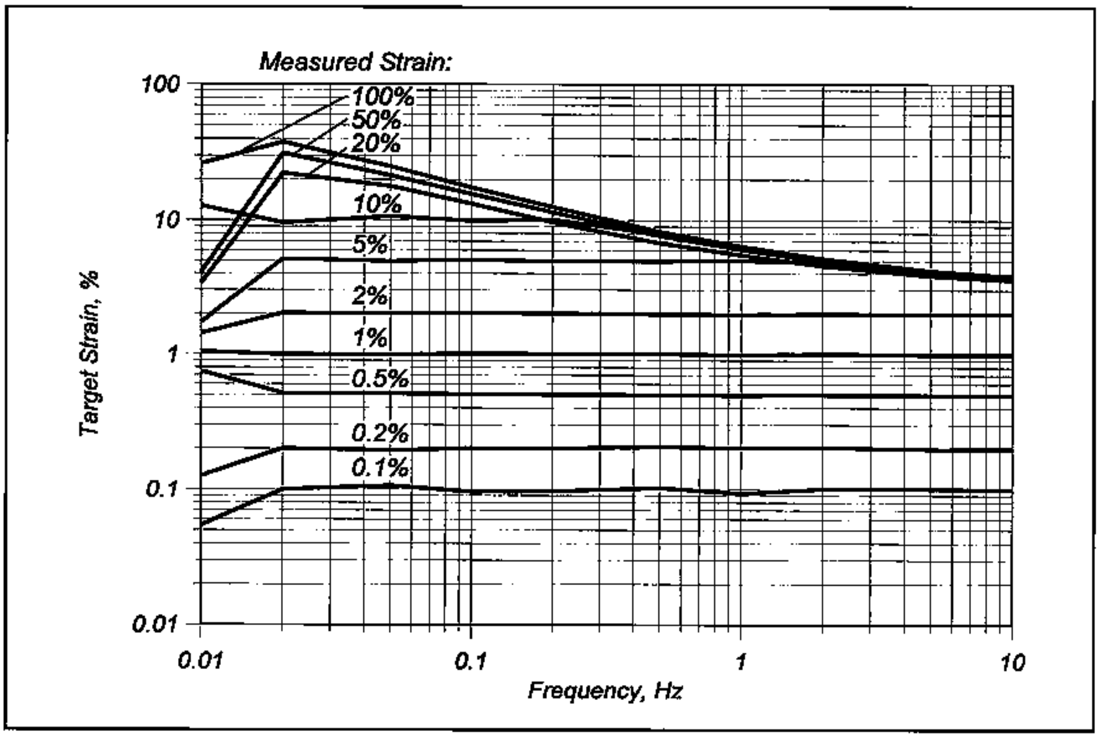


Figure 5.5. Measured Strain Versus Frequency for Various Target Strains at 5°C.

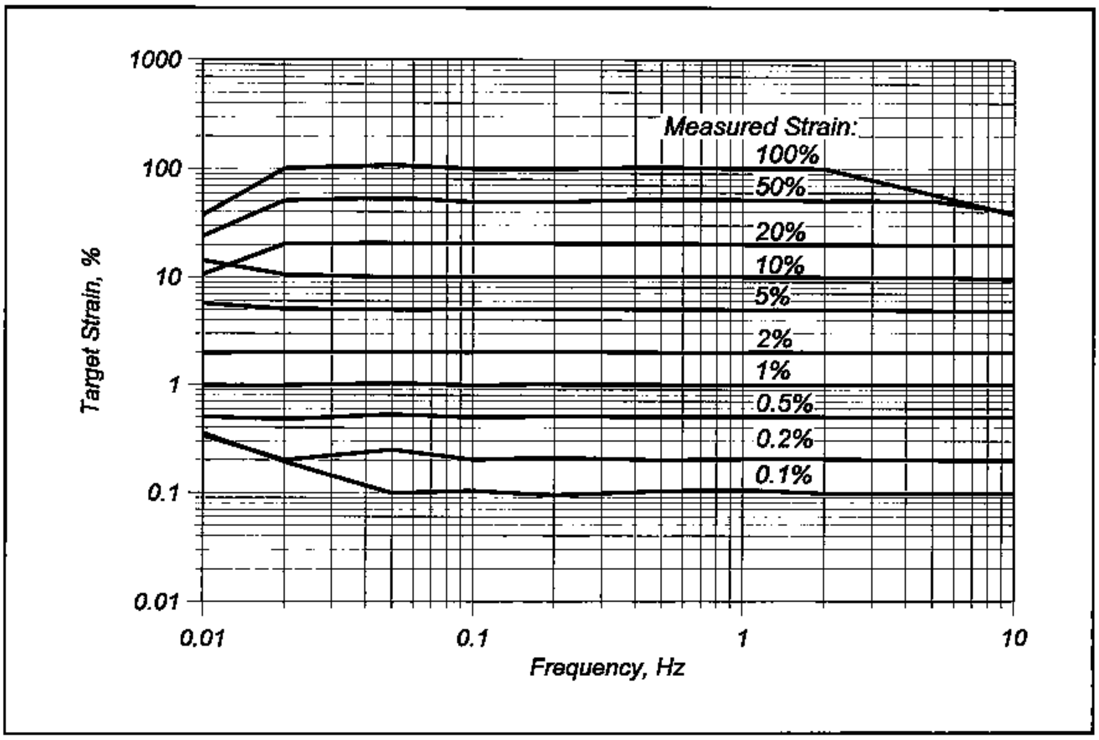


Figure 5.6. Measured Strain Versus Frequency for Various Target Strains at 25°C.

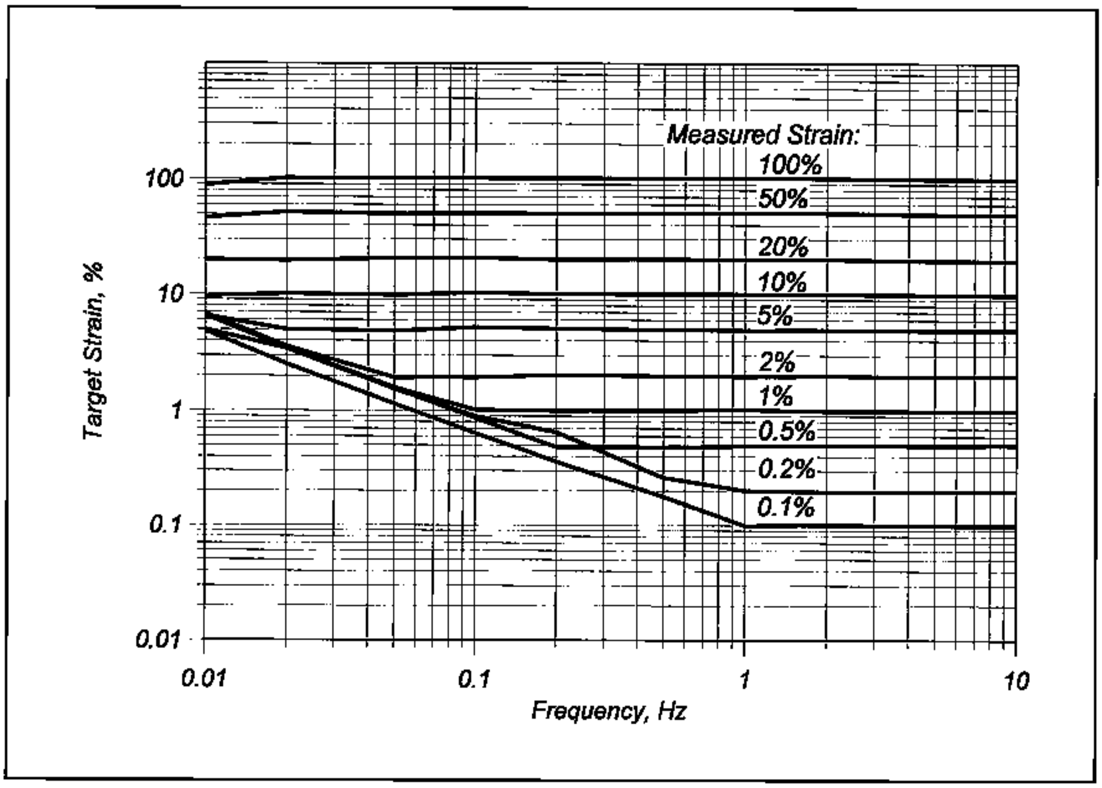


Figure 5.7. Measured Strain Versus Frequency for Various Target Strains at 40°C.

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, coded as AC, CC and DC, respectively. Tests were first conducted on the untrimmed specimen. It was then carefully trimmed and tested again. In both cases, tests were conducted at 25 °C using a gap setting of 25μm. The results are shown graphically in Figures 5.8 and 5.9 which show the effect on the phase angle and the complex shear modulus, respectively. The phase angle test data shown in Figure 5.8 indicate that there was little or only a slight difference between the results of trimmed and untrimmed specimens. Similarly, the complex modulus data in Figure 5.9 indicate that, although there appears to be a slight effect of aggregate, the differences between trimmed and untrimmed specimens appear to be insignificant.

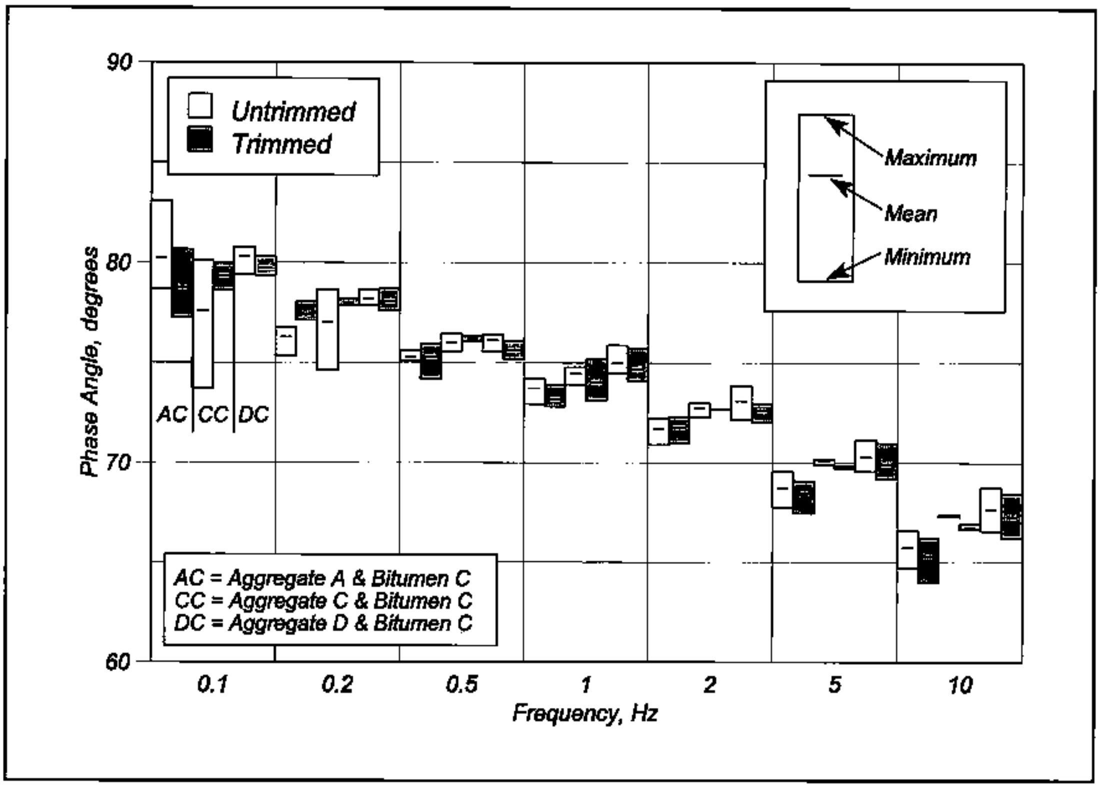


Figure 5.8. Effect of Trimmed Versus Untrimmed Bitumen Specimen on the Phase Angle.

The data were analysed to ensure the above observations were based on statistical evidence. This consisted of using the paired *t* test which is used to test whether or not differences exist between population means which are not independent (9). Use of this test was necessary because the data obtained from the tests carried out on the trimmed specimens were not independent from the data obtained from the untrimmed

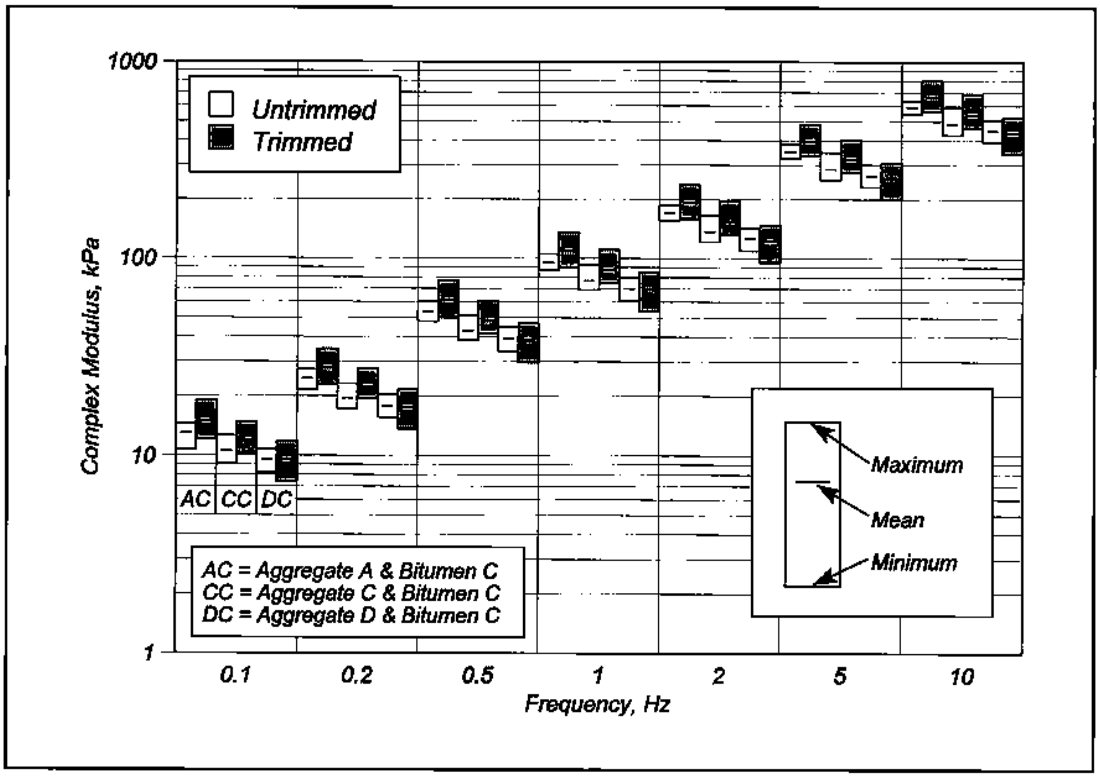


Figure 5.9. Effect of Trimmed Versus Untrimmed Bitumen Specimen on the Complex Shear Modulus.

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., $\mu_1 \neq \mu_2$)

5) Test statistic (paired t statistic):

$$t = \frac{\bar{x}_d}{s_d / \sqrt{n}} \tag{5.1}$$

where:

$\bar{x}_d$ = sample mean difference between phase angles (or complex shear moduli) obtained from tests on trimmed and untrimmed bitumen specimens coated on mineral aggregate

s_d = sample standard deviation of differences

n = number of differences = 3 per aggregate type ∴ degrees of freedom = 2

Reject H_0 in favour of H_a if $|t| > t_{critical}$.

6) A level of significance $\alpha = 0.05$ (i.e., 95% confidence level) was assumed which gave a $t_{critical}$ value of 4.30 (9). Therefore, the null hypothesis (H_0) was rejected in favour of the alternate hypothesis (H_a) if the absolute value of t was greater than $t_{critical}$ (i.e., H_0 rejected in favour of H_a if $t > 4.30$ or $t < -4.30$).

It should be pointed out that the population means (denoted by μ) are true or actual means for the bitumen coated on the various aggregates whereas the sample means (denoted by $\bar{x}$) are only an estimate of the population means.

The results of the paired t tests carried out on the data are shown in Table 5.1. The results indicate that, for all three mineral aggregate types, the mean complex moduli for untrimmed bitumen specimens were not statistically different from the mean complex moduli for trimmed bitumen specimens. The same is true for the phase angle results except at the 10Hz oscillation frequency for Aggregates C and D. These results provide strong evidence that trimming the bitumen specimen is not necessary as the same result (statistically) can be obtained from an untrimmed specimen when tests are carried out at 25°C and using a gap setting of 25μm.

Repeatability of Establishing the Gap Setting

Considerable attention was paid to establishing a consistent gap setting which is the distance between the spindle and base material and defines the thickness of bitumen

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 α = 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 spindle. The results also indicate that the gap between the spindle base and lower disc could be set with reasonable accuracy and at a very thin gap setting of 25μm, the results of tests on specimens that had not been trimmed were not significantly different from those that had, except for the phase angle test results for Aggregates C and D at a frequency of 10Hz.

Table 5.2. Repeatability of Establishing the Zero Gap Setting Using Removable Discs.

Disc	Vernier Reading, μm (adjusted to minimum)	Sample Standard Deviation, μm	95% Confidence Interval for Ten Attempts	95% Confidence Interval for One Attempt
1	5, 10, 10, 5, 5, 5, 10, 5, 0, 5	3.16	$\bar{x} \pm 2.3\mu m$	$\bar{x} \pm 6.2\mu m$
2	5, 10, 5, 5, 5, 10, 10, 10, 10, 5	2.64	$\bar{x} \pm 1.9\mu m$	$\bar{x} \pm 5.2\mu m$
3	5, 5, 5, 5, 0, 5, 5, 5, 5, 5	1.58	$\bar{x} \pm 1.1\mu m$	$\bar{x} \pm 3.1\mu m$
4	15, 5, 15, 5, 0, 10, 0, 10, 10, 5	5.40	$\bar{x} \pm 3.9\mu m$	$\bar{x} \pm 10.6\mu m$
5	0, 5, 0, 5, 5, 5, 0, 0, 0, 5	2.64	$\bar{x} \pm 1.9\mu m$	$\bar{x} \pm 5.2\mu m$
6	5, 10, 5, 5, 5, 5, 0, 5, 5, 5	2.36	$\bar{x} \pm 1.7\mu m$	$\bar{x} \pm 4.6\mu m$

5.4 EXPERIMENT DESIGN

Having established that the novel experimental arrangement (Figure 5.4) could be used to obtain reliable results from dynamic shear modulus tests, a programme was formulated to investigate how mineral aggregates affect the rheological properties and ageing susceptibilities of bitumens. The details of this test programme are discussed in the following paragraphs.

5.4.1 Overview of Test Programme

Samples of bitumens were coated on discs of mineral aggregates and also of stainless steel. Each combination was tested in the DSR to obtain the rheological properties of 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.

Table 5.3. Inorganic Compositions and Water Absorptions of the Mineral Aggregates.

Compound	Percent of compound present in Granite (Aggregate A)	Limestone (Aggregate C)	Greywacke (Aggregate D)	Basalt (Aggregate E)
SiO_2	51.6	1.04	63.2	49.0
Al_2O_3	24.9	0.25	13.8	16.7
Fe_2O_3	19.3	0.07	7.04	0.98
TiO_2	0.48	—	1.88	—
MnO_2	0.21	—	0.19	0.15
CaO	—	55.4	3.04	11.6
MgO	3.4	0.05	3.07	0.22
SO_3	0.13	—	0.50	—
Water Absorption, %	0.7	0.8	1.2	0.8

The bitumen designated as C was a 200pen bitumen from a different source. Bitumen D was a bitumen from the same crude oil source as Bitumens A and B but modified with the styrene-butadiene-styrene (SBS) polymer. Details are summarised in Table 5.4.

5.4.4 Specimen Preparation

The mineral aggregate discs were cut from 23mm diameter cores extracted from "football-sized" boulders of the raw materials. The saw was able to cut them to a uniform thickness of 4mm and to within 10μm of being parallel across any given diameter. The stainless steel discs were manufactured to similar dimensions and surface polished to provide at least as good parallelism as the mineral aggregate discs. After fabrication, the discs were washed in acetone (propanone) which was

Table 5.4. Summary of Bitumens Used in the Test Programme.

Bitumen	Grade (pen)	Crude Oil Source
A	200	1
B	50	1
C	200	2
D	SBS-modified	1

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 coat the aggregate surface. Immediately after coating, the discs were transferred to a level steel plate which served as a heat sink to effect rapid cooling.

When the coated discs cooled to room temperature, they were weighed again. They were then placed in the DSR and a procedure similar to that used to establish the zero gap setting was used to determine the approximate thickness of bitumen. It was initially intended to develop a relationship between added mass of bitumen and resulting bitumen thickness but this did not prove possible, probably due to the different porosities of the aggregates and, therefore, different quantities of bitumen absorbed.

At this point the coated discs were ready for testing in the DSR. Each test commenced within 12 hours of having coated the disc.

5.4.5 Test Conditions

Based on the preliminary experiments conducted while developing the test method, the following test conditions were used throughout the programme:

- Temperature: 25°C
- Gap setting: 25μm
- Strain amplitude (controlled-strain test): 1%
- Frequencies of oscillation: 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1, 2, 5 and 10Hz

5.4.6 Number of Test Specimens

It was originally intended that three specimens for each bitumen-aggregate combination be tested. However, for several reasons this was not always possible but, as shown in Table 5.5, at least one specimen from each combination was successfully tested.

5.5 TEST RESULTS

The test results are tabulated in Appendix C. A typical example is shown in Figure 5.10. As expected, the results indicate an increase in complex modulus accompanied by a decrease in phase angle with increasing oscillation frequency. The 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.

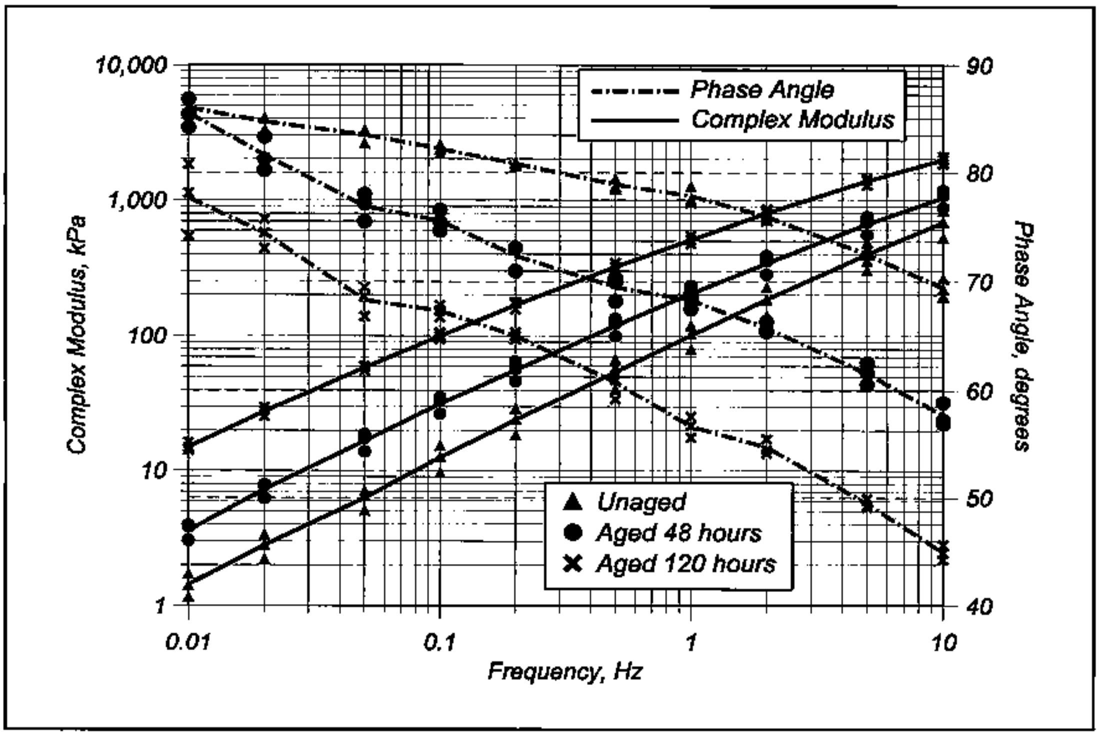


Figure 5.10. Typical Results from Tests Conducted in the DSR on Bitumen A Coated on Aggregate C.

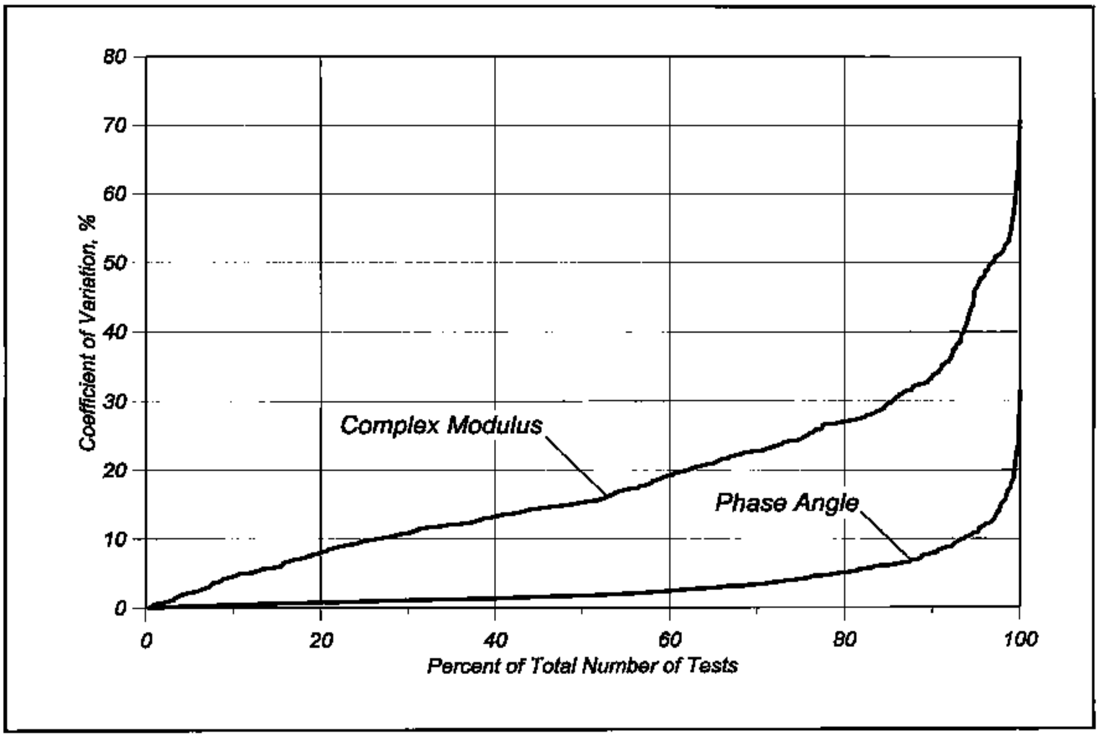


Figure 5.11. Test Variability as Indicated by the Coefficient of Variation.

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.

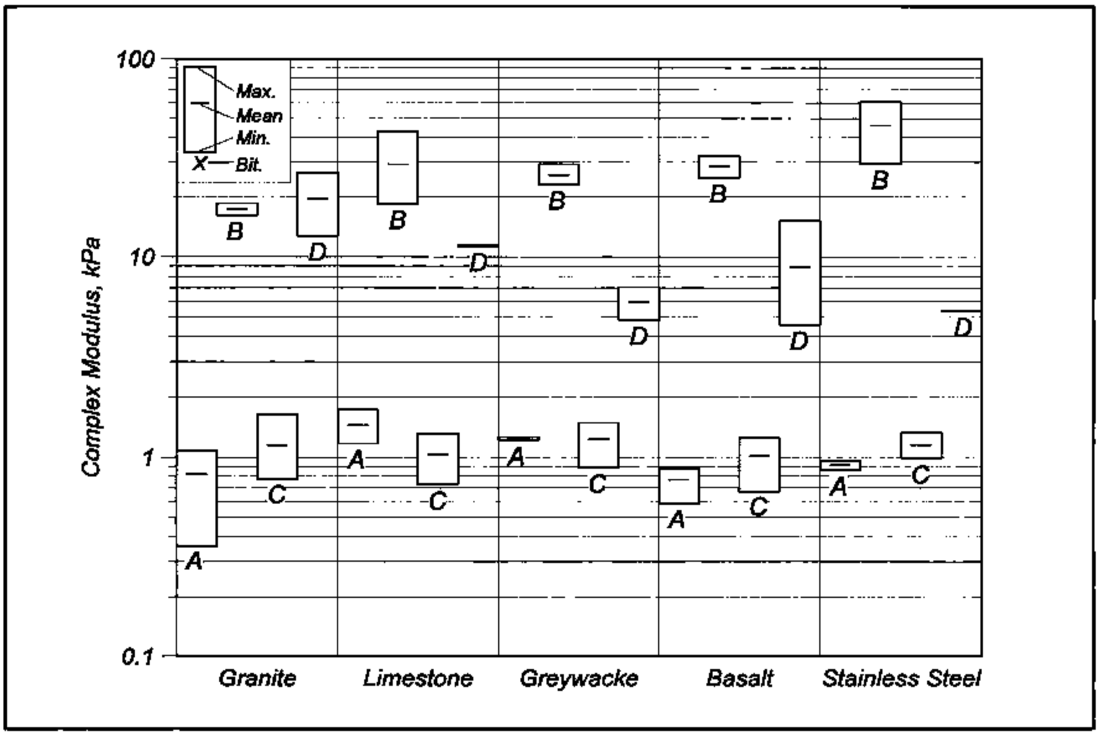


Figure 5.12. Effect of Bitumen-Aggregate Interaction on the Complex Modulus at a Frequency of Oscillation of 0.01Hz.

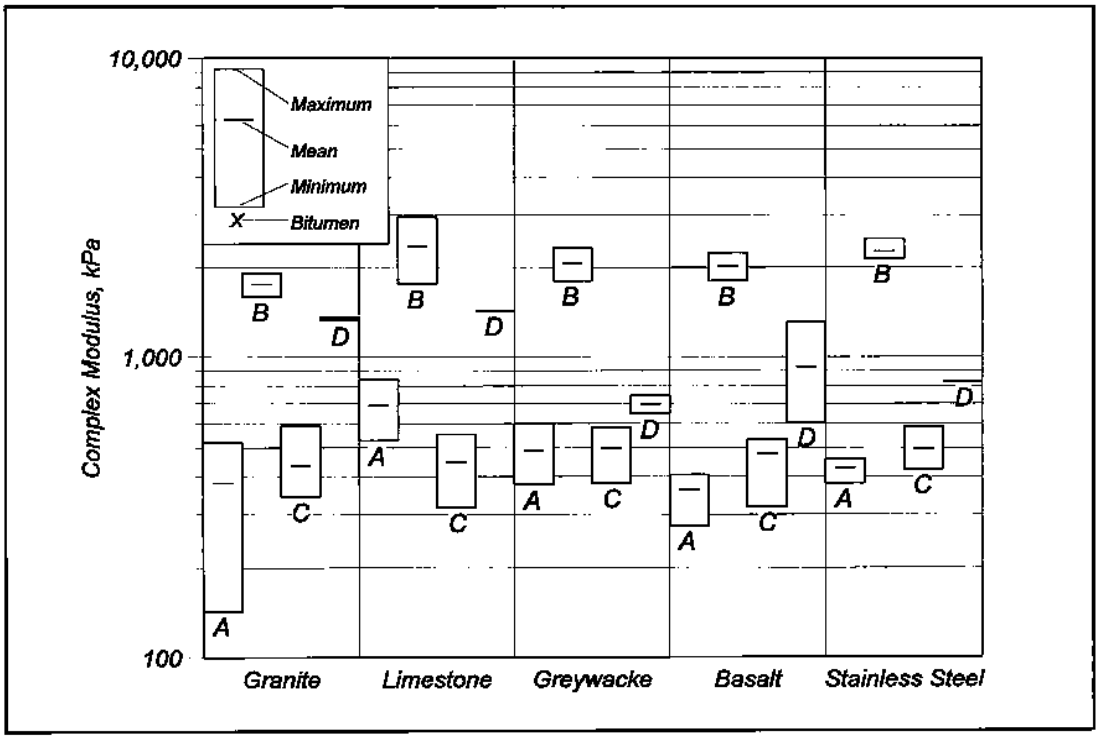


Figure 5.13. Effect of Bitumen-Aggregate Interaction on the Complex Modulus at a Frequency of Oscillation of 10Hz.

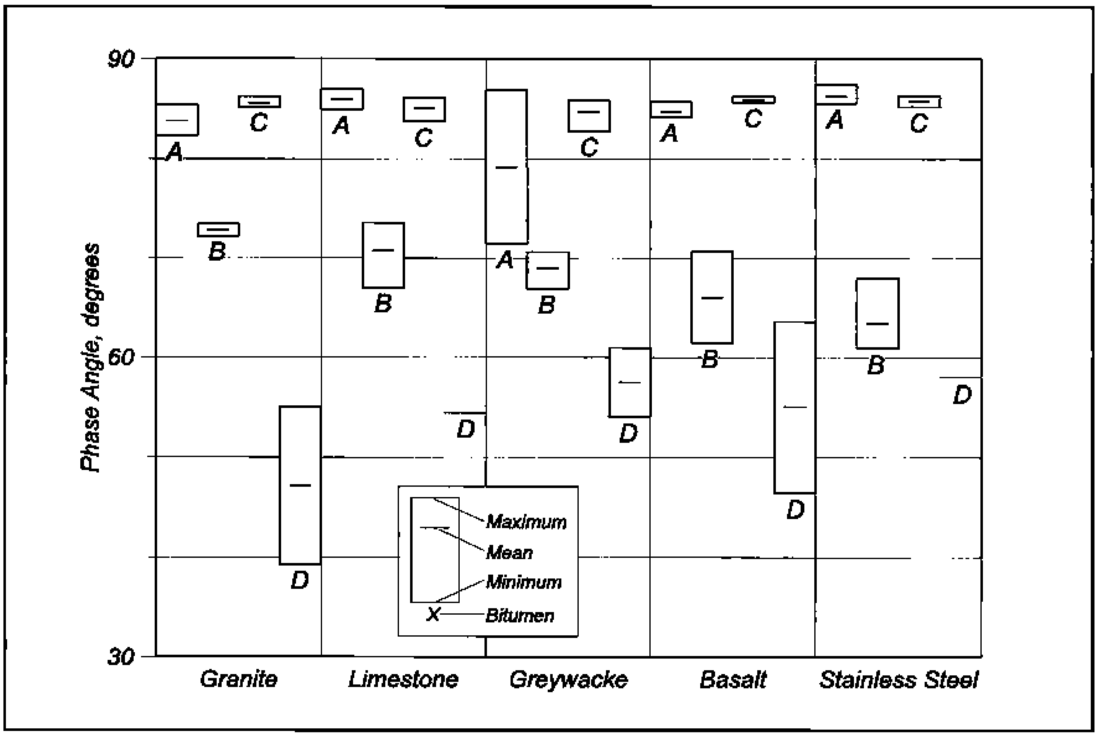


Figure 5.14. Effect of Bitumen-Aggregate Interaction on the Phase Angle at a Frequency of Oscillation of 0.01Hz.

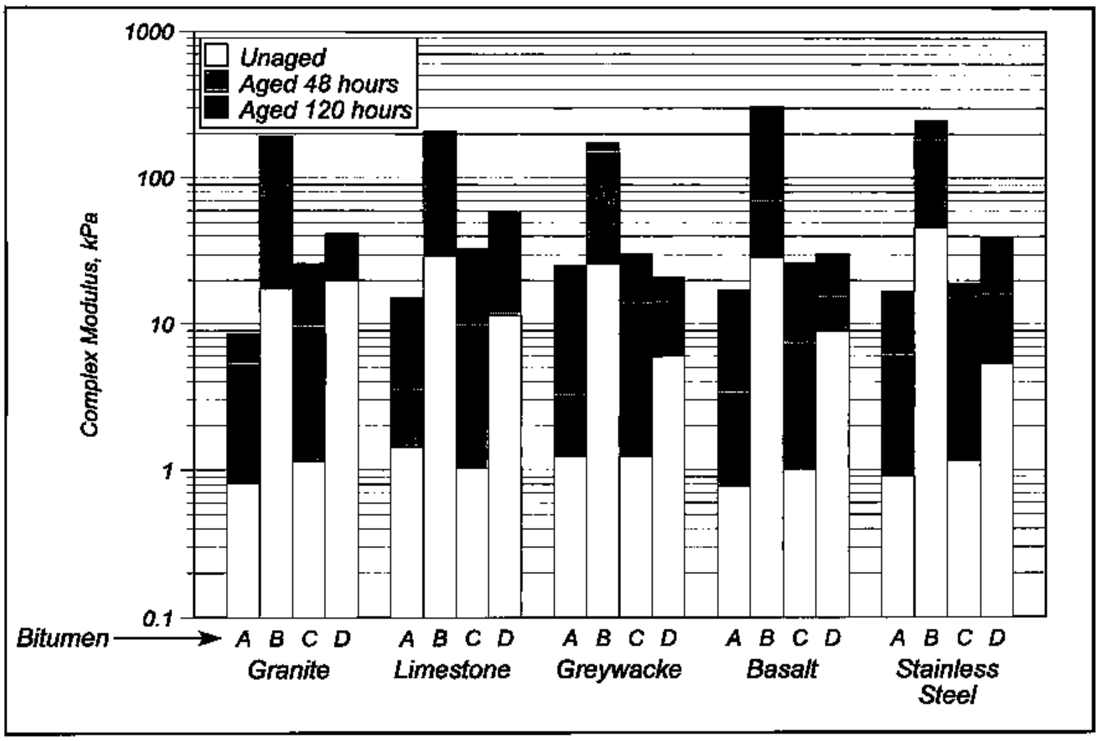


Figure 5.15. Effect of Accelerated Ageing as Indicated by the Complex Modulus at a Frequency of Oscillation of 0.01Hz.

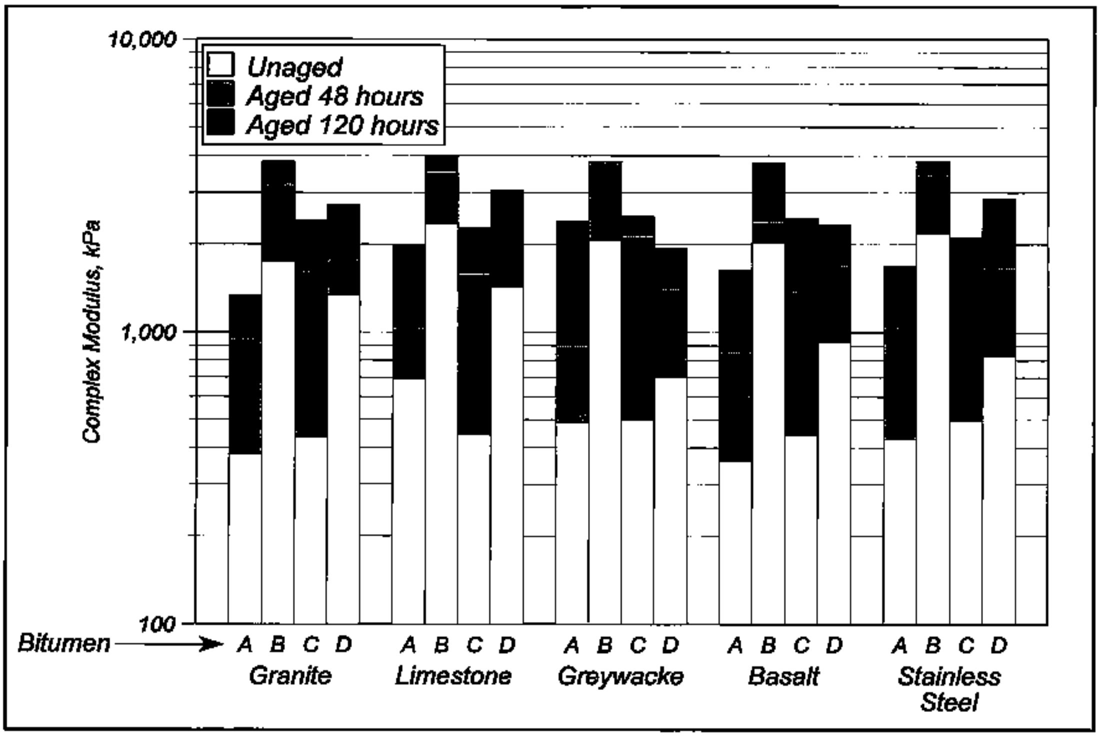


Figure 5.16. Effect of Accelerated Ageing as Indicated by the Complex Modulus at a Frequency of Oscillation of 10Hz.

5.5.2 Analysis of Results

The above general observations of the test results suggest that mineral aggregate may give rise to actual differences in the complex moduli and/or phase angles of at least one of the bitumens investigated. Further analysis of the data, therefore, appears to be justified to determine if these observations are based on statistical evidence.

The data were analysed to determine if differences existed amongst the mean response variables (complex modulus or phase angle) for a particular bitumen coated on the various mineral aggregates. That is, the data were analysed to determine if there existed statistical evidence to support the claim that mineral aggregates influence the physical properties of bitumens. These analyses were carried out on the data obtained from tests on the unaged bitumens as well as from tests after the bitumens had undergone 48 and 120 hours of accelerated ageing. Thus, the analyses also investigated the effect of accelerated ageing on the response variables.

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

One concludes that the population (true or actual) means μ_i and μ_j are not equal if the absolute value of the difference between sample means $\hat{\mu}_i$ and $\hat{\mu}_j$ is greater than the least significant difference (i.e., conclude that $\mu_i \neq \mu_j$ if $|\hat{\mu}_i - \hat{\mu}_j| > LSD_\alpha$). This procedure guarantees that the chance of incorrectly concluding that a difference exists between means when there actually is none is no more than approximately $\alpha \times 100\%$.

Analysis of variance tests (ANOVA or *F* tests) were carried out on the data to determine if, for a particular bitumen type, at least two of the treatment means were different at the 5% significance level, where treatment refers to mineral aggregate type. The results of these tests are shown in Table 5.6 which lists the *F* statistic according to bitumen type. It includes the results of phase angle and complex shear modulus measurements on the unaged bitumens as well as after they were subjected to 48 and 120 hours of accelerated ageing. Note that the boxed values indicate significance meaning that at least two of the treatment means are statistically different. The results for Bitumen D do not include means for Aggregates C and F as only one test for each of these combinations was successful. Consequently, an estimate of the variance for these tests, which is required for ANOVA, was not possible.

It is obvious from these results that there exists a considerable lack of evidence to support the claim that mineral aggregate type affected the phase angles or complex moduli of the unaged bitumens. There are exceptions, however, in that there exists strong evidence that there were differences amongst phase angle means for Bitumens A and B under certain circumstances; namely, at the frequencies of 5 and 10Hz for Bitumen A and at frequencies from 0.05 to 0.5Hz, inclusive, for Bitumen B. There also is the lone result that indicates at least two complex modulus means were different for Bitumen A at a frequency of 0.01Hz. Although it can be said with 95% confidence that these results indicate differences amongst means, they appear to be "special cases" rather than the norm.

The results for tests carried out after 48 hours of accelerated ageing indicate that, for Bitumen C, there exists strong evidence that at least two of the treatment means were

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.

statistically different for complex modulus values over the entire frequency range. Although only four of the ten phase angle results are significant for this bitumen, they do lend credence to the modulus results. The results for the other bitumens indicate a lack of evidence to support the claim that mineral aggregate type affected the phase angles or complex moduli of the bitumens. Two exceptions are noted—namely, the complex modulus results for Bitumen B at frequencies of 5 and 10Hz and the phase angle results for Bitumen D at the 2Hz frequency—but, as with the results for the unaged bitumens, these appear to be special cases rather than the norm. The results for Bitumen C, however, do not appear to be special cases as the they are significant for the entire frequency range.

The results for tests carried out after 120 hours of accelerated ageing indicate that, for Bitumen A, there exists strong evidence that at least two of the treatment means were statistically different for either response variable. That is, there is strong evidence that mineral aggregate type affected the phase angle and complex modulus values of Bitumen A. Conversely, there is a complete lack of evidence to support the claim that mineral aggregate type affected the phase angles or complex moduli of the remaining three bitumens. This is a somewhat surprising result for Bitumen C as the results of the tests after 48 hours of accelerated ageing indicated significance.

The analysis of variance results shown in Table 5.6 indicate that multiple comparison tests could be justifiably performed on the data from tests carried out on Bitumen A after 120 hours of accelerated ageing and on the data from tests carried out on Bitumen C after 48 hours of accelerated ageing. Strictly speaking, multiple comparison tests could also be performed on the other data sets where there exists significance but, as previously mentioned, these appear to be isolated cases. Consequently, inferences based on these data would probably be regarded with scepticism and will, therefore, not be considered further. For these reasons the phase angle data for Bitumen C after 48 hours of accelerated ageing will not be considered either.

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 5.7. Comparison of Differences Amongst Mean Phase Angles 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
0.02	3.05	3.42	3.05	3.05	3.42	3.05	3.05	3.42	3.42	3.05
0.05	2.94	3.29	2.94	2.94	3.29	2.94	2.94	3.29	3.29	2.94
0.1	2.98	3.34	2.98	2.98	3.34	2.98	2.98	3.34	3.34	2.98
0.2	3.52	3.94	3.52	3.52	3.94	3.52	3.52	3.94	3.94	3.52
0.5	3.43	3.84	3.43	3.43	3.84	3.43	3.43	3.84	3.84	3.43
1	4.42	4.95	4.42	4.42	4.95	4.42	4.42	4.95	4.95	4.42
2	3.65	4.08	3.65	3.65	4.08	3.65	3.65	4.08	4.08	3.65
5	3.47	3.87	3.47	3.47	3.87	3.47	3.47	3.87	3.87	3.47
10	3.14	3.51	3.14	3.14	3.51	3.14	3.14	3.51	3.51	3.14

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
0.02	1.80	$\boxed{5.73}$	$\boxed{5.24}$	$\boxed{4.82}$	$\boxed{3.93}$	$\boxed{3.44}$	3.02	0.48	0.91	0.43
0.05	$\boxed{3.87}$	$\boxed{6.91}$	$\boxed{4.65}$	$\boxed{5.84}$	3.03	0.78	1.97	2.25	1.06	1.19
0.1	2.25	$\boxed{5.26}$	$\boxed{7.21}$	$\boxed{5.95}$	3.01	$\boxed{4.96}$	$\boxed{3.70}$	1.95	0.69	1.26
0.2	1.59	$\boxed{6.10}$	$\boxed{5.95}$	$\boxed{5.95}$	4.52	$\boxed{4.36}$	$\boxed{4.36}$	0.15	0.15	0.00
0.5	3.33	$\boxed{6.21}$	$\boxed{6.86}$	$\boxed{6.56}$	2.87	$\boxed{3.53}$	3.22	0.66	0.35	0.31
1	$\boxed{4.77}$	$\boxed{7.06}$	$\boxed{6.68}$	$\boxed{6.78}$	2.29	1.91	2.01	0.38	0.28	0.10
2	3.09	$\boxed{7.50}$	$\boxed{6.22}$	$\boxed{6.70}$	$\boxed{4.40}$	3.13	3.60	1.28	0.80	0.48
5	$\boxed{4.08}$	$\boxed{8.29}$	$\boxed{6.77}$	$\boxed{6.68}$	$\boxed{4.21}$	2.69	2.59	1.52	1.62	0.10
10	$\boxed{4.51}$	$\boxed{8.72}$	$\boxed{6.47}$	$\boxed{6.45}$	$\boxed{4.21}$	1.96	1.94	2.25	2.27	0.02

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

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.

Table 5.9. Comparison of Differences Amongst Mean Complex Moduli of Bitumen C 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	4.91	4.91	4.91	4.91	4.91	4.91	4.91	4.91	4.91	4.91
0.02	6.78	6.78	6.78	6.78	6.78	6.78	6.78	6.78	6.78	6.78
0.05	12.12	12.12	12.12	12.12	12.12	12.12	12.12	12.12	12.12	12.12
0.1	19.24	19.24	19.24	19.24	19.24	19.24	19.24	19.24	19.24	19.24
0.2	30.73	30.73	30.73	30.73	30.73	30.73	30.73	30.73	30.73	30.73
0.5	62.29	62.29	62.29	62.29	62.29	62.29	62.29	62.29	62.29	62.29
1	101.9	101.9	101.9	101.9	101.9	101.9	101.9	101.9	101.9	101.9
2	162.3	162.3	162.3	162.3	162.3	162.3	162.3	162.3	162.3	162.3
5	273.5	273.5	273.5	273.5	273.5	273.5	273.5	273.5	273.5	273.5
10	386.2	386.2	386.2	386.2	386.2	386.2	386.2	386.2	386.2	386.2

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	0.25	4.44	2.27	$\boxed{5.84}$	4.19	2.52	$\boxed{5.59}$	$\boxed{6.71}$	1.40	$\boxed{8.11}$
0.02	0.13	$\boxed{6.93}$	4.17	$\boxed{8.53}$	$\boxed{7.07}$	4.03	$\boxed{8.67}$	$\boxed{11.10}$	1.60	$\boxed{12.70}$
0.05	1.10	$\boxed{13.10}$	8.87	$\boxed{13.27}$	$\boxed{14.20}$	7.77	$\boxed{14.37}$	$\boxed{21.97}$	0.17	$\boxed{22.13}$
0.1	0.77	$\boxed{22.53}$	13.00	$\boxed{23.67}$	$\boxed{23.30}$	12.23	$\boxed{24.43}$	$\boxed{35.53}$	1.13	$\boxed{36.67}$
0.2	1.50	$\boxed{38.43}$	20.73	$\boxed{40.43}$	$\boxed{39.93}$	19.23	$\boxed{41.93}$	$\boxed{59.17}$	2.00	$\boxed{61.17}$
0.5	2.67	$\boxed{78.67}$	41.67	$\boxed{76.33}$	$\boxed{81.33}$	39.00	$\boxed{79.00}$	$\boxed{120.3}$	2.33	$\boxed{118.0}$
1	6.33	$\boxed{128.7}$	66.33	$\boxed{123.7}$	$\boxed{135.0}$	60.00	$\boxed{130.0}$	$\boxed{195.0}$	5.00	$\boxed{190.0}$
2	3.33	$\boxed{211.7}$	101.0	$\boxed{201.7}$	$\boxed{215.0}$	97.67	$\boxed{205.0}$	$\boxed{312.7}$	10.00	$\boxed{302.7}$
5	6.67	$\boxed{370.0}$	165.7	$\boxed{346.7}$	$\boxed{376.7}$	159.0	$\boxed{353.3}$	$\boxed{535.7}$	23.33	$\boxed{512.3}$
10	20.00	$\boxed{530.0}$	233.3	$\boxed{493.3}$	$\boxed{550.0}$	213.0	$\boxed{513.3}$	$\boxed{763.3}$	36.67	$\boxed{726.7}$

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 $\div$ 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 5.10. Summary of Results for the 20mm DBM Mixture with Granite 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
G6	5.7	5.86	628	526	48	939	890
G7	5.3		586			914	
G9	6.3		466			914	
G10	6.2		476			872	
G12	5.8		472			813	
G1	6.1	5.87	498	516	120	1208	1282
G2	6.1		622			1351	
G3	6.5		513			1226	
G4	5.5		494			1132	
G5	5.3		480			1237	
G8	5.3		543			1241	
G11	5.7		546			1218	
G13	6.0		457			1414	
G14	6.3		559			1568	
G15	5.9		451			1226	

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	

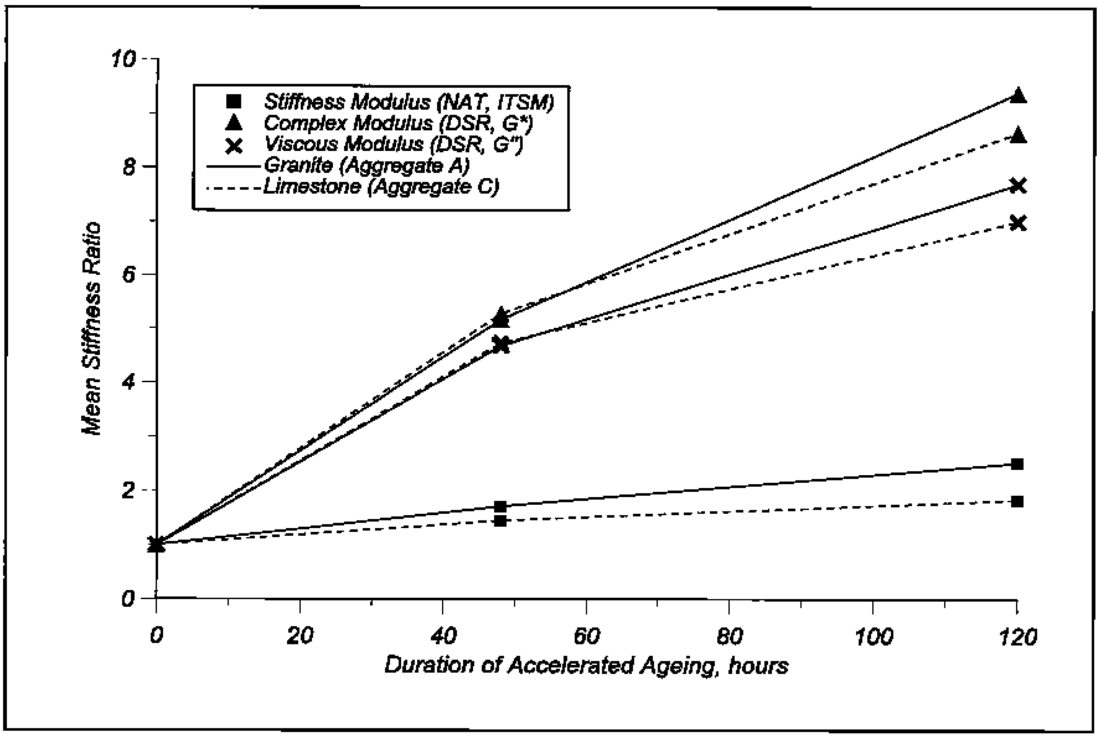


Figure 5.17. Comparison of Stiffness Ratios of Mixtures Tested in the Nottingham Asphalt Tester and the Dynamic Shear Rheometer.

5.6 DISCUSSION

The results of experiments conducted in a dynamic shear rheometer utilising a novel experimental arrangement indicated that complex modulus tests could be reliably carried out on very thin films of bitumens coated on mineral aggregate surfaces. The experiments also indicated that the arrangement could be used to investigate the influence of mineral aggregate on the rheological properties of bitumens as well as changes to these properties due to accelerated ageing.

A test programme involving dynamic shear rheometer tests on four bitumens, of which three were straight-run and one was polymer-modified, coated on four different mineral aggregates and on stainless steel showed that aggregate type or, more likely, bitumen-aggregate interaction, can affect the rheological properties of bitumens. Although the results of tests carried out on the unaged bitumens did not strongly support this claim, the influence of the aggregate became more apparent when tests were carried out on the aged bitumens. Based on sound statistical analyses, these 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.

Table 5.12. Summary of Fisher's LSD Comparisons for Bitumens A and C.

Bitumen	Response Variable	Aggregate Pairs Giving Rise to Statistical Difference in Response Variable
A	Phase Angle (after 120 hours of accelerated ageing)	Granite & Limestone† Granite & Greywacke Granite & Basalt Granite & Stainless Steel Limestone & Greywacke† Limestone & Basalt† Limestone & Stainless Steel
A	Complex Modulus (after 120 hours of accelerated ageing)	Granite & Limestone Granite & Greywacke Granite & Basalt† Granite & Stainless Steel† Limestone & Greywacke† Greywacke & Basalt Greywacke & Stainless Steel
C	Complex Modulus (after 48 hours of accelerated ageing)	Granite & Greywacke Granite & Stainless Steel Limestone & Greywacke Limestone & Stainless Steel Greywacke & Basalt Basalt & Stainless Steel

†Not statistically different at all frequencies.

The two low viscosity straight-run bitumens (i.e., Bitumens A and C) are probably affected by the aggregate surface more than the high viscosity straight-run bitumen (Bitumen B) by virtue of having a greater proportion of unoxidised molecules available after initial coating of the aggregate. In other words, the polar molecules in all three bitumens compete for and bond with the available active sites on the aggregate surface. Once the available sites have been depleted, the proportion of "active" molecules (those that can be oxidised) remaining will be greater in Bitumens A and C relative to Bitumen B. It is likely that subsequent ageing (via oxidation) will be influenced more by the type and amount of "active" molecules present in the bitumens rather than the type of aggregate with which the bitumen comes into contact. Thus, it would appear that the bonds formed when bitumen initially contacts a surface (e.g., aggregate) significantly influence subsequent ageing.

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 relationship between binder performance as measured by DSR tests and performance-related tests on mixtures since it is the mixtures which represent the end-product used in the pavement. In particular, testing bitumen-filler mixtures in the DSR before and after accelerated ageing deserves consideration. Also, based on the limited but apparent correlation between DSR and indirect tensile stiffness modulus tests, a link between DSR tests on bitumens and dynamic modulus tests on bituminous mixtures should be explored.

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6 Discussion, Conclusions and Recommendations

6.1 DISCUSSION

It should be evident from the work presented in this thesis that the effects of age hardening and moisture damage can significantly affect the performance characteristics of bituminous paving mixtures. Pavements built according to present UK specifications are not likely to suffer much from the effects of ageing and water damage because the specifications call for a minimum binder content. However, the present specifications, which are based on experience rather than fundamental engineering properties, are to be replaced in the near future with end-product, performance-based specifications in accordance with European harmonisation. When this occurs there will exist a need for appropriate tools (i.e., test methods) that can be used to evaluate the performance of the product.

Chapters 3 and 4 described the efforts undertaken to develop and evaluate test methods and practices to assess the durability characteristics of bituminous mixtures. These methods, described in full in Appendix A, exploited the strengths of methods developed in the United States for the Strategic Highway Research Program and earlier, but were adapted for use in the United Kingdom.

The work presented in Chapter 3 emphasised the need for a standard procedure for bituminous mixtures prepared in the laboratory (e.g., for mixture design and/or evaluation purposes) and presented data indicating that variations in procedure can result in significant variations amongst specimens with regard to mixture stiffness. Based on this evidence, a sample preparation procedure was proposed for use in the UK (see Appendix A). Validation of the procedure using plant-mixed materials indicated that oven curing (to simulate short-term ageing) of loose dense-graded mixtures subsequent to mixing in the laboratory was necessary to match the stiffness of plant-mixed materials but oven curing of loose gap-graded mixtures was not. It is 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 producing a reasonable amount of moisture damage in mixtures susceptible to such damage as any procedural variation attempted. Some variations were more effective at inducing damage but were also believed to be so severe as to be obviously unrepresentative. Efforts to shorten the duration of the procedure indicated that the cyclic thermal conditioning phase (which is, admittedly, time-consuming and difficult to complete in a normal working day when several specimens are being tested) could be replaced by a static soak period of 72 hours without sacrificing effectiveness of the procedure. However, this is based on a limited number of tests and should, therefore, be applied cautiously.

Evaluations based on mixtures having differences in volumetric proportions of binder and air voids indicated the procedure, as originally proposed, was effective in producing moisture damage to mixtures with a low binder content and/or high void content but had little effect on mixtures with binder and void contents meeting specification criteria. That is, the proposed method for assessing susceptibility to moisture-induced damage appears to be sufficiently sensitive to differences in the components of a mixture that most influence its resistance or susceptibility to moisture damage and can, therefore, be used with confidence for comparative purposes. Thus, the procedure could be successfully used to assess the relative performance of newly constructed bituminous paving mixtures with regard to their sensitivity to water damage.

The work presented in Chapter 5 looked at the durability characteristics of bitumen-aggregate mixtures in a more fundamental way. In this work a dynamic shear rheometer (DSR) was used in a novel way to investigate the effects of accelerated ageing on the rheological properties of bitumens in contact with mineral aggregate. Based on sound statistical analyses, it was shown that mineral aggregate affected the rheological properties of two low viscosity (200pen) straight-run bitumens but had little effect on a high viscosity (50pen) straight-run bitumen and no discernable effect on a bitumen modified with the styrene-butadiene-styrene (SBS) polymer. One of the "aggregates" was stainless steel on which bitumens are normally coated for tests conducted in DSRs.

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

(referred to as stripping). The two phenomena appear to be intimately related as one influences the other and vice versa and both appear to be more influenced by the surface chemistry of the aggregate rather than the bitumen type, although bitumen type is also important.

4) Ageing of the binder in a bituminous mixture is manifested in an increase in its stiffness and is due to changes which give rise to an increase in total associating polarity of bitumen, the strength of the polar associations and the dispersing capacity of the non-associating solvent moiety.

5) The performance of bituminous mixtures is influenced by both the bitumen and the aggregate or, more likely, the interaction between bitumen and aggregate. Therefore, tests to measure or assess the performance of bituminous mixtures ought to be carried out on mixtures, not on neat bitumens. Although tests on neat bitumens have been correlated with the performance of the mixture incorporating the bitumen, the correlations have usually been limited to rather restrictive conditions.

6) Tests developed to predict moisture damage to bituminous mixtures have largely been empirical and, as a consequence, generally fall short of accurately predicting field performance. The Lottman procedure (and its variations) and the Environmental Conditioning System (ECS) method, however, use fundamental tests (i.e., strength and/or stiffness) to assess the effects of accelerated moisture damage and have shown a reasonable ability to do so.

7) The tests developed to predict ageing susceptibility of bituminous paving mixtures have, for the most part, involved testing neat bitumens that had undergone accelerated ageing in a thin film oven to simulate short-term ageing but may also include extended exposure times or pressure oxidative ageing to simulate long-term ageing. While it has been shown that thin film oven ageing techniques can adequately predict the amount of binder hardening that occurs during the mixing process, it is generally agreed that they fall short of accurately predicting the long-term performance of the binder when it is incorporated into a mixture. Pressure oxidative ageing

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

confirmed that the material on which bitumen is coated can influence its physical properties. Strong evidence was provided to show that mineral aggregate type affected the ageing characteristics of the two low viscosity straight-run bitumens but had little or no effect on the high viscosity straight-run bitumen nor the polymer-modified bitumen. These results support the notion that tests should be carried out on mixtures and not just neat bitumens if mixture performance is being assessed.

5) Comparison of indirect tensile stiffness modulus ratios (Nottingham Asphalt Tester) with complex modulus ratios (dynamic shear rheometer) for a limited number of mixtures comprised of the same materials and aged in the same way indicated that both tests ranked the relative ageing susceptibility of the bitumen the same. Although only a limited number of mixtures were evaluated, the results lend credence to the notion that the indirect tensile stiffness modulus test conducted in the Nottingham Asphalt Tester is an effective tool for assessing the relative performance of mixtures with regard to long-term ageing, assuming the tests in the dynamic shear rheometer are indicative of fundamental characteristics of bitumen-aggregate mixtures. At the very least, the results lend credence to the notion that mixtures should be tested if mixture performance is being assessed.

6.3 RECOMMENDATIONS

Based on the evidence presented in this thesis, the following recommendations are made:

1) Bituminous mixture specimens, particularly those used for design and/or performance evaluation, should be fabricated in the laboratory such that they are representative of plant-mixed materials. The sample preparation procedure contained in Appendix A is recommended for this purpose for use in the UK.
2) Tests to assess the performance of a bituminous mixture ought to be carried out on the mixture and evaluations should be based on fundamental engineering properties (e.g., stiffness and/or strength).

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

realised that such an arrangement is practical only for research purposes. The results obtained from the experimental arrangement do, however, emphasise the importance of testing bitumens in contact with mineral aggregates and suggest further investigations on a wider range of materials. These should embrace a wider range of paving-grade bitumens obtained from crude oils with greater compositional diversity than those evaluated in this thesis. A wider range of mineral aggregates commonly used in bituminous paving mixtures should also be investigated with emphasis, in particular, on those having a wider range of porosities and greater mineralogical diversity than those evaluated in this thesis.

7) Investigations of the chemical changes occurring in bitumens as a result of ageing and how these are influenced by mineral aggregates as well as other materials such as stainless steel and/or anodised aluminium also appear warranted. These should embrace a wide range of paving-grade bitumens and a wide range of mineral aggregates with varying porosities and mineralogical compositions. These could accompany the tests suggested in the previous recommendation to provide a link between changes in chemical properties and changes in physical properties.
8) Rheology tests carried out on bitumens in a dynamic shear rheometer need to be linked to the performance of mixtures incorporating the bitumens as a binder. The rheology tests should be carried out on bitumens coated on various substrate materials (i.e., mineral aggregates, stainless steel, etc.). The tests carried out to assess mixture performance (e.g., stiffness modulus, permanent deformation, etc.) should be conducted on mixtures comprised of the same bitumens and aggregates as those used for the rheology tests.
9) Rheology tests (using either a DSR or a bending beam rheometer) carried out on bitumen-filler mixtures should also be investigated. Such tests would provide information about the influence of the filler on the rheological properties of bitumens and possibly the importance of the surface area of the aggregates.

Appendices

A Test Protocols and Practices

B Indirect Tensile Stiffness Modulus

C Rheology Test Data

A Test Protocols and Practices

This appendix contains the three protocols developed for use on the Bitutest project. These are based largely on protocols that were developed in the United States but have been adapted for typical mixtures used in the United Kingdom. The protocols include:

- Standard Practice for Laboratory Preparation of Compacted Bituminous Mixtures
- Standard Practice for Long-Term Oven Ageing of Compacted Bituminous Mixtures and
- Test Method for Measurement of the Water Sensitivity of Compacted Bituminous Mixtures.

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.

3.6 *Metal oven pans* of sufficient size to receive as well as to heat the uncompacted bituminous mixture. The size of the pan should have an area and depth such that the mixture can be spread to a constant depth of 20 to 40 mm.

3.7 *Metal spatula or spoon* of sufficient size to allow rapid and thorough mixing of the uncompacted mixture.

3.8 *Oven gloves* (e.g., household oven gloves).

4 SAMPLING

4.1 Aggregates shall be sampled in accordance with relevant local standards (e.g., BS 812 : Part 102 : 1989).

4.2 Bitumens shall be sampled in accordance with relevant local standards (e.g., BS 3690 : Part 1 : 1989).

5 PREPARATION

5.1 The aggregate shall be graded in accordance with the mixture design or recipe specification for the mixture. The amount of aggregate shall be of sufficient size to obtain a mixture specimen of the desired size.

5.2 Obtain a sufficient quantity of bitumen such that the desired bitumen content can be mixed with the aggregate.

5.3 The desired mixing temperature shall be obtained from the Bitumen Test Data Chart (see Figure A.1) and shall correspond to a viscosity of 2 Poise (0.2 Pa·s) ±2 °C based on the original (unaged) bitumen properties.

5.4 The desired compaction temperature shall also be obtained from the Bitumen Test Data Chart and shall correspond to a viscosity of 3 Poise (0.3 Pa·s) ±2 °C based on the original bitumen properties.

6 PROCEDURE

6.1 Preheat the aggregate for a minimum of 2 hours at the desired mixing temperature.

6.2 Preheat the bitumen to the desired mixing temperature.

NOTE: Bitumens held at the desired mixing temperature for more than two hours should be discarded.

6.3 Preheat the mixer to the desired mixing temperature.

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.

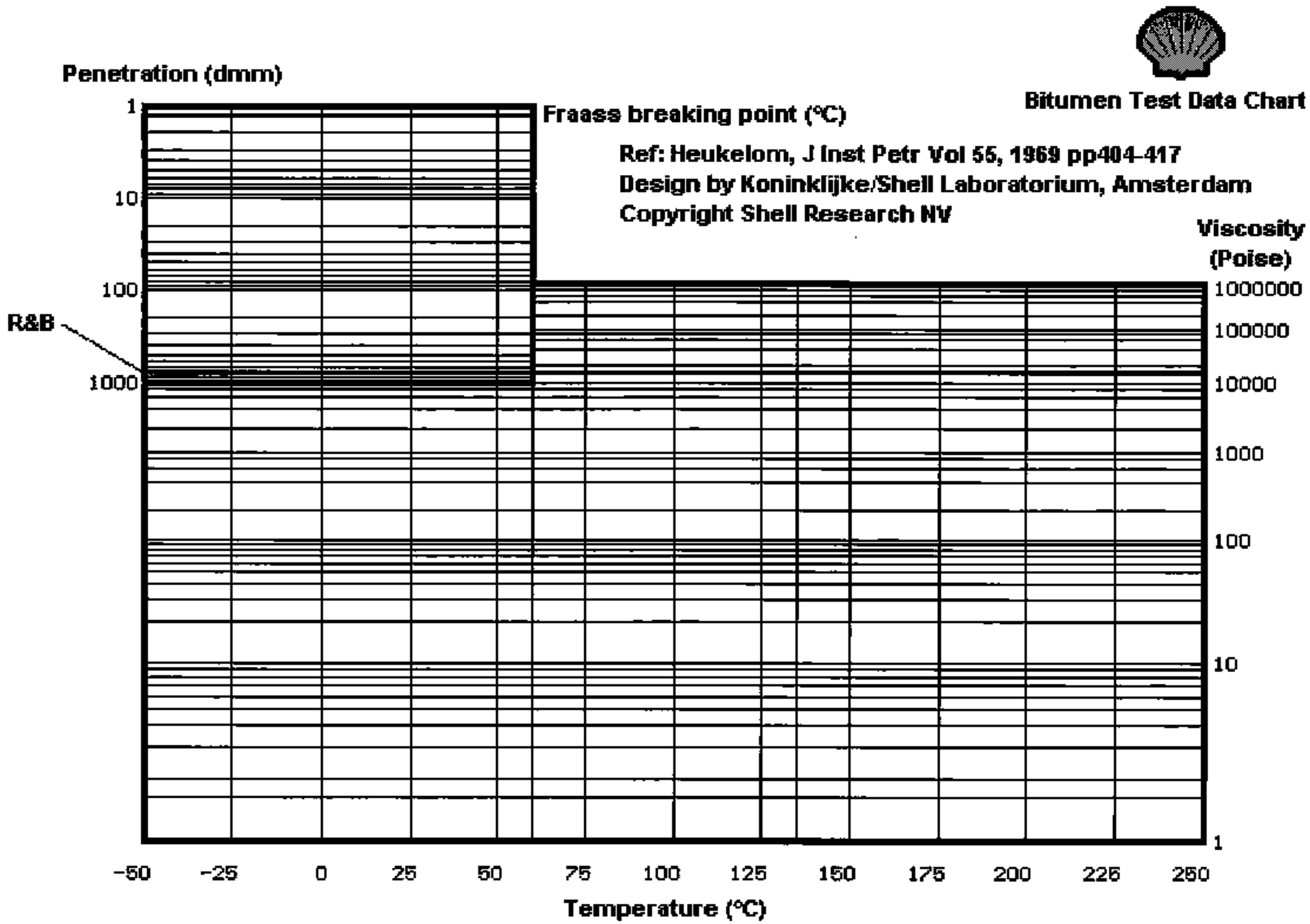

Figure A.1. Bitumen Test Data Chart.

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.

6 PROCEDURE

6.1 Place the specimen in the forced-draft oven at a temperature of 85±1 °C for 120±0.25 hours.

6.2 After 120 hours, turn off the oven, open the door and allow the specimen to cool to room temperature (24 hours should be a sufficient cooling period).

NOTE: Do not touch or remove the specimen until it has cooled to room temperature.

6.3 After the specimen has cooled to room temperature, remove it from the oven. It is now ready for testing as required.

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).

3.5 *Nottingham Asphalt Tester (NAT)* capable of performing the indirect tensile stiffness modulus (ITSM) test in accordance with BS DD213 on compacted bituminous mixture specimens (102mm in diameter by approximately 64mm in height).

4 SAMPLING

Core specimens shall be obtained in accordance with relevant local standards (e.g., BS 598 : Part 100 : 1987).

5 PREPARATION

5.1 Laboratory-moulded specimens shall be prepared 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 Core specimens shall be dried at room temperature (≈20°C) for at least 72 hours prior to testing.

6 PROCEDURE

6.1 Determine the bulk specific gravity of the compacted mixture. Designate the dry mass of the specimen as M_d and the bulk specific gravity as G_{mb}. Determine the maximum specific (Rice) gravity of the loose mixture and designate this as G_{mm}. Calculate the percent air voids (V_v) as follows:

$$V_v = (1 - \frac{G_{mb}}{G_{mm}}) \times 100$$

where:

V_v = percent air voids,
G_{mb} = bulk specific gravity and
G_{mm} = maximum specific gravity

6.2 Determine the unconditioned stiffness modulus in the Nottingham Asphalt Tester. Designate this as $ITSM_U$.

6.3 Place the specimen in the vacuum desiccator, cover it with distilled water at 20°C, seal the apparatus, and apply a partial vacuum of 510±25mm Hg for 30 minutes.

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:

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

where:

ITSM Ratio = stiffness ratio,
$ITSM_{Ci}$ = conditioned stiffness after conditioning cycle i and
$ITSM_U$ = unconditioned stiffness.

6.13 Report the following for each specimen tested:

- Bulk specific gravity (G_{mb})
- Maximum (Rice) specific gravity (G_{mm})
- Percent air voids (V_v)
- Percent saturation (S)
- Unconditioned stiffness ($ITSM_U$)
- Stiffness ratio after each conditioning cycle ($ITSM_{Ci;}$ 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:

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

where:

ν = instantaneous resilient Poisson's ratio
δ_v = instantaneous resilient (elastic) vertical deformation, metres
δ_h = instantaneous resilient (elastic) horizontal deformation, metres
c_1, c_2, c_3 and c_4 = constants dependent on the specimen diameter and loading strip width

and

$$E_R = \frac{P\ (c_5 - \nu \cdot c_6)}{\delta_h \cdot t} \qquad \textbf{B.2}$$

where:

E_R = instantaneous resilient modulus of elasticity, Pascals
P = applied repeated load, Newtons
ν = instantaneous resilient Poisson's ratio
δ_h = instantaneous resilient (elastic) horizontal deformation, metres
t = thickness (height) of specimen, metres
c_5 and c_6 = constants dependent on the specimen diameter and loading strip width

Calculation of the above elastic properties for bituminous mixtures requires measurement of the load and elastic (recoverable) deformations as illustrated in Figure B.1. In theory, determination of the load and deformations appears to be quite simple and straightforward. In practice, however, this may not be the case. The difficulty lies in determination of the inflection point on the unloading portion of the deformation curves which may not be readily discernable, or can be virtually non-existent, depending on the frequency and duration of the load, test temperature, material type, etc. Therefore, it can be reasoned that the indirect tensile test is not a reliable method to use for the determination of the modulus of elasticity of a bituminous mixture. Furthermore, several researchers have shown that the indirect tensile test is not a reliable method for determining Poisson's ratio (8-10).

Due to these problems, the theory has been adapted to allow measurement of the load and total instantaneous horizontal deformation as shown in Figure B.2. The vertical

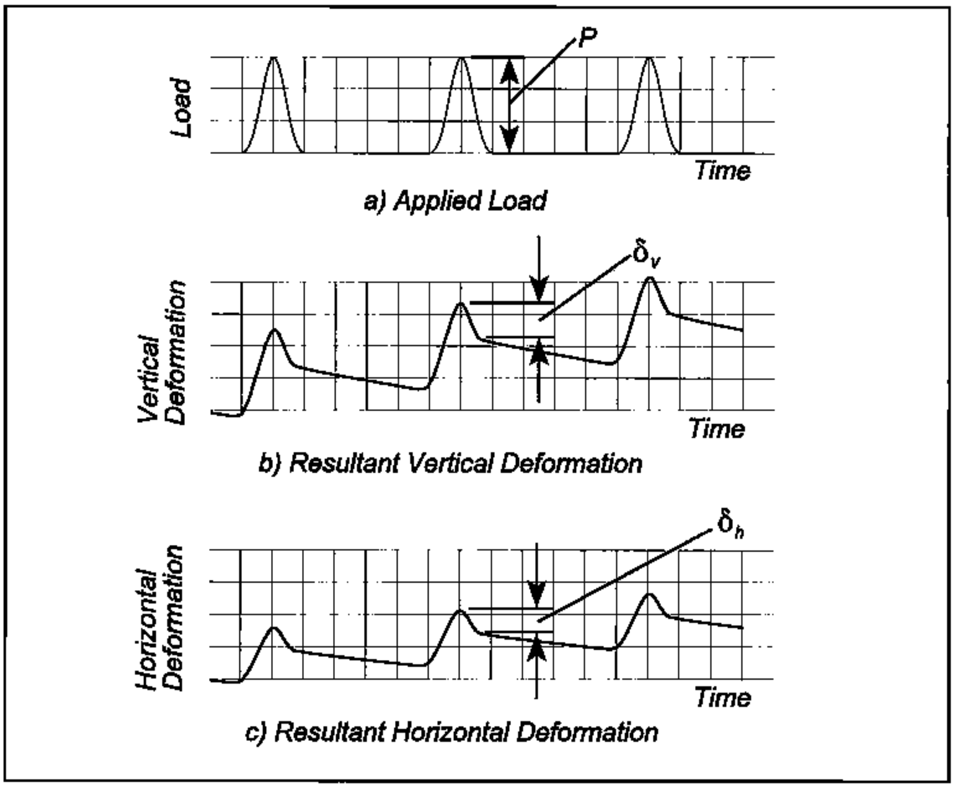

Figure B.1. Typical Load and Deformation Relationships for the Repeated Load Indirect Tensile Test.

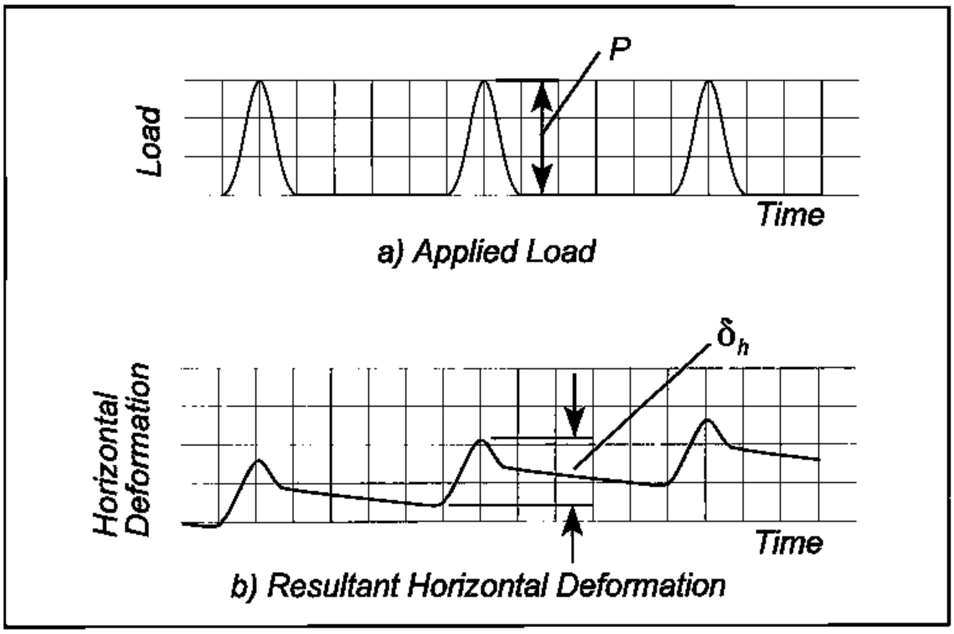

Figure B.2. Typical Load and Deformation Relationships for the Indirect Tensile Stiffness Modulus (ITSM) Test.

deformation is not measured so a value of Poisson's ratio has to be assumed. Because the deformation consists of both the elastic (recoverable) and viscous (time-dependent) components, a resilient modulus of elasticity cannot be calculated. Instead, a stiffness modulus is calculated as follows:

$$S_m = \frac{P\ (c_5 - \nu \cdot c_6)}{\delta_h \cdot t} \qquad \textbf{B.3}$$

where:

S_m = stiffness modulus, resilient modulus or total modulus, Pascals
P = applied repeated load, Newtons
ν = resilient Poisson's ratio
δ_h = resilient total horizontal deformation (as defined in Figure B.2), metres
t = thickness (height) of specimen, metres
c_5 and c_6 = constants dependent on the specimen diameter and loading strip width; e.g., for a 101.6mm (4in) diameter specimen and a 12.7mm (½in) loading strip width, c_5 = 0.2692 and c_6 = -0.9974.

The ITSM software uses Equation B.3 to calculate the stiffness of bituminous mixtures tested in the Nottingham Asphalt Tester.

Factors Affecting Test Results

Numerous factors can significantly affect the stiffness modulus obtained from the ITSM test. Table B.1 lists several of the more important factors and the general effect they have on the experimental result. Of the factors listed, the temperature of the test specimen is, by far, the most important in that small deviations can result in significant variations in the stiffness modulus as illustrated in Figure B.3. The stiffness modulus is less sensitive to loading frequency, particularly in the frequency range commonly associated with typical traffic loading (0.1 to 10Hz) and at low temperatures (11).

Typical variations in stiffness modulus arising from different stress amplitudes applied during ITSM testing are illustrated in Figure B.4. The data from tests on a 30/14 HRA wearing course mixture indicates that moderate to low variations in stiffness modulus result from tests conducted under relatively large differences in

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.

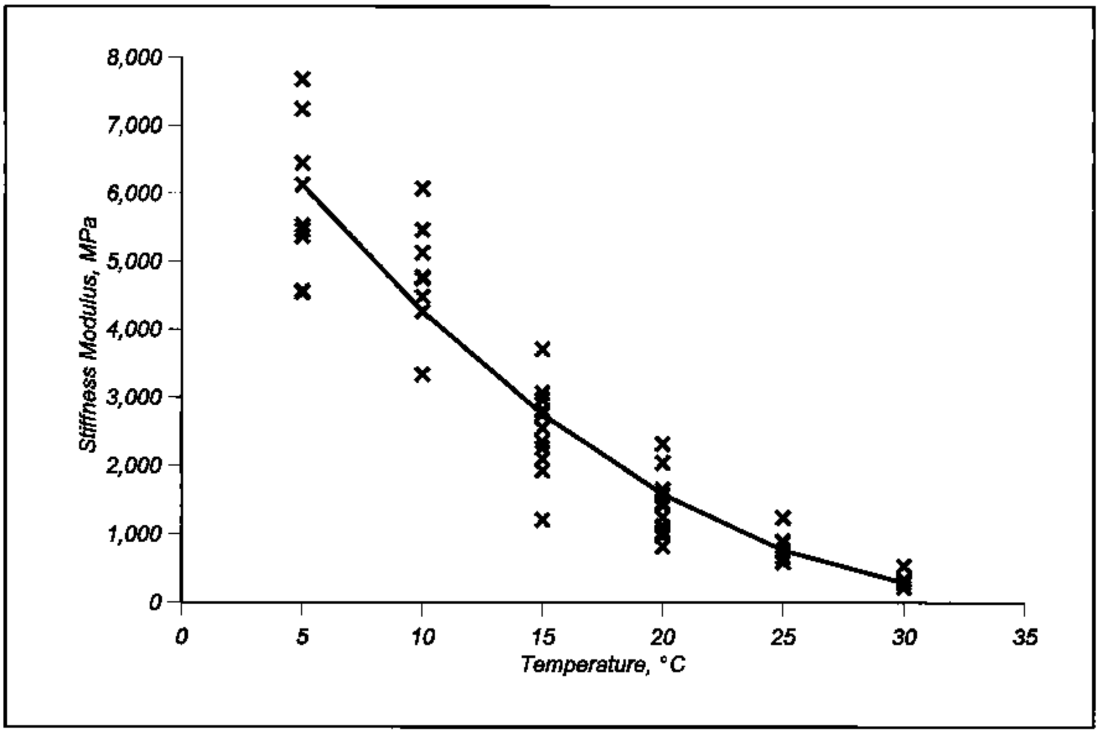

Figure B.3. Typical Results from 20mm DBM Mixtures with 100pen Bitumen and Granite and Gravel Aggregate Showing Effect of Specimen Temperature During ITSM Test.

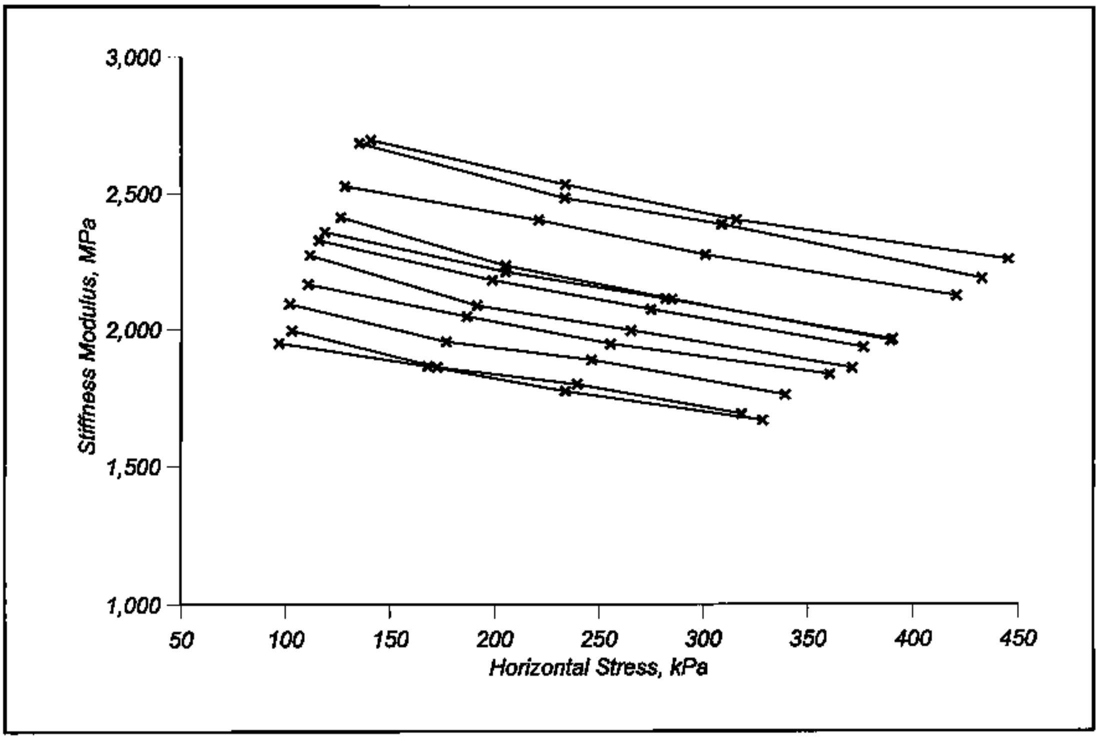

Figure B.4. Typical Results Showing the Effect of Stress Amplitude During the ITSM Test.

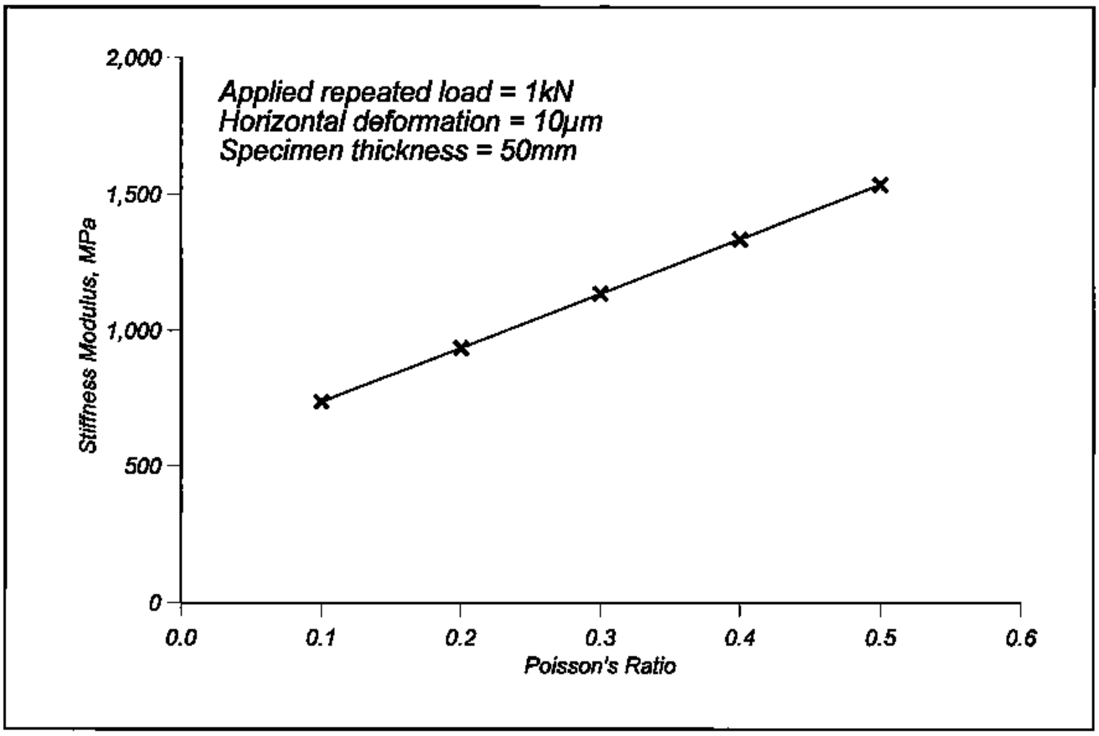

Figure B.5. Hypothetical Stiffness Values Showing Effect of Poisson's Ratio on the ITSM.

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:

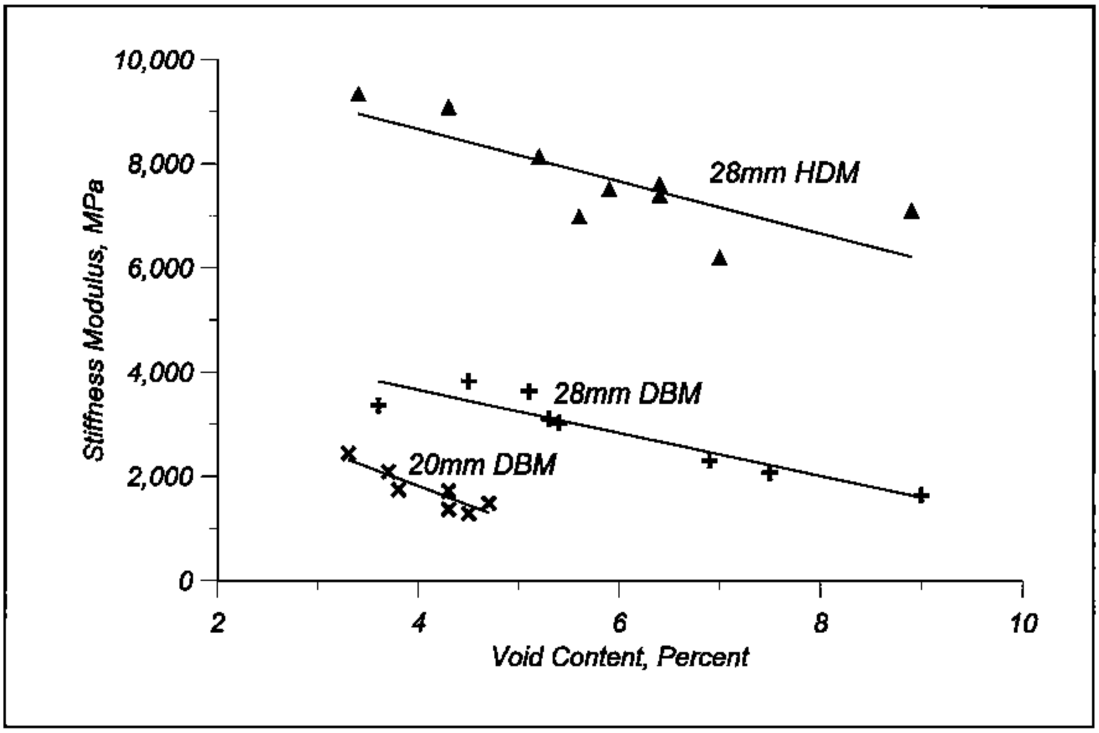

Figure B.6. Typical Results for Three Dense Macadams Showing the Effect of Mixture Air Void Content on the ITSM.

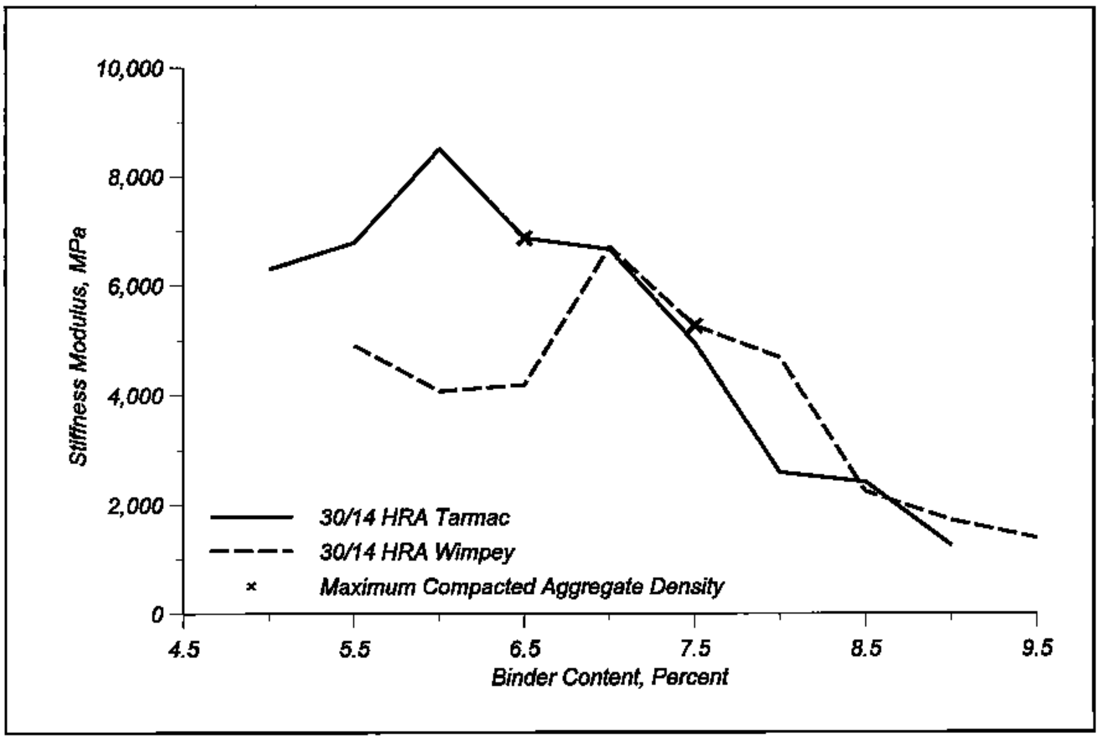

Figure B.7. Typical Variation in Stiffness Modulus Due to Variation in Binder Content for Two Typical HRA Mixtures.

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.

SUMMARY

Application of the theory for the determination of the *instantaneous resilient modulus of elasticity* and the *instantaneous resilient Poisson's ratio* of bituminous mixtures via indirect tension testing presents severe difficulties. Consequently, the theory has been simplified to allow determination of a *stiffness modulus* based on both elastic and viscous deformations.

For practical purposes, determination of the stiffness modulus requires that Poisson's ratio be assumed and although an inaccurate value can lead to an error in the stiffness modulus, the error is relatively small. A value of 0.35 is usually assumed.

The principal factors affecting the stiffness modulus of bituminous mixtures include test conditions such as temperature, loading frequency and stress amplitude together with mixture variables such as binder grade and content, void content and aggregate type and gradation. Test temperature appears to be the most significant variable.

The indirect tensile stiffness modulus (ITSM) test has several advantages over direct tension/compression or flexure tests. The two most significant advantages are that the test is relatively simple and quick and that tests are conducted on cylindrical specimens (moulded or cored). The ability to test cores, which are easily obtained, makes the test particularly attractive to the highway industry in that tests can be easily carried out on in-service materials.

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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:

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

and

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

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

ranged from 1.78×10^{-4}N·m at the 0.01Hz test frequency to 3.85×10^{-3}N·m at the 10Hz test frequency.

```
Main Test Programme                                              BOHLIN CS SYSTEM
Test Series 15, Sample AB008, Aged 120 hours                     Oscillation test
                                                                 1995-05-22  10:59:34
P8DSR gap  0.03 mm
Manual temperature:
Measurement interval    1                      No. of measurement   1
Shear stress 3.83E+04 Pa
Temperature   25.0 -  25.0 C
File name 95220500

Time   Temp   Freq  Phase Viscosity' G*      G'      G"     Strain  Stress Note
 s      C      Hz             Pas    Pa      Pa      Pa                Pa
103.4  25.0   0.010 55.09 2.55E+6 1.96E+5 1.12E+5 1.60E+5 9.06E-3 1.77E+3
257.3  25.0   0.020 52.17 1.82E+6 2.90E+5 1.78E+5 2.29E+5 9.92E-3 2.87E+3
330.2  25.0   0.050 47.41 1.11E+6 4.76E+5 3.22E+5 3.50E+5 1.00E-2 4.76E+3
362.8  25.0    0.10 44.26 7.47E+5 6.72E+5 4.81E+5 4.69E+5 1.02E-2 6.88E+3
380.4  25.0    0.20 41.61 5.10E+5 9.66E+5 7.22E+5 6.41E+5 1.00E-2 9.67E+3
390.0  25.0    0.50 37.00 2.80E+5 1.46E+6 1.17E+6 8.81E+5 9.90E-3 1.45E+4
395.7  25.0     1.0 33.67 1.70E+5 1.92E+6 1.60E+6 1.07E+6 9.93E-3 1.91E+4
400.4  25.0     2.0 30.64 9.86E+4 2.43E+6 2.09E+6 1.24E+6 1.02E-2 2.47E+4
407.0  25.0     5.0 25.97 4.52E+4 3.24E+6 2.92E+6 1.42E+6 1.00E-2 3.25E+4
411.7  25.0    10.0 22.72 2.35E+4 3.82E+6 3.53E+6 1.48E+6 1.00E-2 3.83E+4
```

Figure C.1. Typical Output from the Software Operating the Bohlin Model DSR50 Dynamic Shear Rheometer.

Tables C.1 to C.20 contain a summary of the data obtained from the rheology tests conducted on bitumens coated on mineral aggregates (see Chapter 5). Results obtained from tests on the bitumens before accelerated ageing are labelled *Unaged*, whereas those obtained from tests on the bitumens after accelerated ageing are labelled either *Aged 48 hours* or *Aged 120 hours*, depending on the duration of accelerated ageing. Data is listed for the phase angle (shown as δ) in units of degrees (°), the dynamic viscosity (shown as V) in units of kilopascal-seconds (kPa·s) and the complex shear modulus (shown as G*) in units of kilopascals (kPa).

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

Sample CA001	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	86.97	27.40	1.73	86.86	60.50	3.81	80.81	223.00	14.20
0.02	85.14	26.70	3.37	83.33	62.40	7.89	75.79	197.00	25.50
0.05	83.97	25.70	8.12	77.22	58.10	18.70	68.59	164.00	55.50
0.1	82.23	24.30	15.40	74.70	54.60	35.60	66.73	140.00	95.40
0.2	80.96	23.10	29.40	70.98	48.90	64.90	64.69	113.00	157.00
0.5	79.46	21.10	67.40	70.33	40.20	134.00	62.13	84.90	302.00
1	77.55	18.70	120.00	68.42	34.60	234.00	57.58	64.00	477.00
2	76.06	17.60	228.00	65.40	28.20	390.00	55.49	49.10	749.00
5	71.94	14.80	490.00	61.64	21.10	755.00	49.94	31.30	1290.00
10	68.66	12.50	840.00	57.26	15.70	1170.00	45.62	21.10	1850.00
Sample CA002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	86.14	22.60	1.42	85.45	62.70	3.96	78.18	255.00	16.30
0.02	85.06	22.40	2.83	81.27	60.50	7.69	74.52	229.00	29.80
0.05	82.73	20.80	6.59	75.56	54.00	17.50	66.80	176.00	60.30
0.1	82.64	20.10	12.70	75.51	51.00	33.10	67.83	157.00	106.00
0.2	80.64	19.20	24.40	73.08	43.70	57.30	65.33	129.00	178.00
0.5	78.49	16.90	54.20	68.20	36.60	124.00	60.97	96.30	346.00
1	78.74	16.20	104.00	67.51	31.00	211.00	56.74	72.50	545.00
2	75.66	14.30	186.00	65.54	25.80	356.00	54.12	55.30	858.00
5	72.46	12.20	403.00	60.55	19.20	692.00	49.28	35.00	1450.00
10	69.33	10.40	695.00	56.94	14.50	1080.00	44.86	23.50	2090.00
Sample CA003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	84.92	18.50	1.17	84.21	48.30	3.05	74.19	228.00	14.90
0.02	84.02	17.50	2.22	80.28	49.00	6.25	73.09	214.00	28.10
0.05	83.86	16.10	5.08	78.07	43.20	13.90	69.51	178.00	59.60
0.1	81.77	15.20	9.67	76.63	41.30	26.70	67.33	146.00	99.50
0.2	80.88	14.50	18.50	73.11	35.50	46.70	65.00	125.00	173.00
0.5	78.78	12.80	41.10	69.97	29.90	99.90	59.18	87.70	321.00
1	77.29	12.40	80.10	68.90	25.00	168.00	55.64	67.00	510.00
2	76.24	10.90	141.00	66.32	20.70	284.00	54.50	52.50	810.00
5	73.08	9.30	306.00	62.53	15.80	558.00	49.10	33.80	1400.00
10	70.32	7.92	528.00	58.91	12.00	882.00	44.27	22.00	1980.00

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.4. Summary of Rheology Test Results for Bitumen A on Aggregate E.

Sample EA001	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	85.70	13.90	0.878	85.34	43.30	2.73	77.64	295.00	19.00
0.02	84.70	13.10	1.66	82.53	47.60	6.03	72.16	264.00	34.90
0.05	81.98	13.20	4.19	77.82	43.80	14.10	66.71	204.00	69.90
0.1	79.90	12.30	7.87	75.70	40.00	25.90	61.96	160.00	114.00
0.2	80.39	11.00	14.00	72.41	34.70	45.80	61.59	131.00	188.00
0.5	79.79	9.94	31.70	68.79	29.00	97.70	58.28	95.40	352.00
1	78.50	9.19	58.90	66.06	24.40	168.00	54.69	68.10	524.00
2	75.98	8.42	109.00	63.66	19.90	279.00	51.86	50.60	808.00
5	74.21	7.11	232.00	60.74	14.90	537.00	47.38	31.60	1350.00
10	71.57	6.12	406.00	57.25	11.10	832.00	42.73	20.50	1890.00
Sample EA002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	84.20	13.30	0.839	83.55	67.10	4.25	81.29	289.00	18.30
0.02	83.58	13.00	1.64	80.12	64.70	8.26	71.60	229.00	30.30
0.05	81.95	12.30	3.91	77.29	55.30	17.80	67.34	186.00	63.20
0.1	81.80	11.60	7.35	72.17	47.80	31.50	61.98	147.00	104.00
0.2	79.97	11.10	14.20	72.14	42.20	55.70	58.76	115.00	168.00
0.5	78.10	9.75	31.30	70.07	35.30	118.00	55.99	82.70	313.00
1	77.80	9.15	58.80	67.28	29.00	198.00	54.15	61.80	479.00
2	76.26	8.28	107.00	64.22	23.40	327.00	50.86	44.30	718.00
5	74.18	7.11	232.00	60.30	17.20	623.00	45.76	26.70	1170.00
10	71.59	6.08	402.00	56.16	12.90	978.00	42.67	17.60	1640.00
Sample EA003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	84.38	9.23	0.583	81.74	53.30	3.39	72.56	218.00	14.30
0.02	83.59	8.88	1.12	77.21	51.60	6.64	69.31	185.00	24.80
0.05	81.89	8.48	2.69	76.21	43.90	14.20	68.51	151.00	51.10
0.1	80.94	8.05	5.12	74.36	39.10	25.50	63.07	118.00	82.80
0.2	80.59	7.51	9.57	71.43	32.70	43.40	61.58	100.00	143.00
0.5	78.46	6.78	21.70	68.34	27.30	92.10	57.42	69.50	259.00
1	78.58	6.13	39.30	66.43	22.70	155.00	55.39	52.20	398.00
2	76.12	5.57	72.10	64.31	18.60	259.00	52.01	37.90	605.00
5	73.70	4.80	157.00	60.32	13.60	492.00	47.11	23.30	1000.00
10	72.13	4.17	275.00	57.41	10.40	772.00	43.48	15.30	1390.00

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.6. Summary of Rheology Test Results for Bitumen B on Aggregate A.

Sample AB008	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	72.20	244.00	16.10	63.02	1240.00	87.20	55.09	2550.00	196.00
0.02	70.38	210.00	28.00	56.48	954.00	144.00	52.17	1820.00	290.00
0.05	65.74	163.00	56.00	53.16	642.00	252.00	47.41	1110.00	476.00
0.1	63.60	129.00	90.50	51.35	497.00	400.00	44.26	747.00	672.00
0.2	61.74	104.00	148.00	48.80	350.00	585.00	41.61	510.00	966.00
0.5	58.47	75.20	277.00	45.10	211.00	936.00	37.00	280.00	1460.00
1	56.24	57.70	436.00	41.24	136.00	1300.00	33.67	170.00	1920.00
2	53.14	42.40	666.00	37.91	85.80	1750.00	30.64	98.60	2430.00
5	48.66	26.70	1120.00	32.73	43.00	2500.00	25.97	45.20	3240.00
10	44.39	17.60	1590.00	28.78	23.90	3120.00	22.72	23.50	3820.00
Sample AB009	Unaged			Aged 48 hours			Aged 120 hours		
0.01	73.46	283.00	18.50	61.76	1350.00	96.60	55.38	2500.00	191.00
0.02	69.10	241.00	32.40	55.47	1080.00	164.00	52.31	1750.00	278.00
0.05	65.71	187.00	64.30	52.03	686.00	273.00	47.89	1080.00	458.00
0.1	64.28	157.00	110.00	50.76	525.00	426.00	44.26	728.00	656.00
0.2	61.79	126.00	180.00	48.08	376.00	635.00	41.81	496.00	934.00
0.5	58.98	90.70	333.00	44.06	223.00	1010.00	37.55	277.00	1430.00
1	56.74	69.70	523.00	40.72	145.00	1400.00	34.46	168.00	1860.00
2	53.15	50.80	798.00	36.94	89.70	1880.00	30.92	98.80	2420.00
5	48.52	32.10	1350.00	32.02	45.00	2670.00	26.44	45.70	3220.00
10	44.10	21.20	1910.00	28.47	25.10	3310.00	23.17	24.00	3830.00

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.8. Summary of Rheology Test Results for Bitumen B on Aggregate D.

Sample DB007	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	70.61	382.00	25.40	59.89	1860.00	135.00	54.96	2070.00	159.00
0.02	67.04	320.00	43.60	53.93	1420.00	221.00	51.73	1590.00	254.00
0.05	65.28	249.00	86.20	50.95	923.00	373.00	45.52	948.00	418.00
0.1	62.27	195.00	139.00	49.01	678.00	564.00	46.25	682.00	593.00
0.2	59.38	156.00	228.00	45.96	462.00	808.00	43.25	456.00	835.00
0.5	55.64	108.00	411.00	41.18	267.00	1270.00	38.84	255.00	1280.00
1	53.57	81.00	633.00	38.06	168.00	1710.00	35.96	159.00	1700.00
2	50.01	57.90	950.00	33.13	98.60	2270.00	32.00	93.30	2210.00
5	44.25	34.00	1530.00	28.49	47.10	3100.00	27.79	44.80	3020.00
10	39.74	21.30	2090.00	24.48	24.70	3740.00	24.52	23.90	3620.00
Sample DB008	Unaged			Aged 48 hours			Aged 120 hours		
0.01	69.55	436.00	29.20	61.88	2130.00	152.00	56.00	2040.00	155.00
0.02	66.16	378.00	51.90	53.53	1560.00	244.00	50.59	1560.00	253.00
0.05	63.32	290.00	102.00	50.54	1010.00	410.00	46.22	941.00	409.00
0.1	61.28	237.00	170.00	48.46	721.00	606.00	45.65	704.00	618.00
0.2	59.07	184.00	269.00	45.43	499.00	880.00	42.52	454.00	844.00
0.5	56.03	125.00	474.00	41.16	286.00	1370.00	37.45	253.00	1310.00
1	52.82	91.30	720.00	37.65	179.00	1840.00	35.86	160.00	1710.00
2	48.55	63.50	1060.00	32.83	103.00	2390.00	31.13	91.00	2210.00
5	43.43	37.40	1710.00	27.68	48.50	3280.00	27.08	43.10	2970.00
10	39.22	23.40	2320.00	24.38	25.80	3920.00	24.07	23.10	3560.00
Sample DB009	Unaged			Aged 48 hours			Aged 120 hours		
0.01	66.91	337.00	23.00	57.17	2340.00	175.00	50.58	2610.00	212.00
0.02	66.34	256.00	35.10	52.12	1480.00	236.00	46.93	1980.00	340.00
0.05	64.27	189.00	65.80	50.46	1020.00	414.00	44.81	1180.00	528.00
0.1	63.09	149.00	105.00	46.92	694.00	597.00	43.38	841.00	769.00
0.2	62.00	119.00	169.00	45.78	486.00	852.00	40.71	569.00	1100.00
0.5	58.87	86.60	318.00	40.48	277.00	1340.00	36.52	307.00	1620.00
1	56.24	64.60	488.00	36.27	164.00	1750.00	32.91	183.00	2110.00
2	53.32	47.80	749.00	32.78	100.00	2330.00	29.83	107.00	2710.00
5	48.58	30.20	1270.00	27.85	46.90	3160.00	25.52	48.70	3550.00
10	44.34	20.00	1800.00	24.13	24.70	3800.00	22.45	25.40	4170.00

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.10. Summary of Rheology Test Results for Bitumen B on "Aggregate" F.

Sample FB001	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	61.59	773.00	55.20	59.63	1990.00	145.00	44.09	3890.00	351.00
0.02	60.51	581.00	83.90	54.90	1460.00	224.00	42.75	2570.00	475.00
0.05	55.91	417.00	158.00	49.96	914.00	375.00	36.33	1400.00	740.00
0.1	52.96	295.00	232.00	45.55	621.00	547.00	36.61	945.00	996.00
0.2	52.95	233.00	368.00	44.40	423.00	759.00	32.28	560.00	1320.00
0.5	49.35	147.00	608.00	40.95	253.00	1210.00	29.37	289.00	1850.00
1	47.61	100.00	854.00	38.07	159.00	1620.00	28.90	163.00	2120.00
2	42.12	68.60	1290.00	33.48	91.30	2080.00	24.53	89.60	2710.00
5	37.84	37.10	1900.00	29.19	44.00	2830.00	21.00	38.50	3380.00
10	34.34	22.50	2500.00	26.37	24.60	3480.00	19.04	20.20	3890.00
Sample FB007	Unaged			Aged 48 hours			Aged 120 hours		
0.01	68.02	433.00	29.40	59.02	1470.00	108.00	55.86	2710.00	206.00
0.02	66.83	358.00	49.00	56.70	1160.00	175.00	51.96	2040.00	325.00
0.05	62.12	267.00	95.10	52.51	769.00	305.00	46.60	1250.00	542.00
0.1	61.04	215.00	154.00	51.02	567.00	458.00	45.91	899.00	787.00
0.2	58.75	166.00	244.00	48.16	402.00	679.00	42.84	601.00	1110.00
0.5	54.54	115.00	443.00	43.62	237.00	1080.00	37.83	328.00	1680.00
1	52.16	83.10	661.00	39.95	153.00	1500.00	33.47	196.00	2230.00
2	48.58	58.40	979.00	36.52	95.40	2010.00	30.48	115.00	2850.00
5	44.19	34.80	1570.00	31.23	46.70	2830.00	26.02	52.60	3770.00
10	39.85	21.80	2140.00	27.35	25.70	3510.00	22.56	27.40	4490.00
Sample FB008	Unaged			Aged 48 hours			Aged 120 hours		
0.01	60.95	539.00	38.70	50.86	4090.00	331.00	48.35	2990.00	251.00
0.02	54.84	389.00	59.80	47.18	2930.00	503.00	48.10	2180.00	368.00
0.05	52.46	281.00	111.00	43.89	1730.00	785.00	44.99	1320.00	586.00
0.1	56.75	217.00	163.00	41.74	1210.00	1140.00	43.48	906.00	827.00
0.2	55.68	166.00	252.00	37.42	729.00	1510.00	39.72	585.00	1150.00
0.5	54.55	117.00	451.00	31.70	359.00	2150.00	36.44	329.00	1740.00
1	52.80	86.30	681.00	28.10	200.00	2670.00	32.87	199.00	2310.00
2	48.63	60.40	1010.00	24.50	106.00	3220.00	29.76	115.00	2910.00
5	44.13	36.10	1630.00	20.84	45.20	3990.00	25.60	52.90	3850.00
10	39.77	22.80	2240.00	18.13	22.40	4530.00	22.27	27.30	4520.00

Table C.10. Summary of Rheology Test Results for Bitumen B on "Aggregate" F (Continued).

Sample FB009	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	63.26	860.00	60.50	51.82	2060.00	165.00	57.40	2480.00	185.00
0.02	47.94	475.00	80.30	45.52	1410.00	249.00	52.84	1720.00	271.00
0.05	50.53	327.00	133.00	43.33	818.00	374.00	48.79	1120.00	467.00
0.1	49.78	239.00	197.00	44.14	585.00	528.00	45.29	741.00	655.00
0.2	51.38	179.00	288.00	42.92	399.00	736.00	42.60	499.00	926.00
0.5	50.64	119.00	482.00	40.61	233.00	1120.00	38.20	272.00	1380.00
1	49.31	84.40	699.00	37.44	147.00	1510.00	34.93	173.00	1900.00
2	46.98	59.10	1020.00	34.48	90.60	2010.00	31.64	99.60	2390.00
5	43.04	34.90	1600.00	30.88	45.90	2810.00	26.55	45.60	3210.00
10	39.34	21.90	2170.00	27.71	25.70	3470.00	23.40	24.30	3840.00

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

Sample DC001	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	14.10	0.886	76.71	185.00	12.00	58.82	487.00	35.70
0.02	84.07	13.10	1.66	74.68	171.00	22.20	52.43	335.00	53.10
0.05	81.69	12.10	3.85	71.86	141.00	46.50	59.43	274.00	100.00
0.1	81.35	11.30	7.20	70.55	114.00	75.70	58.71	208.00	153.00
0.2	81.14	10.70	13.70	68.53	95.40	129.00	58.36	165.00	243.00
0.5	78.10	9.62	30.90	65.53	75.00	259.00	57.04	116.00	436.00
1	76.87	8.73	56.30	63.06	60.60	427.00	54.45	86.70	670.00
2	75.09	7.89	103.00	59.99	48.10	699.00	50.93	62.60	1010.00
5	72.99	6.67	219.00	54.65	33.10	1270.00	46.37	38.60	1680.00
10	70.41	5.72	381.00	49.45	23.00	1910.00	41.52	24.60	2330.00
Sample DC002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	82.75	20.80	1.31	72.93	244.00	16.00	70.49	407.00	27.10
0.02	82.53	19.60	2.49	69.89	188.00	25.10	69.47	351.00	47.10
0.05	81.15	18.30	5.81	71.22	160.00	53.20	64.75	272.00	94.60
0.1	80.31	17.20	11.00	69.04	134.00	90.20	63.40	218.00	153.00
0.2	78.63	16.20	20.80	67.56	112.00	153.00	60.43	172.00	249.00
0.5	76.64	14.20	46.00	65.38	88.70	307.00	58.06	129.00	476.00
1	75.35	12.70	82.60	63.05	71.10	501.00	55.05	97.10	745.00
2	74.10	11.20	147.00	59.61	55.60	810.00	50.99	70.30	1140.00
5	71.59	9.44	313.00	54.35	37.80	1460.00	45.79	42.20	1850.00
10	68.41	7.86	531.00	48.91	26.00	2170.00	40.54	26.50	2560.00
Sample DC003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	85.86	23.40	1.48	72.66	227.00	14.90	70.17	420.00	28.10
0.02	83.09	22.40	2.83	72.90	203.00	26.70	68.77	345.00	46.40
0.05	81.33	21.30	6.76	70.76	167.00	55.70	66.02	277.00	95.10
0.1	79.90	19.80	12.60	70.54	143.00	95.20	65.05	230.00	159.00
0.2	78.57	17.80	22.80	67.93	120.00	163.00	61.57	177.00	253.00
0.5	77.88	15.70	50.60	65.37	94.90	328.00	58.78	128.00	470.00
1	75.51	14.20	92.10	62.96	77.60	548.00	55.71	96.70	736.00
2	74.01	12.50	163.00	59.40	60.60	885.00	52.05	71.00	1130.00
5	71.19	10.40	344.00	53.94	40.90	1590.00	46.41	43.00	1870.00
10	68.29	8.63	584.00	48.77	28.20	2360.00	41.35	27.40	2610.00

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.15. Summary of Rheology Test Results for Bitumen C on "Aggregate" F.

Sample FC001	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	85.23	21.00	1.32	77.93	184.00	11.80	72.75	222.00	14.60
0.02	84.47	20.50	2.59	72.50	157.00	20.70	69.98	190.00	25.40
0.05	82.71	20.10	6.36	72.34	131.00	43.30	67.15	150.00	51.00
0.1	80.99	18.40	11.70	71.02	117.00	77.40	65.95	127.00	87.10
0.2	79.31	17.10	21.80	69.02	102.00	137.00	63.77	102.00	143.00
0.5	77.80	15.10	48.60	65.72	77.70	268.00	60.78	75.00	270.00
1	76.84	13.80	89.10	63.33	63.20	444.00	57.97	58.20	431.00
2	74.92	12.30	160.00	60.79	50.00	719.00	55.04	43.70	670.00
5	72.28	10.40	342.00	56.12	34.80	1320.00	50.72	28.50	1160.00
10	69.73	8.77	587.00	50.94	24.40	1980.00	46.28	19.20	1670.00
Sample FC002	Unaged			Aged 48 hours			Aged 120 hours		
0.01	86.36	15.70	0.991	78.29	215.00	13.80	72.53	392.00	25.80
0.02	84.12	15.30	1.93	74.81	205.00	26.60	68.16	311.00	42.10
0.05	81.65	14.00	4.45	70.62	166.00	55.20	65.59	247.00	85.00
0.1	80.42	12.90	8.19	68.72	145.00	98.00	63.67	208.00	146.00
0.2	78.80	12.20	15.60	67.57	121.00	165.00	61.96	164.00	234.00
0.5	77.45	10.80	34.70	65.57	94.50	326.00	57.65	117.00	434.00
1	76.80	9.94	64.20	62.48	75.00	532.00	56.42	89.90	678.00
2	74.71	8.74	114.00	59.64	59.30	863.00	52.57	65.50	1040.00
5	72.58	7.44	245.00	53.87	39.50	1530.00	47.18	40.20	1720.00
10	69.83	6.34	424.00	48.22	26.90	2270.00	42.19	25.80	2420.00
Sample FC003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	85.83	18.10	1.14	55.20	281.00	21.50	71.94	265.00	17.50
0.02	83.81	17.30	2.18	59.70	216.00	31.50	68.21	221.00	29.90
0.05	83.34	16.70	5.30	64.41	165.00	57.40	67.12	178.00	60.70
0.1	79.18	15.30	9.80	66.32	130.00	89.10	64.11	147.00	103.00
0.2	79.09	14.20	18.10	65.56	108.00	149.00	62.88	115.00	163.00
0.5	79.10	12.50	40.00	64.23	83.90	293.00	59.15	84.40	309.00
1	75.16	10.90	70.60	61.68	67.90	485.00	57.82	65.80	488.00
2	75.36	10.00	130.00	59.21	53.40	782.00	54.54	48.60	750.00
5	72.29	8.40	277.00	53.97	36.00	1400.00	49.08	31.00	1290.00
10	69.88	7.13	477.00	48.89	24.90	2080.00	44.84	20.70	1840.00

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

Sample CD003	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	54.45	148.00	11.40	56.89	161.00	12.10	56.15	777.00	58.80
0.02	56.75	124.00	18.60	59.45	134.00	19.60	49.27	492.00	81.50
0.05	59.02	89.10	32.60	59.58	101.00	37.00	54.63	347.00	134.00
0.1	61.60	72.90	52.10	61.26	79.70	57.10	56.30	273.00	206.00
0.2	63.49	61.70	86.70	63.03	66.80	94.30	57.06	212.00	318.00
0.5	63.79	48.40	170.00	62.29	51.10	181.00	57.01	151.00	564.00
1	64.31	40.20	281.00	60.90	41.50	298.00	54.06	112.00	868.00
2	63.61	33.10	465.00	59.73	33.00	479.00	51.48	82.60	1330.00
5	60.86	25.10	902.00	56.35	23.70	896.00	45.37	50.20	2210.00
10	56.76	19.00	1430.00	51.83	17.00	1350.00	39.51	31.00	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.19. Summary of Rheology Test Results for Bitumen D on Aggregate E.

Sample ED002	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	46.44	175.00	15.20	56.58	200.00	15.10	58.18	481.00	35.60
0.02	50.00	128.00	21.00	58.09	167.00	24.70	53.72	301.00	46.90
0.05	53.35	96.40	37.70	59.38	122.00	44.60	58.48	231.00	85.00
0.1	58.83	77.40	56.80	62.70	104.00	73.50	58.20	183.00	135.00
0.2	61.06	63.00	90.50	62.64	83.20	118.00	59.99	147.00	213.00
0.5	61.82	48.40	173.00	62.47	64.30	228.00	59.56	109.00	398.00
1	62.24	39.40	280.00	62.14	52.40	372.00	57.67	84.00	624.00
2	61.00	32.20	463.00	60.20	41.60	603.00	55.11	64.40	986.00
5	58.01	23.00	851.00	55.79	29.60	1120.00	49.61	41.20	1700.00
10	54.93	17.10	1310.00	50.98	21.10	1700.00	44.04	26.90	2440.00
Sample ED003	Unaged			Aged 48 hours			Aged 120 hours		
0.01	55.13	87.80	6.73	59.53	246.00	17.90	57.15	465.00	34.80
0.02	55.39	65.60	10.00	58.20	185.00	27.30	57.92	339.00	50.30
0.05	61.02	52.50	18.90	60.07	139.00	50.40	59.30	261.00	95.20
0.1	61.84	41.40	29.50	60.49	117.00	84.20	59.31	206.00	151.00
0.2	63.61	34.80	48.80	62.36	95.30	135.00	58.57	163.00	240.00
0.5	64.96	27.30	94.70	62.93	73.90	261.00	58.79	121.00	445.00
1	65.78	23.10	159.00	61.06	58.70	421.00	56.71	94.30	709.00
2	65.66	19.50	269.00	59.65	46.90	683.00	53.75	70.40	1100.00
5	63.76	15.10	529.00	55.02	32.40	1240.00	48.10	44.30	1870.00
10	60.71	12.00	862.00	50.00	22.70	1860.00	42.46	28.30	2640.00
Sample ED004	Unaged			Aged 48 hours			Aged 120 hours		
0.01	63.64	65.10	4.56	57.09	192.00	14.40	59.53	287.00	21.00
0.02	57.38	45.60	6.80	56.53	150.00	22.60	58.71	219.00	32.30
0.05	60.96	35.80	12.90	59.65	113.00	41.20	60.24	165.00	59.70
0.1	62.76	28.80	20.40	61.49	93.40	66.80	60.80	132.00	95.00
0.2	64.52	24.00	33.40	62.40	77.10	109.00	61.76	107.00	153.00
0.5	65.85	19.00	65.40	63.04	58.90	207.00	60.79	82.00	295.00
1	67.16	16.20	110.00	61.09	47.40	340.00	59.38	64.40	470.00
2	66.40	13.40	184.00	60.02	38.00	552.00	56.61	49.10	739.00
5	65.02	10.70	370.00	56.33	26.70	1010.00	51.96	33.00	1310.00
10	62.92	8.62	608.00	51.99	19.20	1530.00	47.35	22.50	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