Bitumen Durability

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Contents

Executive summary

This report describes research in 2004 and 2005 aimed at improving the means by which the durability of bitumens manufactured or imported to New Zealand for use in chipseals is assessed and monitored.

Bitumen durability refers to the long-term resistance to oxidative hardening of the material in the field. Although, in-service, all bitumens harden with time through reaction with oxygen in the air, excessive rates of hardening (poor durability) can lead to premature binder embrittlement and surfacing failure resulting in cracking and chip loss.

Some means of assessing durability by accelerating the process in the laboratory is necessary. However, no internationally accepted 'standard' exists for bitumen durability, as for some other bitumen tests (e.g. penetration).

Research objectives

Over the past 10 years a procedure developed in the early 1990s has been used as a means to assess the durability of potential bitumen imports to New Zealand. The test measures the moduli of the test bitumen at 5°C (9 Hz) before and after oxidation in 1.0 mm films at 60.0°C and under a pressure of 2069 kPa (300 psi) of air.

This report deals with two distinct aspects of the tests and its application:

(a) The development of a new acceptance (pass/fail) criterion based on a modulus data from chipseals, rather than simply comparative hardening rate as at present.

(b) Investigation of some practical difficulties associated with application of the test to imported bitumens, particularly the fact that it is impractical to measure the durability of the actual shipped bitumen being imported to New Zealand (even if test equipment was available), as the time between manufacture and shipping is usually very short.

(a) Durability test acceptance criteria

Up until the early 1990s all bitumen used in New Zealand was manufactured at the Marsden Point oil refinery almost exclusively from Safaniya crude. Under the current durability test procedure, the rate of hardening (in terms of the increase in complex shear modulus at 5°C at 9 Hz) of the test bitumen is compared to that of 'standard' Safaniya bitumen of the appropriate grade, produced from the Marsden Point refinery in 1990. A more suitable durability test specification limit would be the modulus of Safaniya bitumen found in seals at the end of their life when cracking, chip loss, and scabbing has become significant enough to warrant resealing (the 'modulus at failure'). Bitumens that harden beyond that limit in the durability test would fail the test. This approach uses Safaniya bitumen as a benchmark and would prevent the use of bitumens with lower durability (and hence shorter seal lives).

The time taken in the laboratory oxidation test (60°C, 300 psi) for a particular grade of Safaniya bitumen to reach the modulus at failure level would serve as the standard test period for that grade (i.e. so many hours at 60°C and 300 psi). Trial bitumens would be subjected to the same period in the test and the modulus measured to determine if it exceeds the modulus at failure.

Determination of the modulus at failure

A database of very low-trafficked, local authority streets, e.g. residential cul-de-sacs, was developed. Because of their very low traffic volumes, the lifetime of seals on these streets is most likely to be governed by bitumen oxidation as opposed to flushing or other failure modes. Streets within a single city (Lower Hutt) were used so that it could be reasonably assumed that climate conditions for all sites were approximately uniform. All the seals had been were constructed with a single coat, grade 3, 4, or 5 chip using 180/200 binder (from the Marsden Point refinery) with 2-4 parts per hundred of kerosene. It was assumed that for the streets selected, construction practices used, seal condition assessment practices (to decide which streets to reseal) and pavement deflections present were typical of the country as a whole. The sealing history of the streets selected was used to determine the 80th percentile life time (20 years), and this was taken as an estimate of the maximum seal lifetime.

To determine the modulus at failure, seals of various ages in Lower Hutt were cored and the bitumen recovered. Damage to seals with highly oxidised bitumen is most likely to occur at low temperatures during the winter months when the bitumen is most brittle. Hence the moduli of the extracted bitumens were measured at 5°C (9 Hz), close to the average daily minimum winter (July) temperature for the sites sampled. A value of 100 MPa was determined for the 20-year old seals (with a range of 50-150 MPa). The wide spread of data was ascribed to variations in microclimate and construction variables such as bitumen and chip application rates.

In a region with a higher average winter temperature, although the 100 MPa limit would still apply, the 5°C measurement temperature might not be strictly appropriate. Seals would fail when the moduli reached 100 MPa but at the higher temperature. However, nationwide 95% of average daily minimum winter air temperatures fall in the relatively narrow range -2.7 to 7.8°C. The observed wide spread in moduli values caused by non-(macro)-climate and construction effects, significantly outweighs the potential error introduced by assuming that a single temperature (5°C) is applicable to all regions of the country.

Modifications to the test and acceptance criteria

(1) From previous work, on the original test, the time required (the new test duration) for Safaniya 180/200 and 80/100 grade bitumens to reach the 100 MPa failure modulus was calculated at 8400 and 3200 hours respectively. Specimens are prepared and oxidised as normal except that the test is run for at least 30 days (720 hours) with samples taken at intervals. The moduli measured at 5°C (9 Hz) are extrapolated to the test duration of 3200 hours for 80/100, and 8400 hours for 180/200 grades. If the calculated modulus exceeds 100 MPa the bitumen is deemed to have failed the test.

The drawback with this approach is that, because of the magnitude of the failure modulus, significant extrapolation from the measured data is required. To avoid data extrapolation two alternative approaches to assessing durability were developed:

(2) Oxidation at 80°C (2069 kPa) for 30 or 67 days for 80/100 and 180/200 grades respectively. If the modulus of the test bitumen is greater than 100 MPa the bitumen fails the test. However, this introduces the possibility of significant error, as the relative rates of oxidation of two bitumens may be different from those at field temperatures.

(3) Oxidation at 60°C (2069 kPa) for 30 days. If the modulus of the test bitumen is greater than 86 or 61 MPa for 80/100 or 180/200 grades respectively then the bitumen fails the test. The limits are based on the moduli of Safaniya 80/100 and 180/200 bitumens after 30 days in the test plus 50% (the range of the 20-year failure modulus). This approach assumes that the relative average rate after 30 days (of the test bitumen compared with that of Safaniya bitumen), is a good approximation of that after the much longer time needed to reach the 100 MPa, 20-year modulus.

None of these options are ideal, but the third is preferred as it is simple to perform and avoids problems associated with higher test temperatures.

(b) Practical difficulties associated with the application of the durability test

Importation of bitumen to New Zealand typically involves the supply of a sample for testing to the M/1 specification (including durability testing) and approval by Transit New Zealand (TNZ) some months in advance of the planned shipment date. Production runs for shipment are however typically manufactured only immediately before shipping. Although the main physical properties of the bitumen can be measured and checked for compliance with the TNZ M/1 specification, it is not practical (because of time constraints) to directly confirm the durability before shipment. The possibility thus exists that the shipped bitumen is from a different crude source, a mixture of crude sources, or production route, and has a poorer durability than that originally tested.

Bitumen 'fingerprint'

The usefulness of the standard M/1 test results to develop a 'fingerprint' parameter for a given a bitumen was investigated. Such a parameter could be easily compared to data previously obtained during preliminary testing of the trial bitumen without need for additional testing at shipping. The parameter could be used as an additional requirement for the producing refinery to meet or, alternatively, would be used as an indicator of potential changes that would instigate a confirmatory durability test to be performed on arrival of the shipment in New Zealand.

The retained penetration result from the Rolling Thin Film Oven (RTFO) test and the temperature sensitivity (viscosity ratio) of the bitumen were considered potentially most useful as fingerprint parameters. These properties have the advantage of being expressed as ratios so that inevitable variations of bitumen properties (within the grade limits) from the sample used for approval compared to the production run (even assuming the same source and production route) would not affect the results.

Plots of the RTFO-retained penetration and viscosity ratio for a number of bitumens meeting TNZ M/1 but from different crude sources showed that the precision of the measurements was not sufficient to uniquely characterise a bitumen. Also the specification limits in TNZ M/1 already, necessarily, compress the value of the properties to a narrow range. However these parameters still provide a useful indicative means of confirming the identity of bitumen being shipped to New Zealand, by comparison with data from the sample used for earlier approval testing.

Normally each TNZ M/1 parameter is based on a single measured value. If each laboratory were to make multiple measurements of the retained penetration or viscosity ratio it is likely that the precision of the measurements would be improved significantly, but data was not available to quantify this effect.

Recommendations

1. *The current test used to assess the durability of bitumens in New Zealand should be modified following one of the approaches outlined in Section 2.3.3. Option 3 is preferred, which sets limits of 86 or 61 MPa for 80/100 or 180/200 grades respectively after a 30-day test period at 60°C and 2069 kPa.*
2. *The durability test should be include in the TNZ M/1 specification with a 'report' requirement.*
3. *For imported bitumens the viscosity ratio and RTFO retained penetration should be used as an approximate check that the material to be shipped is the same as the sample used to gain approval.*

Abstract

Three options to improve the test method and acceptance criteria used to assess the in-service durability of bitumen used for chipsealing in New Zealand were investigated in 2004-2005. The preferred option consists of oxidation at 60°C under 2069 kPa air for 30 days. A bitumen would pass if the resulting modulus (at 5°C) did not exceed 86 or 61 MPa for 80/100 or 180/200 grades respectively. The limits are based on the moduli of 'benchmark' Safaniya 80/100 and 180/200 bitumens after the test, plus 50% (based on the modulus range of 20-year old field samples).

For bitumens imported to New Zealand some means of confirming (without re-testing durability) that the material shipped is the same as the sample for which durability had earlier been measured and approval given, is desirable. The viscosity ratio (70C/135°C) and Rolling Thin Film Oven retained penetration ratio were investigated as a means of providing a simple bitumen 'fingerprint' without necessitating additional or specialised testing. As both parameters are ratios, they would not be affected by variations within a grade but only by crude source or production route. It was found that these parameters were too imprecise for the purposes of characterisation.

1. Introduction

This report describes research aimed at improving the means by which the durability of bitumens manufactured or imported to New Zealand for use in chipseals is assessed and monitored.

Bitumen durability refers to the long-term resistance to oxidative hardening of the material in the field. Although, in-service, all bitumens harden with time through reaction with oxygen in the air, excessive rates of hardening (poor durability) can lead to premature binder embrittlement and surfacing failure resulting in cracking and chip loss. In practice all bitumen used in New Zealand for sealing by Transit New Zealand (TNZ) or local authorities must meet the requirements of the TNZ M/1 specification (TNZ 1995a). The specification states that approval for use of a bitumen in New Zealand is subject to the provision of information on satisfactory in-service durability performance.

Bitumen durability is usually assessed by comparison to a benchmark Safaniya bitumen (known to have satisfactory field performance) using results from a laboratory oxidation test. The present work deals with improvements to that process but does not involve attempting to model and predict bitumen hardening and seal life in the field (Oliver 2004).

As seal temperatures rarely exceed 60°C in temperate climates, oxidative hardening of bitumen in chipseals is a very slow process taking place over many years. Some means of assessing durability by accelerating the process in the laboratory is necessary. However, unlike some other bitumen tests (e.g. penetration) no internationally accepted 'standard' test exists for bitumen durability. A wide range of methods has been reported in the literature for accelerating the rate of bitumen oxidation, based on either increasing the temperature or the concentration of oxygen in the bitumen, or both (for reviews see Airey 2003, Bell 1989). Neither approach is a perfect model for oxidation in the field. The use of high temperatures can result in changes to the relative oxidation rate of different bitumens, so that two bitumens that have similar rates at ambient temperature may have different rates at higher temperatures (Branthaver et al*.* 1993). This effect is illustrated in Figure 1.1 which compares the increase in shear modulus (G* at 5°C, and 9 Hz) for two bitumens during oxidation under 2069 kPa of air. At 40°C the oxidative hardening rate of the Safaniya bitumen is very similar to that of the Venezuelan bitumen, but at 80°C the relative rates diverge. Assessment of the relative durability behaviour of the two materials would depend on the oxidation test temperature selected.

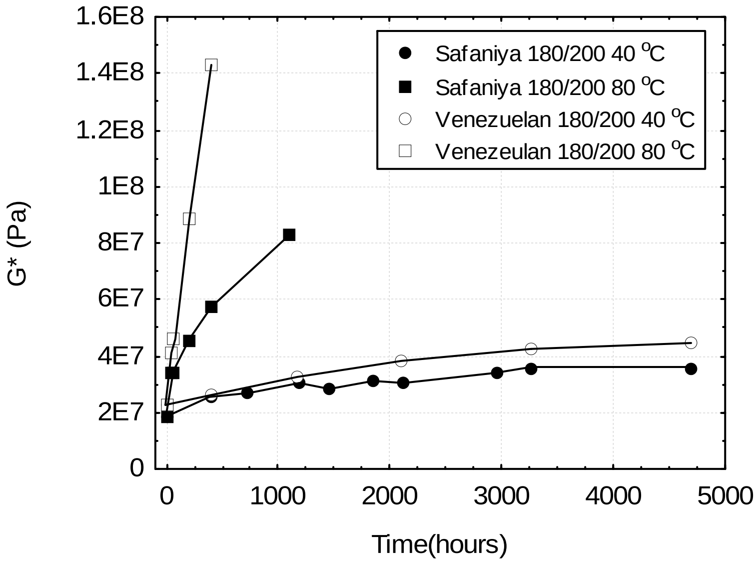

Figure 1.1 Effect of oxidation temperature (40°C and 80°C) on relative oxidation rate for a Venezeulan 180/200 and Safaniya 180/200 bitumens (Herrington 2000).

In the field oxygen gradually diffuses into bitumen films where it reacts. The concentration of oxygen is lower in the centre of the film than at the upper surface. In the laboratory the reaction rate can be increased by increasing the average concentration of oxygen in the film, usually by increasing the air pressure above the film or by using very thin films. Some evidence exists that increasing the average oxygen concentration may affect the spatial distribution of oxidation products between maltene and asphaltene phases in the bitumen compared to that found at lower concentrations (Domke et al. 1999). However, for Safaniya bitumen at 60°C the use of high oxygen pressure did not have a significant effect on the viscosity achieved for a given degree of oxidation (Herrington 2005).

1.1 Research objectives

Over the past 10 years a procedure developed in the early 1990s (Herrington 2000) has been used as a means to assess the durability of potential bitumen imports to New Zealand (see Appendix 1). The test measures the moduli of the test bitumen at 5°C (9Hz) before and after oxidation in 1.0 mm films at 60.0°C and under a pressure of 2069 kPa (300 psi) of air. The 5°C modulus was selected as a low temperature parameter relevant to cracking and oxidation-related failure mechanisms.

This report deals with two distinct aspects of the tests and its application:

- the development of a new acceptance criterion based on a modulus limit determined from 'end of life' seals rather than simply hardening rate (Chapter 2),
- investigation of some practical difficulties associated with application of the test to imported bitumens (Chapter 3), particularly the fact that it is impractical to measure the durability of the actual shipped bitumen being imported to New Zealand (even if test equipment was available), as the time between manufacture and shipping is usually very short.

2. Durability test acceptance criteria

2.1 Background

Up until the early 1990s all bitumen used in New Zealand was manufactured at the Marsden Point oil refinery from a small group of approved Middle East crudes and in practice almost exclusively from Safaniya crude. Under the current durability test procedure, the rate of hardening of the test bitumen is compared to that of 'standard' Safaniya bitumen of the appropriate grade, produced from the Marsden Point refinery in 1990. Hardening of the bitumen is measured in terms of the increase in complex shear modulus at 5°C at 9 Hz. The measurement is made at 5°C as seal damage caused by oxidised bitumen is most likely to occur at low temperatures. When the test was developed the assumption was made that, as the initial physical properties of the bitumen are controlled by the viscosity and penetration limits in the M/1 specification, the actual value of the moduli need not be considered in the test (only the change). However, experience has shown that for bitumens meeting the requirements of M/1, variations in temperature sensitivities can be such that the initial value of the moduli can vary by factors of 200-300% even though penetrations at 25°C may be similar. In qualitative terms high moduli at low temperatures are not desirable, and as it is clear that the M/1 specification does not adequately control the moduli at 5°C, then basing durability assessment on hardening rate alone is insufficient and a specific modulus value is needed to serve as a specification limit. Bitumens that harden beyond that limit in the laboratory durability test would fail the test.

2.2 Methodology

2.2.1 Development of new durability test acceptance criteria

A suitable durability test specification limit would be the modulus of Safaniya bitumen found in seals at the end of their life when cracking, chip loss, and scabbing has become significant enough to warrant resealing (the 'modulus at failure'). Bitumens that harden beyond that limit in the durability test would fail the test. This approach uses Safaniya bitumen as a benchmark and would prevent the use of bitumens with lower durability (and hence shorter seal lives). The time taken in the laboratory oxidation test (60°C, 300 psi) for a particular grade of Safaniya bitumen to reach the modulus at failure level would serve as the standard test period for that grade (i.e. so many hours at 60°C and 300 psi). Trial bitumens would be subjected to the same period in the test and the modulus measured to determine if it exceeds the modulus at failure.

The following sections describe various aspects of the implementation of this approach in more detail.

2.2.2 Determining 'end of life' age

The age at which seal failure caused by oxidation-related problems occurs will depend on traffic loading, although the actual rate of hardening (caused by reaction with oxygen) will be independent of traffic. To remove traffic as a dominant variable, a database of very low-trafficked, local authority streets e.g. residential cul-de-sacs, was developed. Such streets typically had traffic levels of less than 100 vehicles/lane/day (v/l/d). Because of their very low traffic volumes, the lifetime of seals on these streets is most likely to be governed by bitumen oxidation as opposed to flushing or other failure modes.

Streets within a single city (Lower Hutt) were used so that it could be reasonably assumed that climate conditions for all sites are approximately uniform. The effect of climate is discussed further in Section 2.2.4.

As bitumen film thickness will affect the oxidation rate (because of the shorter diffusion path for oxygen) the streets selected were constructed with a narrow range of chip sizes. With a few exceptions all seals had been were constructed with a single coat, grade 4 or grade 3 chip (as are most New Zealand seals) using 180/200 binder (from the Marsden Point refinery) and with 2-4 parts per hundred (pph) of kerosene.

The sealing history of the streets was used to determine an average, ultimate seal lifetime. It was assumed that for the streets selected, construction practices used, seal condition assessment practices (to decide which streets to reseal) and pavement deflections present, were typical of the country as a whole. These streets were taken to represent the ultimate lifetime that can be achieved using typical seal construction practices. Failure is essentially entirely related to the durability limits of the bitumen.

2.2.3 Determination of modulus at failure

To determine the modulus at failure, seals of various ages in Lower Hutt were cored and the bitumen recovered by extraction with dichloromethane. All the sites were within a 3-4 km radius. Details of the coring and bitumen recovery process are given in Appendix 2. Some sites had multiple cores removed. Figures 2.1 to 2.4 show photographs of a typical site and the cores removed.

Figure 2.1 View of typical grade 3 seal surface (with lichen).

Figure 2.2 Typical field site. Note lichen growth on old seal section.

Figure 2.3 Core before bitumen extraction.

Figure 2.4 Removal of chips for bitumen extraction.

The modulus value determined from 180/200 bitumen applies also to 80/100 and 130/150 bitumens. Binders of different grades will require different times in the field to reach the modulus at failure level but (given that the chemical composition of different bitumens is very similar) it is reasonable to assert that the actual modulus at which failure starts to occur will be essentially the same. Hence a modulus at failure obtained from seals of a given grade in a given city should be applicable to other grades and the rest of the country as long as the construction practices used and pavement deflections present are typical.

2.2.4 Modulus measurement treatment

A key variable in determining the failure modulus is the temperature at which the measurement is made. Damage to seals with highly oxidised bitumen is most likely to occur at low temperatures during the winter months when the bitumen is most brittle. For a particular site, as the bitumen oxidises damage to the seal accumulates over successive winters until a failure point has been reached. The highest average level the bitumen modulus has reached at that point in time (the modulus at failure) would be obtained by measuring the modulus at the average daily minimum winter temperature (or some other suitable measure of winter temperature) for that site. For Lower Hutt the average daily minimum temperatures for July (1961 to 1990) was 4.8°C, hence the moduli of the extracted bitumens was measured at 5°C (see Table 2.1). The range of minimum and maximum temperatures (also given in Table 2.1) indicate a consistent climate over the area from which the sites were selected.

Table 2.1 Average temperatures for Lower Hutt 1961-1990 (Tomlinson & Sansom 1994).

Station	Average minimum daily temperature (°C) (July)	Average maximum daily temperature (°C) (February)
Taita	4.5	21.4
Avalon	5.1	21.8
Gracefield	5.5	20.0
Wainuiomata	4.2	21.2

2.3 Results and discussion

2.3.1 Determining the 'end of life' age

Results of the analysis of the Lower Hutt street database are given in Table 2.2 and the distribution of seal lives shown in Figure 2.5.

Table 2.2 Seal lifetimes for low-trafficked streets in Lower Hutt.

Property (Number of streets = 133)	Value (years)
Mean lifetime	13.7
Minimum lifetime	1.0
Maximum lifetime	42.7
Lower quartile	6.4
Upper quartile	18.1
80 percentile	20.1
90^{th} percentile	26.1

The mean lifetime, 13.7 years, is about that expected (12-14 years) for <100 vehicles per day (vpd) grade 3 and 4 reseals (TNZ 1995b). The distribution of seal lives is however not normal and a high proportion of seals fail well below the expected life, which is also characteristic of State Highway seals and warrants further investigation (Ball & Patrick 2005).

To avoid distortion of the results from outliers, the upper 80^{th} percentile, 20.1 years, was used as a reasonable estimate of the maximum seal age of 'end of life' sites.

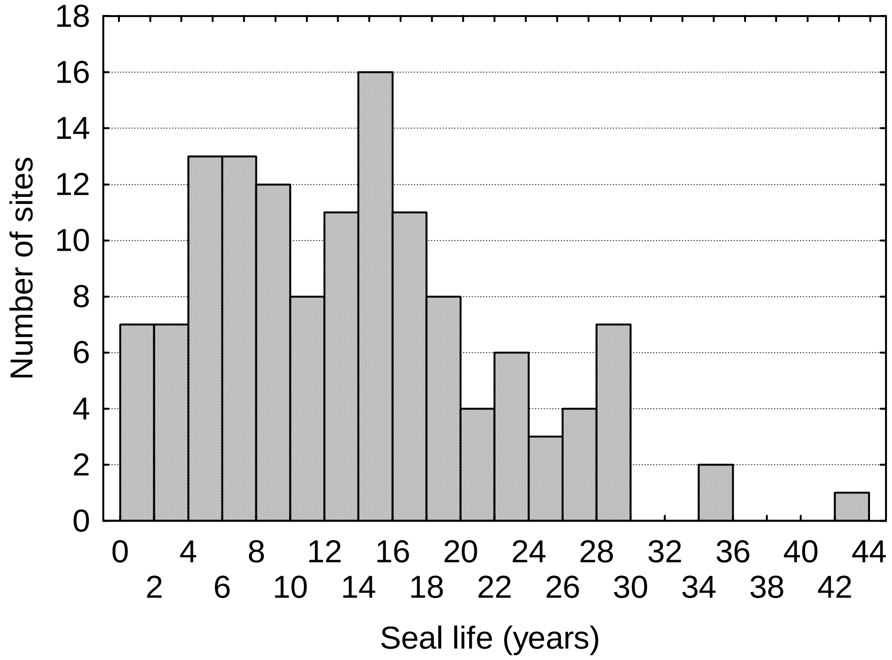

Figure 2.5 Distribution of seal lives for low-trafficked streets in Lower Hutt.

2.3.2 Field site data

Extracted bitumen moduli are plotted in Figure 2.6 (the data are given in tabular form in Appendix 3). The seals at the sites chosen were not necessarily at the point of failure (in most cases, except for the older sites, seal condition was good), but were selected simply to span a wide a range of seal ages.

The zero points in the plot are the moduli of Safaniya 180/200 bitumens manufactured at Marsden Point in 1986, 1990, and 1997. Data obtained earlier from a grade 3 seal at Chatto Creek (State Highway 85, Central Otago), and from grade 2 and 4 artificial seals exposed outdoors in Lower Hutt (Ball 1999), are included for comparison.

Immediately apparent is the large scatter of the data which becomes greater as age increases. Replicate measurements on the extracted bitumen (and replicate extractions on two cores) indicated a standard deviation of 5.5 MPa for the modulus measurement itself, which is not significant compared to the variation in the data, i.e. the variation is not simply caused by extraction and measurement error (Table 2.3).

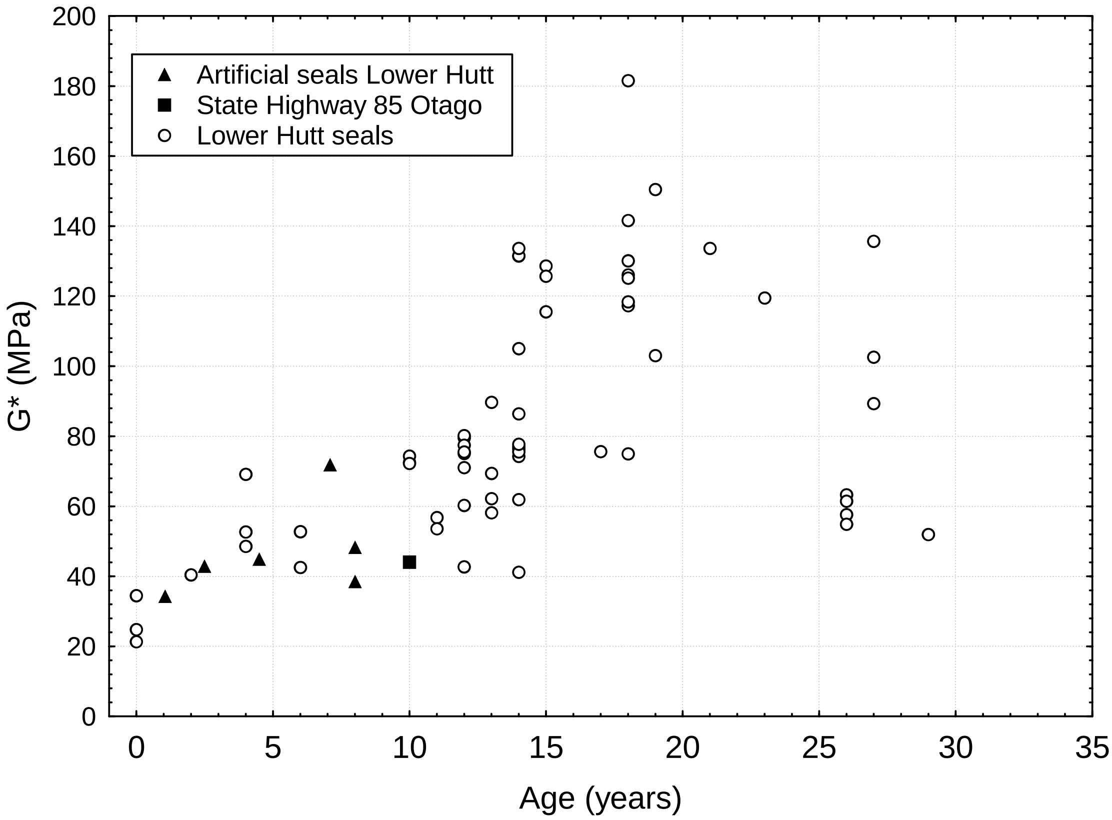

Figure 2.6 Shear moduli (G*) measured at 5°C, 9 Hz for bitumens extracted from seals.

Table 2.3 Precision of the moduli measurement.

Site	**Age** (years)	**Replicate**	**Shear Modulus (MPa)**
Cleary St	18	1	134.9
		2	115.4
Kennedy Gr.(1)	14	1	129.3
		2	133.6
Maire St(1)	26	1	58.20
		2	56.87
Newburn Gr.	23	1	113.1
		2	125.9
Pilmuir St	21	1	133.2
		2	134.2
Pooled standard deviation			**5.5**

(1) Replicate extraction from the same core.

As the seals were constructed over a very long period of time, variation in the moduli data because of variations in the initial bitumen moduli are also to be expected, but the zero time data indicate that this can only explain a small proportion of the scatter. As each extraction involves only a very small sample of the seal, multiple cores were taken from several of the sites to investigate variation across the road (Table 2.4). The variation observed is in some cases significantly greater than that expected from error associated with the extraction process and modulus measurement and must be caused by site specific factors. The observed variations within and between sites within what is a relatively small geographic area, is probably caused by a combination of factors such as localised micro-climates, bitumen application rates, residual kerosene, aggregate absorption of bitumen oils, and mixing into underlying bitumen, all of which will affect oxidation rates.

Table 2.4 Moduli variation within sites.

Site	Age (years)	Core	Shear Modulus (MPa)	Mean Shear Modulus (MPa ± standard deviation)
Anson Gr.	14	1	105.1	96 ± 13
		2	86.36	
Barraud St	15	1	128.6	123 ± 7
		2	115.6	
		3	125.7	
Cleary St	18	1	125.2	125 ± 6
		2	130.1	
		3	118.4	
Kamahi St	14	1	61.93	109 ± 41
		2	131.6	
		3	133.7	
Kennedy Gr.	14	1	131.5	83 ± 45
		2	77.74	
		3	41.09	
Kopara Gr.	12	1	77.48	75 ± 3
		2	75.52	
		3	71.03	
Maire St	26	1	57.54	58 ± 3
		2	54.87	
		3	61.48	
Ngaturi Gr.	27	1	135.7	109 ± 24
		2	102.6	
		3	89.3	
St Columbans Gr.	18	1	126.1	150 ± 29
		2	141.6	
		3	181.5	

Bitumen design application rates, and thus average film thickness, increase with chip size (grade 5 to 2) so that oxidation rates would be expected to be lower for the larger chip sizes (because of a longer oxygen diffusion path). The Lower Hutt seal data (artificial seals excluded) in Figure 2.6 are replotted in Figure 2.7 but with the chip grade marked.

The distribution of chip grade is not uniform over seal age. The older seals tend to be grade 3 while the younger tend to be grade 4, probably reflecting changes in sealing policy. No clear trends are apparent: for a given chip grade, seals of approximately equivalent age show a wide range of moduli. The distribution of moduli for each grade is similar (Figure 2.8) although the grade 5 seals appear to have on average higher moduli than the grade 3 even though they are younger. The effect of film thickness on the increase in viscosity with age has been demonstrated previously with artificial grade 2, 3 and 4 seals (Ball 1999). Even though the seals were carefully constructed on smooth substrates the differences were relatively small. Australian field data (Oliver 2005) showed a wide distribution of results for seals of 10 and 14 mm nominal chip size in the same climate zone. No significant difference between the viscosity of the bitumen after 8 years in the field was found. Direct observation of seals of the same nominal chip size (and presumably similar design application rates) had shown that film thicknesses could be significantly different. Differences between chip grades (bitumen film thickness) will tend to be obscured by both localised variation in substrate surface texture and differences in chip application rates (which are generally not well defined), both of which can have a major effect on the resulting film thickness.

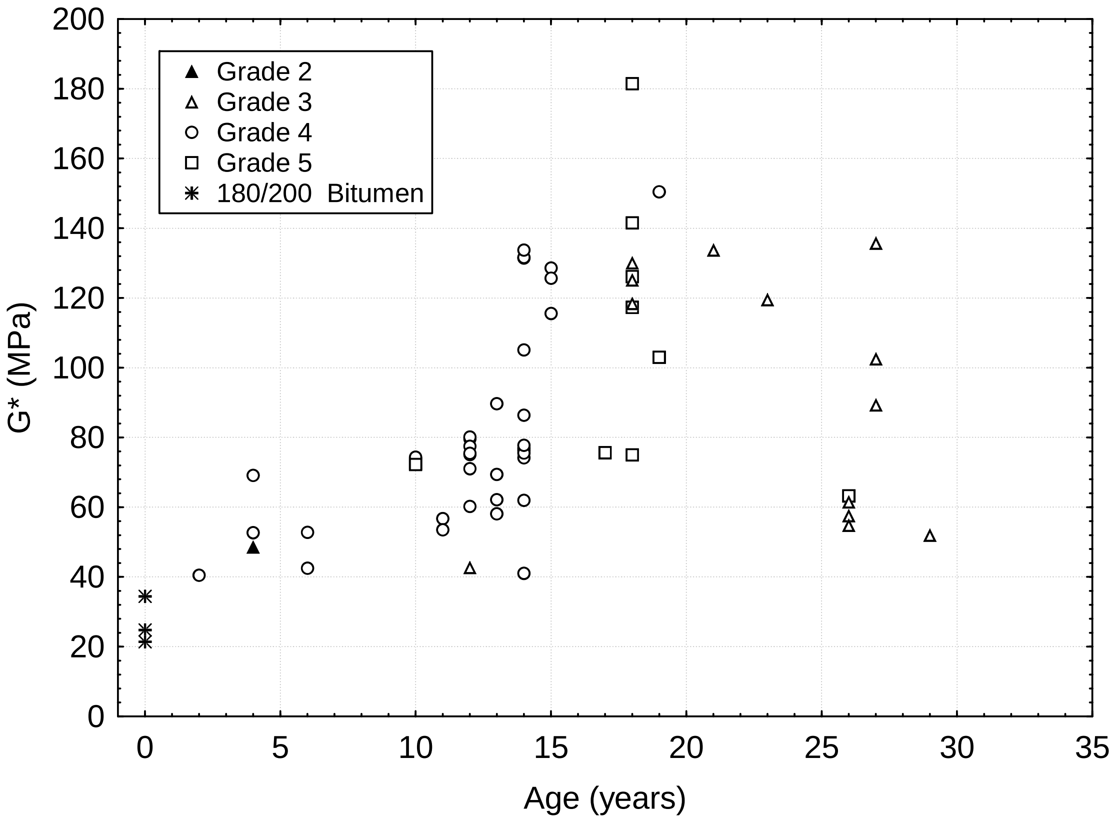

Figure 2.7 Shear moduli (G*) measured at 5°C, 9 Hz, for bitumens extracted from Lower Hutt seals showing effect of age and chip grade.

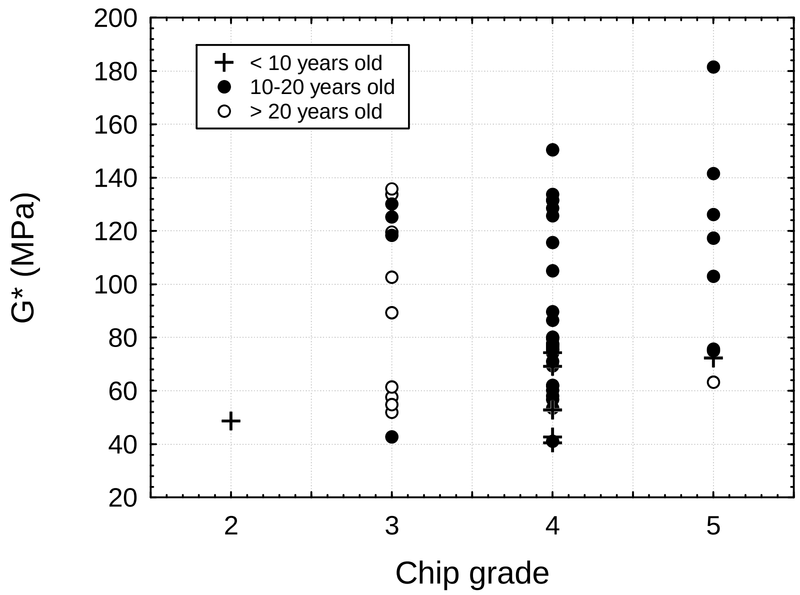

Figure 2.8 Shear moduli (G*) measured at 5°C, 9 Hz, for bitumens extracted from Lower Hutt seals showing effect of chip grade.

It should be noted that all seals were constructed with 2-3 pph kerosene. Earlier field trials in Lower Hutt have shown that about 75% of kerosene added is lost within a year of application and a slow rate of loss continues subsequently (Ball 1992, Herrington & Ball 1994). Any residual kerosene would be lost during the high vacuum stage of the recovery process (Appendix 2), although it is highly unlikely that any would remain after 20 years (so that the effect on the failure modulus would be negligible). In any case, loss of residual kerosene is of course not significant for the present work, as unmodified bitumen is used in the durability test and the object was to determine a bitumen modulus for use as a test criterion.

From Section 2.3.1 the 80^{th} percentile seal age in Lower Hutt was calculated at 20 years. At this age the bitumen moduli (the failure moduli), range from about 50 to 150 MPa with an average of about 100 MPa (Figure 2.6). The moduli data discussed above are measured at 5°C, close to the average minimum winter temperature of the seals concerned. If the bitumen used in these sites were used at a site with a higher average winter temperature, although the modulus at failure value should in principle be the same, seals would have reached failure point when the failure modulus was reached at the higher temperature. Seals at a warmer location would thus take longer to reach the failure modulus than at a colder one (although this may be offset by a faster rate of oxidation in the warmer climate). In theory it may be overly conservative to test bitumen destined for use in the warmer climate using moduli measurement temperature suitable for the colder region as in some cases the warmer site may never experience temperatures that low. The above discussion is illustrated schematically in Figure 2.9 (for most bitumens the relationship between log G* and temperature is linear over the range of interest).

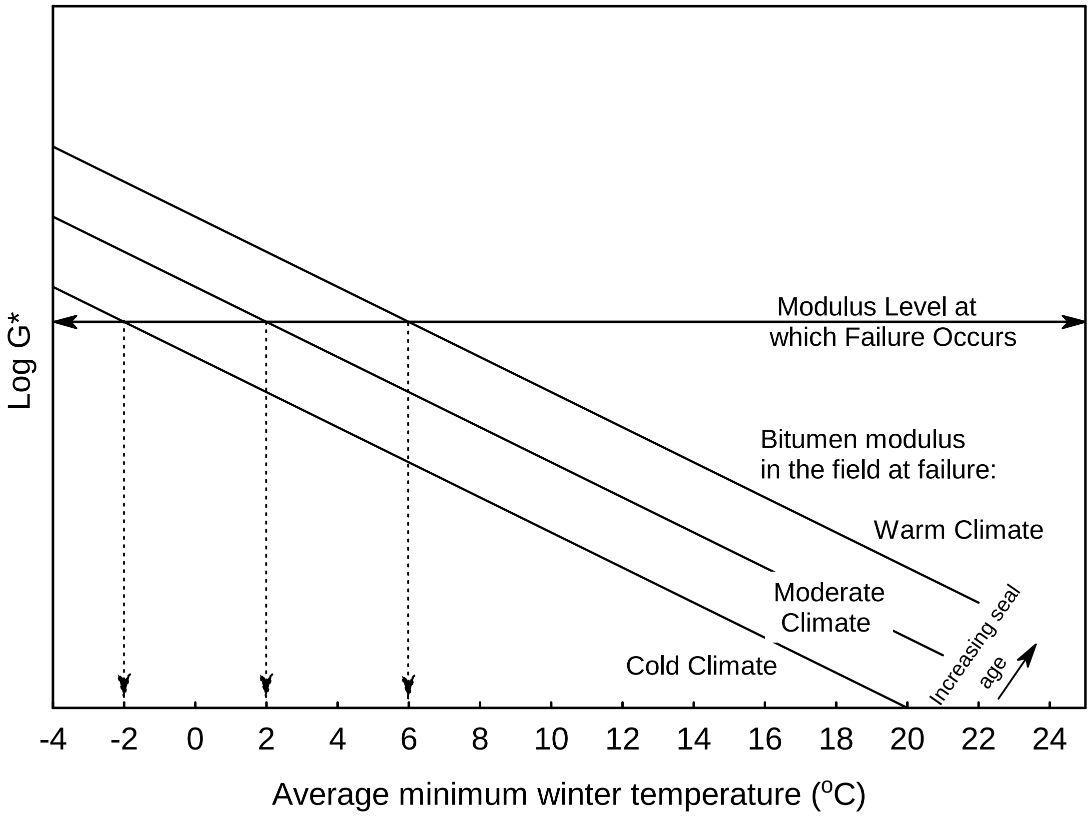

Figure 2.9 Effect of climate on the temperature at which the failure modulus should be measured.

Acceptance criteria or a specification based on this reasoning would in practice require the country to be divided into climate zones and different moduli measurement temperatures adopted for each region. As the time required (at 60°C and 2069 kPa air) for the benchmark Safaniya bitumen to reach the modulus at failure level will vary with measurement temperature, a different laboratory test duration would be necessary for each region.

Such an approach allows the possibility that bitumen from a particular source may be suitable for use in some parts of the country but not in others. This is the case with bitumens of different grades at present but differentiation on the basis of source would potentially introduce considerable extra logistical problems and complications.

However, nationwide 95% of average minimum winter temperatures (June-July) are in the −2.7 to 7.8°C range (Tomlinson & Sansom 1994). Given this relatively narrow (10°) range of winter temperatures, to be practically meaningful the introduction of climate zones would require a high degree of precision in the modulus at failure level. For comparison, a typical Safaniya 180/200 bitumen with a modulus of 21 MPa at 5°C (9 Hz) has a modulus of 38 MPa at 0°C and 0.8 MPa at 10°C. Hence over a 10°C range the modulus changes by about 37 MPa compared to the observed range of failure moduli of about 100 MPa. Thus the development of acceptance criteria based on temperature zones was not considered warranted at present. Continued use of a 5°C moduli measurement in the test was considered a reasonable compromise.

2.3.3 Durability test acceptance criteria and test modification

As discussed above, with a modulus limit set at 100 MPa then the duration of the durability test would be determined by the time taken for a standard Safaniya bitumen of the appropriate grade to reach the average failure modulus of 100 MPa.

From previous work (Herrington 2000), it is known that in general the increase in bitumen modulus during the durability test (60°C, 2069 kPa air) can be described by Equation 2.1.

$$Log\ G^* = Log\ G_0^* + P(1-exp(-K_f\, t)) + Kt \qquad \text{(Equation 2.1)}$$

where: G^* = shear modulus after time t
G_0^* = initial shear modulus
t = test time at 60°C and 2069 kPa air
P, K_f and K are fitted constants

Values of the constants for Safaniya 180/200 and 80/100 grades, based on results given in Herrington (2000) and supplemented with additional experimental data up to 2000 hours oxidation time, are given in Table 2.5. Data for 60/70 and 130/150 grades is not available at present. The curves are plotted in Figure 2.10 (data points have been omitted for clarity). The dashed lines in Figure 2.10 were drawn using the upper value of the 95% confidence interval for each parameter and represent an estimate of the upper limit of the oxidation rate for the 80/100 and 180/200 bitumens respectively.

Table 2.5 Fitted parameters (± 95% confidence limits) for Safaniya 80/100 and 180/200 bitumens.

Bitumen	G_0^* (MPa)[1]	P	K_f	K
180/200	24 ± 4[2]	0.168 ± 0.02	0.0128 ± 0.0003	(5.15 ± 0.05) × 10^{-5}
80/100	26 ± 4[2]	0.238 ± 0.03	0.076 ± 0.0006	(6.5 ± 3) × 10^{-5}

[1] The value of G_0^* is based on measurement of Safaniya bitumens produced in 1987, 1990 and 1997, and for 80/100 on that of 1990 and 1997 bitumens

[2] Based on pooled standard deviation

From Figure 2.10 Safaniya 180/200 and 80/100 bitumens require 8800 and 5300 hours respectively to reach a modulus of 100 MPa. This is beyond the range of experimental data currently available, although experiments carried out at 40°C confirm that Equation 2.1 is still obeyed at least up to 5000 hours. The error involved in the extrapolation (as indicated by the dashed lines) is reasonable for the 180/200 but larger for the 80/100 caused by a greater scatter in the data and needs to be refined.

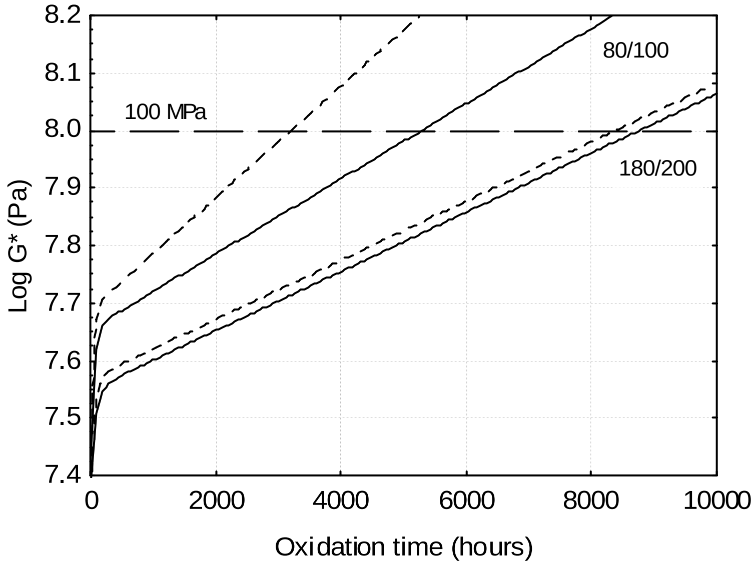

Figure 2.10 Behaviour of Safaniya 80/100 and 180/200 bitumens oxidised at 60°C and 2069 kPa air (durability test). Dashed lines represent upper estimates of oxidation rate.

For the purposes of the durability test it is reasonable to take the minimum estimated time for 180/200 and 80/100 Safaniya bitumens to reach the failure modulus (3200 and 8400 hours respectively) as the new test duration. Examination of the durability of trial bitumens over such long test periods is clearly impracticable and extrapolation of a curve fitted to the test bitumen data would be required in practice. A minimum test period however of about 30 days (720 hours) would be necessary to ensure the approximately linear portion of the modulus–time curve had been reached. If the predicted modulus of a test bitumen exceeded the 100 MPa limit it would fail the test.

Further work is required to obtain test data for standard Safaniya 60/70 and 130/150 bitumen grades, so as to determine an appropriate test duration. Additional data for the 80/100 and 180/200 grades up to 100 MPa is also highly desirable.

It should be noted that the use of a specific upper limit on modulus value after oxidation in the test has consequences for bitumens that are more temperature-sensitive than Safaniya. All bitumens used in New Zealand must meet a fairly narrow 25°C penetration range so that at 25°C all bitumens will probably have similar moduli values. At the low temperatures relevant to durability assessment, bitumens more temperature-sensitive than Safaniya are always likely to have a higher initial modulus than the equivalent Safaniya grade. Such bitumens may not pass the test even if their rate of hardening is less than that of Safaniya. This is illustrated in Figure 2.11.

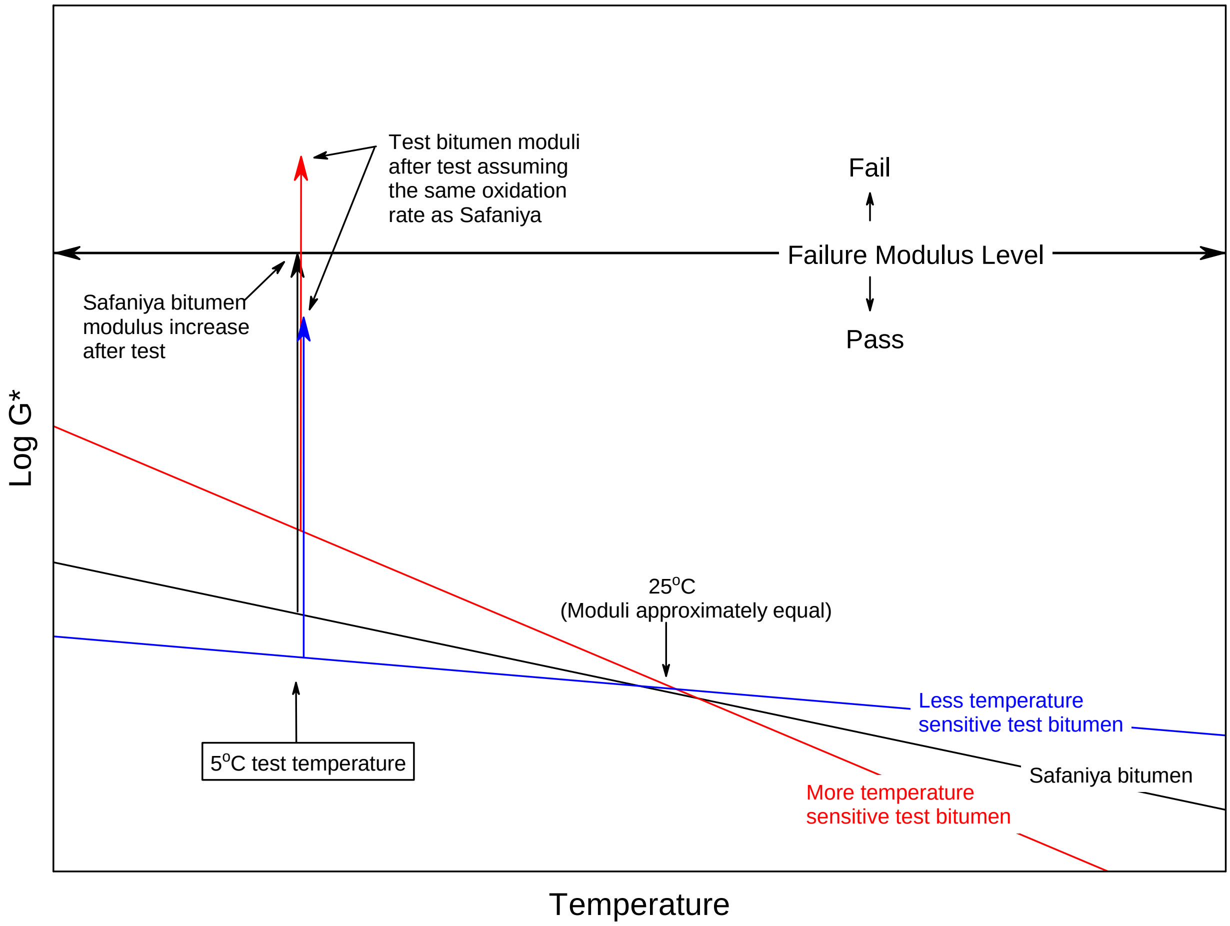

Figure 2.11 Schematic representation of moduli changes during the durability test illustrating the effect of bitumen temperature sensitivity.

To avoid possible errors introduced by data extrapolation, the test could be run at a higher temperature (higher air or oxygen pressures give rise to only minimal increases in oxidation rate). At 80°C (and 2069 kPa) for example, a test period of 67 days (30 days for an 80/100 grade) would be required for Safaniya 180/200 bitumen to reach the 100 MPa modulus level (Herrington 2000). However as discussed in the introduction (Figure 1.1), this introduces the possibility of significant error, as the relative rates of oxidation of two bitumens may be different from those at field temperatures. The initial moduli of the Safaniya and Venezuelan bitumens in Figure 1.1 were 22 and 24 MPa respectively. After only 400 hours at 80°C the Safaniya modulus was 70 MPa whereas that of the Venezuelan was 140 MPa. If the same relative rates were observed at 40°C the Venezeulan bitumen modulus should be about 52 MPa whereas in practice it was only half that value, i.e. the relative rate is about half that expected from the 80°C data.

A third and simpler approach to assessing durability would be to recognise the fact that construction and site factors (e.g. film thickness) appear to have a major affect on oxidation rates in the field. The moduli of bitumen in seals near failure vary widely. From Figure 2.6, after 20 years the range is about ± 50% of the mean. Small variations in inherent bitumen durability are thus likely to be less important in comparison to these effects and the purpose of the test is thus primarily to detect extremely poor materials. On this basis the durability test would consist of a 30 day test period at 60°C at which time the test bitumen modulus must not exceed that of the benchmark Safaniya by more than 50%. From Figure 2.10 the upper allowable modulus would thus be 61 MPa for 180/200 and 86 MPa for 80/100. This approach assumes that the relative average rate after 30 days (of the test bitumen compared with that of Safaniya bitumen) is a good approximation of that after the much longer time needed to reach the 100 MPa, 20-year modulus.

In summary three different approaches for assessing durability are suggested:

(1) Oxidation at 60°C (2069 kPa) for 30 days, curve fitting and extrapolation of the fit to predict the modulus at 3200 or 8400 hours depending on the grade. If the modulus of the test bitumen is greater than 100 MPa the bitumen fails the test.

(2) Oxidation at 80°C (2069 kPa) for 30 or 67 days for 80/100 and 180/200 grades respectively. If the modulus of the test bitumen is greater than 100 MPa the bitumen fails the test.

(3) Oxidation at 60°C (2069 kPa) for 30 days. If the modulus of the test bitumen is greater than 86 or 61 MPa for 80/100 or 180/200 grades respectively then the bitumen fails the test.

None of these options are ideal, but the third is preferred as it is simple to perform and avoids problems associated with higher test temperatures.

If a particular grade of bitumen meets the requirements of the test then the testing of other grades manufactured from that base stock may be unnecessary unless the modification process significantly alters the oxidation rate of the bitumen or increases its temperature sensitivity. It is envisioned that the testing requirements in such situations would be at the discretion of Transit NZ.

3. Assessing the durability of imported bitumens

3.1 Background

Importation of bitumen to New Zealand typically involves the supply of a sample for testing to the M/1 specification (including durability testing) and approval by Transit NZ, some months in advance of the planned shipment date. Production runs for shipment are however typically manufactured only immediately before shipping. Essentially, all quality control testing must be carried out within a day, and using internationally recognised standard methods, usually ASTM (American Society for Testing and Materials). The current durability test method takes about one week and is not used elsewhere in the world. Numerous other durability tests exist but usually also take extended periods to complete and are 'non-standard'. Hence although the main physical properties of the bitumen can be measured and checked for compliance with the M/1 specification, it is not practical to directly confirm the durability before shipment. The possibility thus exists that the shipped bitumen is from a different crude source, a mixture of crude sources or production route, and has a poorer durability than that originally tested. The use of chemical analyses for metals or biomarkers to 'fingerprint' the bitumen is not feasible because of time, expertise and the facilities likely to be available.

In the absence of any alternative the usefulness of the standard M/1 test results to develop parameters for characterising a given bitumen was investigated. Such parameters could be easily compared to data previously obtained during preliminary testing of the trial bitumen without need for additional testing at shipping. The parameters would be used as an additional specification requirement for the producing refinery to meet or alternatively, the parameter would be used as an indicator of potential changes that would instigate a confirmatory durability test to be performed on arrival of the shipment in New Zealand.

3.2 Characteristic parameters based on TNZ M/1 test results

Details of the bitumens used in this investigation are given in Tables 3.1 and 3.2. The bitumens all met the physical requirements of TNZ M/1 and were from a range of crude sources or mixtures of crude sources. Tests were carried out according to the methods given in Table 1 of TNZ M/1.

Table 3.1 180/200 bitumen properties.

Bitumen	Crude Origin	Penetration (dmm)	Retained Pen (%)	Viscosity at 70°C (mm^2s^{-1})	Viscosity at 135°C (mm^2s^{-1})	Viscosity Ratio (V_{70}/V_{135})
1	Venezuela	172	69	18,000	179	101
2	Venezuela	183	64	18,700	233	80
3	Venezuela	183	61	16,100	213	76
4	Venezuela	172	69	18,000	191	94
5	Venezuela	184	60	19,730	244	81
6	Mid East	177	65	19,000	238	80
7	Venezuela	186	62	20,400	263	78
8	Mid East	181	77	15,300	182	84
9	Venezuela	194	58	16,700	214	78

Table 3.2 80/100 bitumen properties.

Bitumen	Crude Origin	Penetration (dmm)	Retained pen. (%)	Viscosity at 70°C (mm^2s^{-1})	Viscosity at 135°C (mm^2s^{-1})	Viscosity ratio (V_{70}/V_{135})
1	Venezuela	82	62	56,000	409	137
2	Asia	84	67	44,500	362	123
3	Mid East	96	62	37,900	338	112
4	Mid East	93	66	36,000	315	114
5	Asia	92	70	42,900	365	118
6	Mid East	88	60	78,400	438	179
7	Mid East	85	66	45,800	378	121
8	Venezuela	89	61	37,600	435	86
9	Venezuela	85	64	42,100	323	130
10	Mid East	80	75	65,900	461	143
11	USA	88	63	38,400	303	127

Previous researchers have shown that no simple relationship exists between the physical properties of a bitumen and its resistance to oxidative hardening, i.e. it is not possible to predict field durability from simple physical measurements of the bitumen. However it can be postulated that factors that affect durability, in particular crude source and processing route (e.g. air blowing, solvent precipitation), are also likely to affect the temperature sensitivity (viscosity ratio) of the bitumen and the Rolling Thin Film Oven (RTFO) test result (the RTFO test is essentially a high temperature oxidation test). Of the test data provided by M/1 (listed in Table 3.3) these properties were thus considered potentially most useful for bitumen characterisation.

Table 3.3 TNZ M/1 tests.

Test Parameter	Method
Penetration at 25°C (dmm)	ASTM D5
Viscosity at 70°C (mm^2s^{-1})	AS 2341.3, ASTM D2170
Viscosity at 135°C (mm^2s^{-1})	AS 2341.3, ASTM D2170
Flash point (°C)	ASTM D92
Solubility in trichloroethylene (%)	ASTM D2042
Retained penetration after RTFO Test (%)	M1:1995 Section 1.1, ASTM D5
Ductility after RTFO Test (m)	ASTM D113

These properties also have the advantage of being expressed as ratios: the RTFO test result is the ratio of penetrations before and after the oven treatment and the temperature sensitivity can be defined as the ratio of viscosities at 70°C (or 60°C) and 135°C. Hence inevitable variations of bitumen properties (within the grade limits), from the sample used for approval compared to the production run (even assuming the same source and production route) would not affect the results.

Plots of viscosity ratio (70°/135°C) versus retained penetration are given in Figures 3.1 and 3.2. The error bars for the viscosity ratios represent one standard deviation of the mean calculated from the multiple laboratory precision data given in ASTM D2170-01a and ASTM D2171-01. Precision data for the retained penetration is that given in Ball (1984) from a New Zealand multiple laboratory round robin carried out in 1984.

From Figures 3.1 and 3.2, clearly the error associated with the test procedures (in particular the retained penetration results) is such that the parameters are not unique even for the relatively limited number of bitumens investigated. Normally each parameter is based on a single measured value. If each laboratory were to make multiple measurements of the retained penetration or viscosity ratio, it is likely that the precision of the measurements would be improved significantly. Even so, these parameters do demonstrate sufficient differentiation to suggest that they may provide a useful indicative means of checking bitumen identity. Hence if the retained penetration and viscosity ratio results for the bitumen being shipped fall outside the range expected from the earlier approval sample, then the two bitumens are probably different.

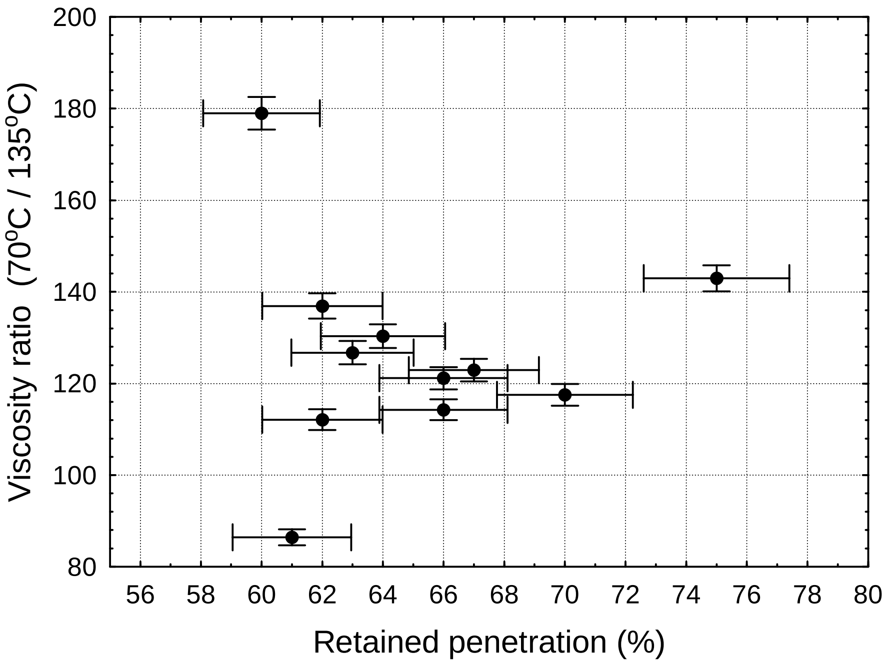

Figure 3.1 Retained penetration and viscosity ratio for 80/100 bitumens (±σ).

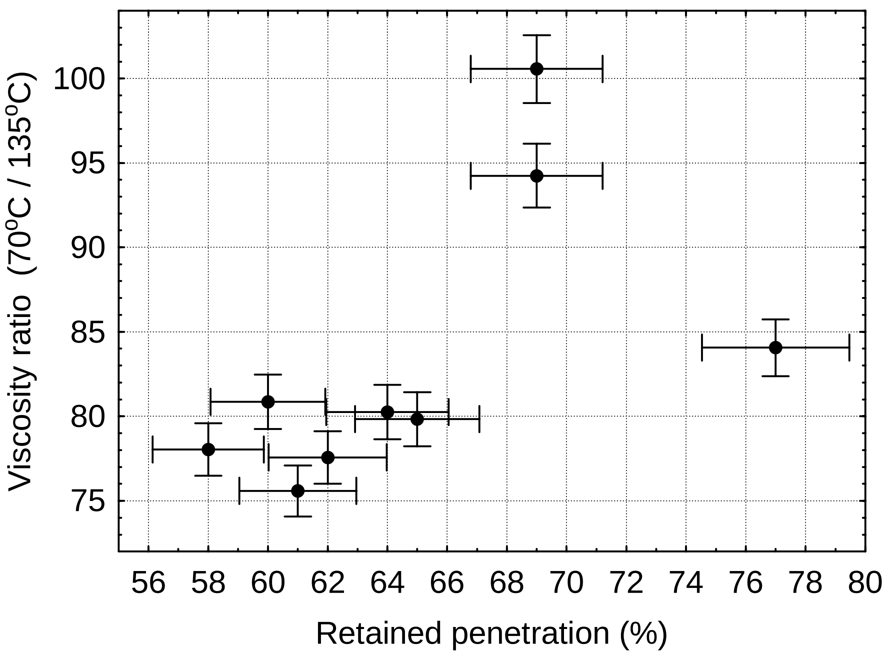

Figure 3.2 Retained penetration and viscosity ratio for 180/200 bitumens (±σ).

The relationship between the RTFO-retained penetration and the durability test result (expressed as a percentage increase in modulus) is shown in Figure 3.3. Although, as mentioned above, the RTFO test is a measure of susceptibility to high temperature oxidation, only a weak correlation to the durability test result was observed (r^2=56% for a linear correlation).

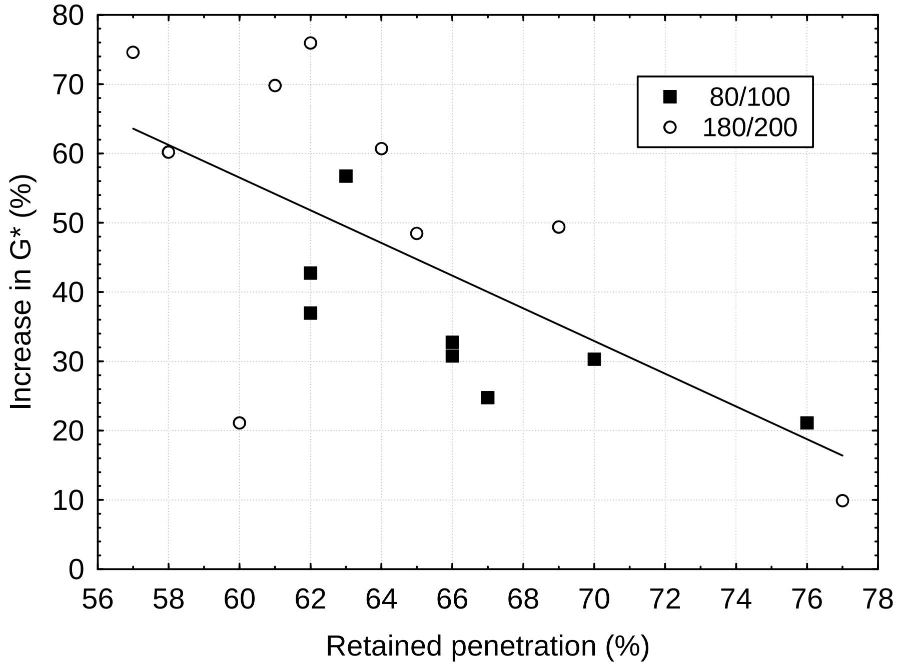

Figure 3.3 Relationship between RTFO-retained penetration and durability test.

4. Conclusions

4.1 Failure modulus

Based on an analysis of very low-trafficked seals in Lower Hutt, the modulus of Safaniya 180/200 bitumen in seals at failure caused by oxidation (80th percentile seal life), was about 100 ± 50 MPa. The assumption has been made that sealing practices (bitumen application rates, chipping rates, etc.) and methods used to assess seal condition in the sites studied are typical of those used throughout the country. The failure modulus value was derived for 180/200 bitumens but should also apply to other grades even though the time taken to reach the failure modulus value will be shorter. To determine the failure modulus, the moduli of bitumen specimens were measured at 5°C, close to that of the average daily minimum winter temperature for the sites studied. Ideally bitumen moduli in the test (both trial bitumen and Safaniya) should be measured at a temperature related to the winter temperature in the areas where the bitumen is to be used. However it appears from the data reported here that the error introduced by adopting a single test measurement temperature of 5°C will be negligible compared to the effect of microclimate and construction variables. In fact one of the most striking observations from the field data is the large range of moduli obtained for seals in a very small geographic area that can be considered to have a uniform climate.

Based on the field site results three approaches are outlined for modifying the durability test (Section 2.3.3) that can form the basis for further discussions with industry.

4.2 Characterisation based on TNZ M/1 test

The retained penetration and calculated viscosity ratio (70°C/135°C) do not uniquely characterise a bitumen because of the error inherent in the measurements. Also the specification limits in TNZ M/1 already necessarily compress the value of the properties to a narrow range. However these parameters still provide a useful indicative means of confirming the identity of bitumen being shipped to New Zealand by comparison with data from the sample used for earlier approval testing. As both parameters are ratios, this method of checking identity would not be affected by variations within a grade but only by crude source or production route.

5. Recommendations

- **The current test used to assess the durability of bitumens in New Zealand should be modified following one of the approaches outlined in Section 2.3.3. Option 3 is preferred, which sets limits of 86 or 61 MPa for 80/100 or 180/200 grades respectively after a 30-day test period at 60°C and 2069 kPa.**

  Further work should be carried out to improve the estimate of test limits for 180/200 and 80/100 bitumens and to enable test durations for 60/70 and 130/150 grades to be determined. In the interim the test durations for 180/200 and 80/100 should be used for 130/150 and 60/70 grades respectively. The question of how to handle the durability requirements of multigrade bitumens or other novel products (for which no Safaniya equivalents exist) needs to be considered.

- **The durability test should be included in the TNZ M/1 specification with a 'report' requirement.**

  Approval of a bitumen (by Transit NZ) should be on the basis of a satisfactory durability test result on a pre-shipment sample, as is the present case but it is usually impractical to test the durability of the actual shipped bitumen before it arrives in New Zealand. For this reason the shipment should be tested after arrival (a condition of the initial Transit NZ bitumen approval) and, depending on the result Transit NZ would take action as appropriate (and as agreed beforehand with the supplier). It is envisioned that for regular suppliers, the supplier and Transit NZ may develop a quality assurance plan that would not require testing of every shipment.

- **For imported bitumens the viscosity ratio (70°C/135°C) and RTFO-retained penetration should be used as an approximate check that the material to be shipped is the same as the sample used to gain approval.**

  The multi-laboratory precision values given in Section 3.2 should be used as a basis for determining an acceptable range of results. Further work should be carried out to determine if making multiple measurements of each parameter would make a significant improvement to the precision.

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Appendix 1 Current durability test method

A1.1 Apparatus and materials

- Cylinder of industrial grade compressed dry air, fittings and hose for connection to pressure vessel.
- Pressure vessel system comprising:
  - pressure vessel certified for operation at 300 psi and 60°C;
  - thermocouple, thermistor or platinum resistance thermometer and temperature-logging device accurate to ± 0.1°C to measure the temperature inside the vessel;
  - pressure release valve that prevents the pressure inside the vessel exceeding the maximum design pressure;
  - pressure regulator capable of controlling the pressure inside the vessel at 300 ±5 psi;
  - pressure gauge readable to 5 psi and calibrated at 300 psi (2.07 kPa);
  - slow release bleed valve to allow de-pressurisation of the vessel from the test pressure to atmospheric pressure over 15 minutes.
- Stainless steel or aluminium sample holders capable of holding at least 1.0 g of bitumen as a uniform horizontal film 1.0 mm thick.
- A metal rack or holder capable of supporting at least 10 sample holders in a horizontal position so that the bitumen film thickness within the sample holders remains uniform.
- A stirred temperature-controlled fluid bath or forced draft oven capable of maintaining the temperature inside the pressure vessel at 60.0 ± 0.1°C. If a water bath is used it must be fitted with a water inlet and level control device so as to automatically a maintain a constant water level over long periods. The bath or oven must be sufficiently large to allow air or the bath fluid to freely circulate around the vessel and contain a shelf or stand to allow the vessel to held in a horizontal (level) position. It is recommended that the position of the vessel relative to the sides of the bath or oven is kept fixed.
- Dynamic shear rheometer or similar instrument capable of applying sinusoidal loading and measuring the dynamic shear modulus of bitumen at 9.0Hz and 5°C.
- Hotplate.
- Balance readable and accurate to ± 0.001 g.

A1.2 Procedure

- **Calibration of pressure vessel temperature**
  - Place the pressure vessel in the 60°C water bath or oven to be used in the test and pressurise to 300 ± 5 psi. The thermocouple should be positioned near the vertical axis of the vessel and at approximately half height. After 4 hours log the temperature inside the vessel at 15 minute intervals for at least 24 hours and adjust if necessary so that a temperature of 60.0 ± 0.1°C is maintained.
- **Density**
  - Measure the density of the test bitumen at 25°C according to the method given in ASTM D70 and calculate the density at 60.0°C using ASTM D 4311.
- **Preparation of sample films**
  - Weigh a sufficient quantity of bitumen onto the centre of the sample holder to achieve a 1.0 ± 0.5% mm film at 60°C (typically about 1.22-1.23 g). Heat the sample holder on a hot plate at 100-120°C for 3-4 minutes to achieve an even film. Allow to cool to room temperature on a level surface.
- **Oxidation of the bitumen films**
  - Before the test is begun ensure that the pressure vessel (containing the sample holder rack) has been maintained at 60.0° for at least four hours. Remove the pressure vessel from the bath or oven and place the sample holders in the vessel. Return to the bath and pressurise the vessel to 300 ± 5 psi. The sample loading operation must be completed within 10 minutes and preferably as rapidly as possible to avoid cooling of the vessel. After 15-20 minutes check the pressure in the vessel and readjust if necessary.
- **Removing the bitumen films from the pressure vessel**
  - After 80 hours (± 15 minutes) allow the pressure vessel to de-pressurise slowly over about 15 minutes (to avoid excessive bubbling of the samples), and before removing the vessel from the bath. Remove a sample holder and scrape the oxidised film into a small airtight vial and store in a freezer.
- **Modulus measurement**
  - Exact analysis details will vary according to the type of rheometer used but analysis must be consistent from sample to sample.
  - The modulus measurement is carried out using an 8-mm parallel plate geometry with a 1.0 mm gap. Larger diameter plates can be used if it can be demonstrated that the compliance of the instrument is not significantly affecting the measured modulus. A preliminary stress sweep may be necessary to ensure that the strain used lies within the linear viscoelastic region.
  - Samples are annealed in a vacuum oven (at <1 psi) at 100°C for 30 minutes. A sub-sample is placed on the base plate and allowed to cool to about 60°C. The sample is compressed to the 1.0 mm gap and trimmed. To ensure proper wetting of the plates, the sample should not be compressed if it is below 45°C. The sample is brought to the test temperature of 5.0 ± 0.1°C and the modulus measured at 9 Hz.

Appendix 2 Seal coring and bitumen recovery

None of the sites cored were flushed. Most exhibited good texture usually with extensive lichen growth (see Figures 2.2 - 2.4). Cores (150 mm diameter) were taken from the approximate outside wheel path although in many case obvious wheel paths were not apparent. Clean water (without additives) was used as the coring lubricant. Some sites had multiple cores removed across the width of the road.

The sample was warmed at 60°C for 0.5 - 1 hour until soft enough to easily dislodge chip using a screwdriver. Sufficient chips (10 - 20) to recover about 0.2 - 0.5 g of bitumen were removed and placed in a beaker to cool. About 75 ml of dichloromethane (A.R. grade) was added and left with occasional stirring for 1 hour, covered and in the dark.

The solution was passed through a 150 micron sieve and the aggregate washed with 2 x 10 ml fresh dichloromethane. After centrifugation for 20 minutes at 2000 rpm (939 g) the bitumen was filtered under vacuum (water pump) through Whatman grade 1 and GFC filters (grade 1 paper on the bottom).

Approximately equal portions of the solution were poured onto polished 245 x 340 mm stainless steel plates. Stainless steel was used instead of glass to reduce the possibility of selective surface adsorption of polar species. A wide bladed spatula was used to spread the solution, allowing the solvent to evaporate and leave a thin film of bitumen.

The plates were immediately placed in an airtight cabinet (approximately 60 L) in the dark and purged with nitrogen at >10 L min^{-1} for ten minutes, after which time the purge rate was decreased to about 1 - 2 L min^{-1}. After 50 minutes the plates were removed and the bitumen was scraped off with a single-sided razor blade.

The last traces of solvent were removed by heating the combined bitumen scrapings (about 3 g) at 100°C for 30 minutes under >29.9″ Hg vacuum. The samples were stored in a freezer at −18°C.

Shear moduli measurements were made using a 8 mm parallel plate geometry with a 1.0 mm gap on a Carrimed Instruments CSL 500 rheometer fitted with a water bath for temperature control of the specimen. Moduli were measured at a strain of 0.4 - 0.5%, within the linear viscoelastic range. Specimens were heated to 120°C in an oven for 10.0 minutes on the bottom plate, allowed to cool to room temperature (5.0 minutes) and heated to 60°C (5.0 minutes) for sample compression to 1.030 mm. The specimens were trimmed with a heated tool before final compression to 1.000 mm.

To determine whether the procedure significantly affected the viscosity of the recovered bitumens, samples of 180/200 bitumen were 'extracted' using the procedure and the initial and post-extraction moduli compared (see Table A2.1). Further data on the extraction procedure (in this case modified slightly for asphalt mix) have been reported previously (Herrington 2005) and are shown in Table A2.2. In both cases the bitumen used was manufactured from Middle Eastern crudes at the Marsden Point refinery.

The extraction process has had only a small effect on the bitumen properties, the mean moduli and viscosities after extraction are not significantly different from the original bitumen at the 95% confidence level. The phase angle results however show a larger variation and are significantly different at the 95% confidence level, but not at the 98% level.

Table A2.1 Effect of extraction procedure on shear modulus (5.0°C, 9.0 Hz).

	Modulus (MPa)		**Phase angle (°)**	
Run	**180/200 Bitumen**	**180/200 Bitumen after extraction**	**180/200 Bitumen**	**180/200 Bitumen after extraction**
1	21.62	18.61	44.14	46.67
2	25.75	21.24	41.71	45.46
3	26.73	21.86	41.43	45.31
Mean (± SD)	**24.70 ± 2.7**	**20.57 ± 1.7**	**42.43 ± 1.5**	**45.81 ± 0.7**

Table A2.2 Effect of extraction procedure on viscosity (60°C, 0.1 s^{-1}).

	Viscosity (Pas)	
Run	**80/100 bitumen**	**80/100 bitumen after extraction**
1	444	427
2	376	400
3	410	320
Mean (± std dev)	**397 ± 34**	**395 ± 55**

Appendix 3 Field site bitumen moduli

Data for individual cores used in the study. Average values (marked with an *) are given for cores that had repeat moduli measurements made.

Sample No.	Site	Age (years)	Chip grade	Core No.	G* (5°C,9Hz)	Phase angle (°)
6/04/384	Kennedy Gr.	14	4	1	131.45	18.26*
6/05/190	Kennedy Gr.	14	4	2	77.74	25.88
6/05/191	Kennedy Gr.	14	4	3	41.09	30.37
6/04/385	Cleary St	18	3	1	125.15	19.40*
6/05/194	Cleary St	18	3	2	130.10	18.39
6/05/195	Cleary St	18	3	3	118.40	19.31
6/04/386	Freyberg St	13	4	1	69.41	27.18
6/04/387	Kopara Gr.	12	4	1	77.48	26.59
6/05/200	Kopara Gr.	12	4	2	75.52	26.60
6/05/201	Kopara Gr.	12	4	3	71.03	27.49
6/04/388	Kamahi St	14	4	1	61.93	27.70
6/05/198	Kamahi St	14	4	2	131.60	18.64
6/05/199	Kamahi St	14	4	3	133.70	19.59
6/04/389	Atiawa Cres.	10	5	1	72.51	27.46
6/04/390	Feist St	12	4	1	79.57	25.46
6/04/391	Barton Gr.	11	4	1	56.76	29.69
6/04/392	Ingram St	13	4	1	62.13	28.44
6/04/393	Aldersgate Gr.	14	4	1	76.31	25.17
6/04/394	Hart Ave	12	4	1	75.09	25.47
6/04/395	Anson Gr.	14	4	1	105.10	21.05
6/05/203	Anson Gr.	14	4	2	86.36	24.28
6/04/396	Fenchurch Gr.	14	4	1	74.19	26.17
6/04/397	Barraud St	15	4	1	128.60	20.15
6/05/196	Barraud St	15	4	2	115.60	19.88
6/05/197	Barraud St	15	4	3	125.70	18.60
6/04/398	Harley Gr.	13	4	1	89.65	22.86
6/04/399	Barberry Gr.	14	4	1	76.97	25.10
6/04/400	Ariki St	14	6	1	67.28	27.36
6/04/401	Banksia Gr.	12	4	1	80.13	24.38

Sample No.	Site	Age (years)	Chip grade	Core No.	G* (5°C,9Hz)	Phase angle (°)
6/04/403	Cherry Blossom Gr.	12	4	1	60.23	28.76
6/04/404	Cornish St	12	3	1	42.73	35.86
6/04/405	Scholefield St	29	3	1	51.98	29.27
6/04/406	Picasso Gr.	26	5	1	63.24	25.45
6/04/407	Fairfield Ave	10	4	1	74.34	26.57
6/04/408	Eastview Gr.	11	4	1	53.55	30.22
6/04/410	Dublin St	13	4	1	58.13	27.92
6/04/411	Hinau St	14	4	1	75.54	26.28
6/04/412	Maire St	26	3	1	57.54	28.99*
6/05/30	Maire St	26	3	2	54.87	29.23
6/05/202	Maire St	26	3	3	61.48	26.62
6/04/413	Hay St	17	5	1	75.61	24.98
6/04/414	Belgrave St	10	5	1	72.27	26.97
6/05/26	Puketapu Gr. Nth	18	5	1	75.00	26.69
6/05/27	Pilmuir St	21	3	1	133.70	18.35*
6/05/28	Ngaturi Gr.	27	3	1	135.70	17.61
6/05/188	Ngaturi Gr.	27	3	2	102.60	23.57
6/05/189	Ngaturi Gr.	27	3	3	89.30	24.81
6/05/29	Newburn Gr.	23	3	1	119.50	21.21*
6/05/31	Riddiford St	19	4	1	150.50	18.05
6/05/32	St Columbans Gr.	18	5	1	126.10	21.52
6/05/192	St Columbans Gr.	18	5	2	141.60	17.18
6/05/193	St Columbans Gr.	18	5	3	181.50	17.74
6/05/33	Peach Tree Gr.	19	5	1	103.00	21.84
6/05/34	Sunshine Cr.	18	5	1	117.30	21.07
6/05/179	Waipounamu Dr.	2	4	1	40.47	36.03
6/05/180	Cottle Park Dr.	4	2	1	48.59	32.40
6/05/181	Tawa St	4	4	1	69.13	27.77
6/05/182	Mawson St	4	4	1	52.70	31.22
6/05/183	Molesworth St	6	4	1	52.81	31.57
6/05/184	Meremere St	6	4	1	42.56	34.59

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