Durability specification limit for asphalt bitumen grades

July 2010

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NZ Transport Agency research report 412

ISBN 978-0-478-36459-0 (print)
ISBN 978-0-478-36458-3 (electronic)
ISSN 1173-3756 (print)
ISSN 1173-3764 (electronic)

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Herrington, PR and GR Bentley (2010) Durability specification limit for asphalt bitumen grades. *NZ Transport Agency research report no.412*. 18pp.

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Keywords: asphalt, bitumen, durability

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Acknowledgements

The author would like to thank Mr John Vercoe of EDI Works Infrastructure Ltd and Ms Rachel Smith of Fulton Hogan Ltd for their help in this project.

Abbreviations and acronyms

OGPA	Open-graded porous asphalt
RTFO	Rolling thin film oven

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Executive summary

The NZ Transport Agency specification TNZ M/1 (2007) for bitumen specifies a durability test that is used as part of the bitumen approval process to screen for bitumens that might be prone to oxidation and rapid hardening in the field. The specification sets a value of 100MPa for the modulus at 5°C and 9Hz of bitumen after treatment in the test. This specification value was determined for 180–200 bitumen used in chip seals. As the overall oxidation rates of bitumens in asphalt mixes is likely to be affected by factors not present in relation to chip seal bitumens, the relevance of the 100MPa specification limit to asphalt bitumen grades has been questioned. Recently a suggestion was made to increase the limit to 120MPa for 60–70 grade bitumen and to 130MPa for 40–50 grade. This research was undertaken to determine the relationship of the moduli of aged bitumen in asphalts to the 100MPa limit. Determining the relationships between the numerous variables affecting asphalt oxidation rates in the field was beyond the scope of the present work, instead the approach taken was to obtain data on a random sampling of dense asphalt surfacings of varying ages and from varying locations around the country. The effects of variables such as percentage air voids, initial bitumen modulus, bitumen source and plant temperatures are thus included in the data, but cannot be isolated explicitly.

Field samples of dense mix manufactured using 60–70 or 80–100 penetration grade bitumen were cored from sites in Christchurch, Wellington, Tauranga and Auckland. Samples of 40–50 asphalt were not collected as there are no known sites of any significant age where 40–50 bitumen has been used in an asphalt surfacing. Bitumen was recovered from the core samples using a solvent (dichloromethane) extraction method. The method was shown to have no significant effect on the measured modulus values by extraction of standard 40–50 and 80–100 bitumens. The modulus of the recovered bitumen was measured at 5°C and 9Hz by dynamic shear rheometer in accordance with the durability test procedure.

The moduli of bitumens extracted from the field cores showed a wide scatter at a given field age. The moduli of the 60-70 samples were not significantly higher than those of the 80–100 at the equivalent age indicating the importance of variables other than the initial modulus in governing average oxidation rate. Various factors can act to disguise differences in the initial moduli of the 60–70 and 80–100 bitumens. Plant conditions, for example, were found to produce differences in moduli of up to 163% for 80–100 asphalt samples with the same mix design, bitumen and from the same plant when taken over three consecutive days.

Conclusion

The results show that setting the specification limit for 60–70 bitumen at 120MPa as proposed would be reasonable. No field data for 40–50 bitumen was available as use of this grade as an asphalt surfacing is currently very limited. The proposed value of 130MPa is based on durability test values found for 40–50 bitumen produced from Middle Eastern crudes at the NZRC Marsden Point refinery (currently the only suppliers of 40–50 in New Zealand). This value is close to the approximate upper bound of the 60–70 and 80–100 data and until information can be obtained, is an appropriate specification limit.

This research has highlighted serious limitations in the current durability test procedure (developed for chip seal binders) when applied to asphalt grade bitumens. Therefore review and modification of the procedure is required.

Abstract

The object of this study was to gather data on the levels of bitumen oxidation found in dense asphalt surfacings. The information was used to determine if the proposal to raise the 100MPa modulus (at 5°C, 9Hz) limit set in the NZTA bitumen specification (NZTA M/1:2007) to 120MPa and 130MPa for 60–70 and 40–50 bitumen grades respectively, was appropriate. Samples of dense asphalt manufactured with 60–70 or 80–100 grade bitumen (excluding polymer modified bitumens) were sampled from field sites of varying ages up to 40 years. The oxidised bitumen was extracted and the modulus measured. The research highlighted serious limitations in the current durability test procedure (originally developed for chip seal binders) when applied to asphalt grade bitumens and review and modification of the procedure is required. However, based on the results obtained, setting the durability test limits in the NZTA M/1 bitumen specification to 120MPa for 60–70 and 130MPa for 40–50 grades, as recently proposed is considered reasonable until further information is available.

1 Introduction

1.1 Background

The New Zealand transport agency specification NZTA M/1(2007) for bitumen specifies a durability test that is used as part of the bitumen approval process to screen for bitumens that might be prone to oxidation and rapid hardening in the field (the test also acts as a de facto control on low temperature properties). The specification sets a value of 100MPa for the modulus at 5°C and 9Hz of bitumen after treatment in the test.

The test procedure (NZTA 2008) involves oxidising a 1mm film of bitumen at 60°C for 80 hours under 300psi of air. Comparison with field data indicated that the increase in modulus from this treatment was approximately equivalent to 10 years of field ageing of Safaniya 180–200 bitumen in chip seals, but that the range of moduli values measured was very wide (Herrington et al 2006). The approximate upper bound of this data at 10 years age (100 MPa), was set as the durability test limit in the M/1 specification and for want of other data was applied to all bitumen grades.

As the overall oxidation rates of bitumen in asphalt mixes is likely to be affected by factors not present in relation to chip seal bitumens, the relevance of the 100MPa specification limit to asphalt bitumen grades has been questioned. In particular, factors such as the high temperatures and thin films present during asphalt manufacture and the air voids in compacted asphalt that allow ingress of oxygen may give rise to greater average oxidation rates than in chip seal bitumens. Of course the asphalt grades have higher moduli to begin with so will reach the limit in a shorter time even if the oxidation rate in the test is the same as Safaniya 180–200 bitumen. Recently, at a NZTA Bitumen Industry Group meeting, a proposal was made to increase the limit to 120MPa for 60–70 grade bitumen and to 130MPa for 40–50 grade.

The object of the present study was to gather data on the levels of oxidation found in asphalt surfacings and to use this information to determine if the 100MPa modulus level was appropriate for 60–70 and 40–50 bitumen grades.

1.2 Research methodology

The major factors affecting the average rate of oxidative hardening (as measured by the modulus) of bitumen in asphalt mix are:

1. bitumen chemistry (crude oil source and refinery production route)
2. bitumen grade
3. manufacturing process (temperature, time and film thickness)
4. compaction level (ie the percentage of air voids present as a function of time)
5. ambient temperature in the field. Levels of UV radiation are generally considered of minor importance as penetration of light beyond the first few microns into the bitumen surface is negligible (Dickinson et al 1958). This however assumes that diffusion of oxidised surface species deeper into the film can also be ignored
6. aggregate type.

Attempting to obtain sufficient samples to statistically assess the effect of all these variables would be prohibitively expensive. In any case much of the information necessary (eg bitumen source, plant temperatures, initial air voids) is either not known or is not accessible. Instead the approach taken was to obtain data from a random sampling of dense asphalt surfacings of varying ages and from varying locations around the country. The effects of the variables listed above are thus included in the data, but cannot be isolated explicitly. The surfacings selected were generally in good condition, no attempt was made to deliberately select or exclude surfaces that were showing signs of failure.

Note that base mixes, used as part of the pavement structure and not directly exposed to the atmosphere, were not included in the study. These materials are not widely used in New Zealand and their rate of oxidation is likely to be negligible compared with surfacing mixes, due to the long diffusion path for oxygen.

2 Methodology

2.1 Sampling programme

Samples of dense mix were obtained from Christchurch, Wellington, Tauranga and Auckland. These locations were considered representative of climatic conditions through the country (at least areas where significant quantities of asphalt mix are used). Samples of asphalt manufactured using both 60–70 and 80–100 bitumen grades although because of differences in practice 60–70 samples were only available from Auckland. There are no known sites of any significant age where 40–50 bitumen has been used in an asphalt surfacing.

Asphalts employing polymer modified bitumens were not included in the study. The main reasons for this were:

- the primary intention was to determine a suitable durability test limit in the M/1 specification for penetration grade bitumens
- recovering polymer modified bitumen from field cores without potentially affecting the microstructure and polymer distribution within the bitumen is presently impossible.

As relatively few open-graded porous asphalt (OGPA) surfacings are used (these tend to be on motorways and are thus extremely expensive to sample) and the majority contain polymer modified binders, OGPAs were also excluded from the study.

Sites were located using council databases. A visual inspection of the road surface was made to ensure that the database information on the age of the site seemed realistic and 150 or 100mm diameter cores taken (in some cases duplicates). The cores were, as far as possible, taken outside the outer wheel path (but not on the shoulder). Where possible the cores were taken from in front of driveways (where cars would not have parked) and away from overhanging trees or other structures likely to shade the surface.

Plant mix samples for the historical field site samples were not available. To provide an estimate of the 'zero time' modulus value to compare with the field data, samples of mix were taken from plants in Wellington, Tauranga and Christchurch over the 2009–2010 season.

2.2 Bitumen extraction and modulus measurement

The top 10mm of the core was removed and used in the analysis. A roughly triangular segment was broken away from the specimen and placed in a beaker. Sufficient dichloromethane (AR grade) to just cover the sample was added and left with occasional stirring for one hour, covered and in the dark.

The solution was decanted into tubes and centrifuged for 20 minutes at 2000rpm (939g). The resulting solution was then filtered under vacuum (water pump) through Whatman grade 1 and GFC filter papers (grade 1 paper on the bottom).

Approximately equal portions of the solution were poured onto polished 245mm x 340mm, 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. After three to four minutes 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 1–3g) at 100°C for 60 minutes under >29.9” Hg vacuum. The samples were stored in a freezer at -18°C.

Shear modulus (G*) measurements were made using a 8mm parallel plate geometry with a 1mm gap on a Carrimed CSL 500 rheometer fitted with a water bath for temperature control of the specimen. Moduli were measured at a strain of 0.4% to 0.5%; within the linear viscoelastic range. Specimens were heated to 120°C in an oven for 10 minutes on the bottom plate, allowed to cool to room temperature (five minutes) and heated to 60°C (five minutes) for sample compression to 1.030mm. The specimens were trimmed with a heated tool before final compression to 1.000mm.

The extraction procedure (with slight differences) has been shown previously to have no significant effect on the physical properties of the extracted bitumen (Herrington 2006; Herrington et al 2007). Further experiments to verify the process were conducted in the present study using harder grade bitumens. Replicate samples of straight 40–50 and 80–100 bitumens (without aggregates present) were put through the extraction process. The results are given in table 2.1.

Table 2.1 Effect of recovery process on bitumen modulus at 5°C and 9Hz

Bitumen grade	Initial modulus (MPa)	Modulus after extraction (MPa)	Initial phase angle (°)	Phase angle after extraction (°)
40–50	77.45	89.47	27.41	25.16
40–50	89.40	77.14	25.02	25.54
40–50	98.98	98.42	24.47	23.83
40–50	86.00	97.10	26.48	24.39
Mean	**87.96**	**94.03**	**25.85**	**24.73**
80–100	45.92	46.89	34.75	34.48
80–100	48.79	46.38	32.80	34.88
80–100	53.67	47.36	32.77	32.22
80–100	53.03	50.23	33.35	32.71
80–100	52.41	44.53	33.23	34.73
Mean	**50.76**	**47.08**	**33.38**	**33.81**

A paired t-test, comparing the means of the differences to zero showed that the means of the moduli or phase angle after extraction were not significantly different from the initial values at the 95% confidence level, ie the extraction process had no effect, confirming earlier findings.

3 Results

The moduli of bitumens extracted from the field cores using the methods described above are shown in figures 3.1 and 3.2. For the reasons discussed above the data shows a wide scatter. Similarly large variations in field data were also observed in aged chip seal binders (Herrington et al 2006).

Extreme outlying data points may reflect problems during construction. This could include overheating during manufacture and poor compaction or for the two 40-year samples with very low moduli, contamination of the surface or an error in the local authority database. The curved lines were fitted by trial and error to encompass 90% of the 129 data points and this data subset was used for comparison to the proposed durability test limits.

In figure 3.1, 60–70 and 80–100 data is differentiated. The moduli of the 60–70 data are not significantly higher than those of the 80–100 at the equivalent age. As the penetration of the two grades is controlled at 25°C the initial moduli of the two grades at 5°C are in general not widely different and manufacturing, differences in air voids and other factors may be disguising any differences due to initial moduli. Only one 60–70 plant sample was obtained and this falls squarely in the range of results found for the 80–100 asphalts. Considerable variation in moduli of the 80–100 plant dense asphalt samples was seen, this data is given in table 3.1. The mix design differed between plants but for a given plant was nominally the same material over the entire sampling period.

Figure 3.1 Moduli according to asphalt bitumen grade

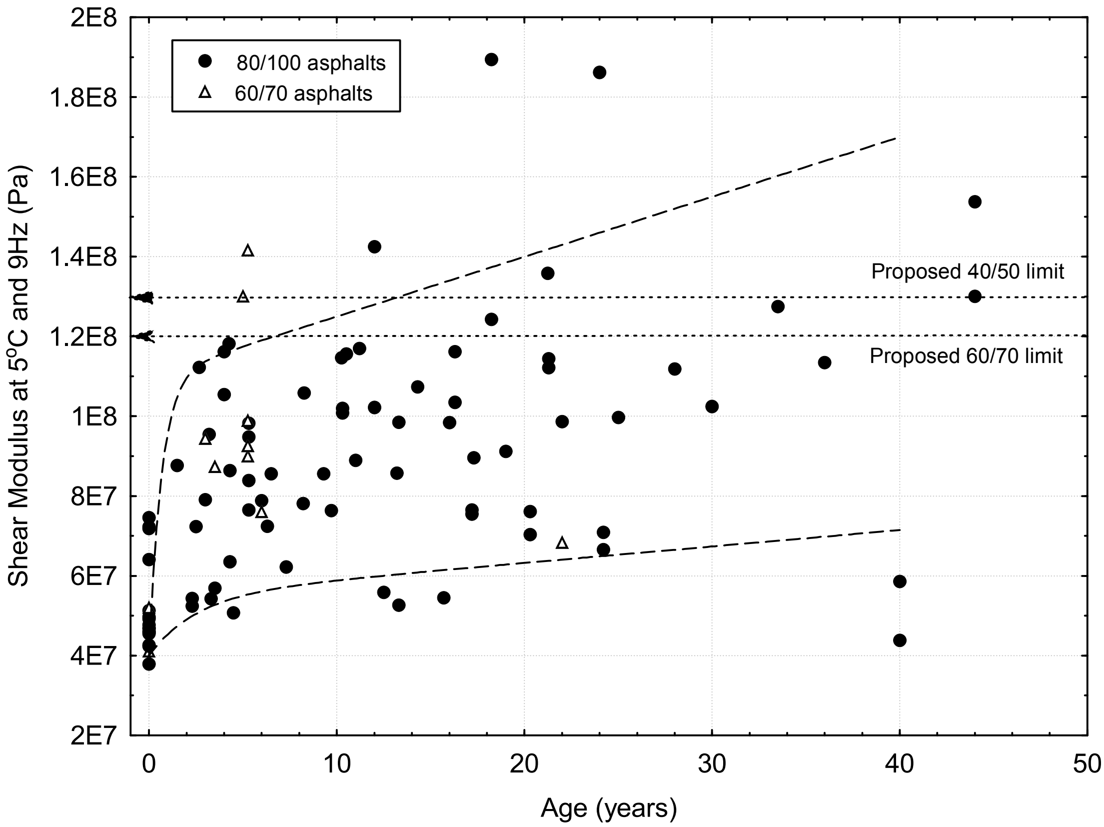
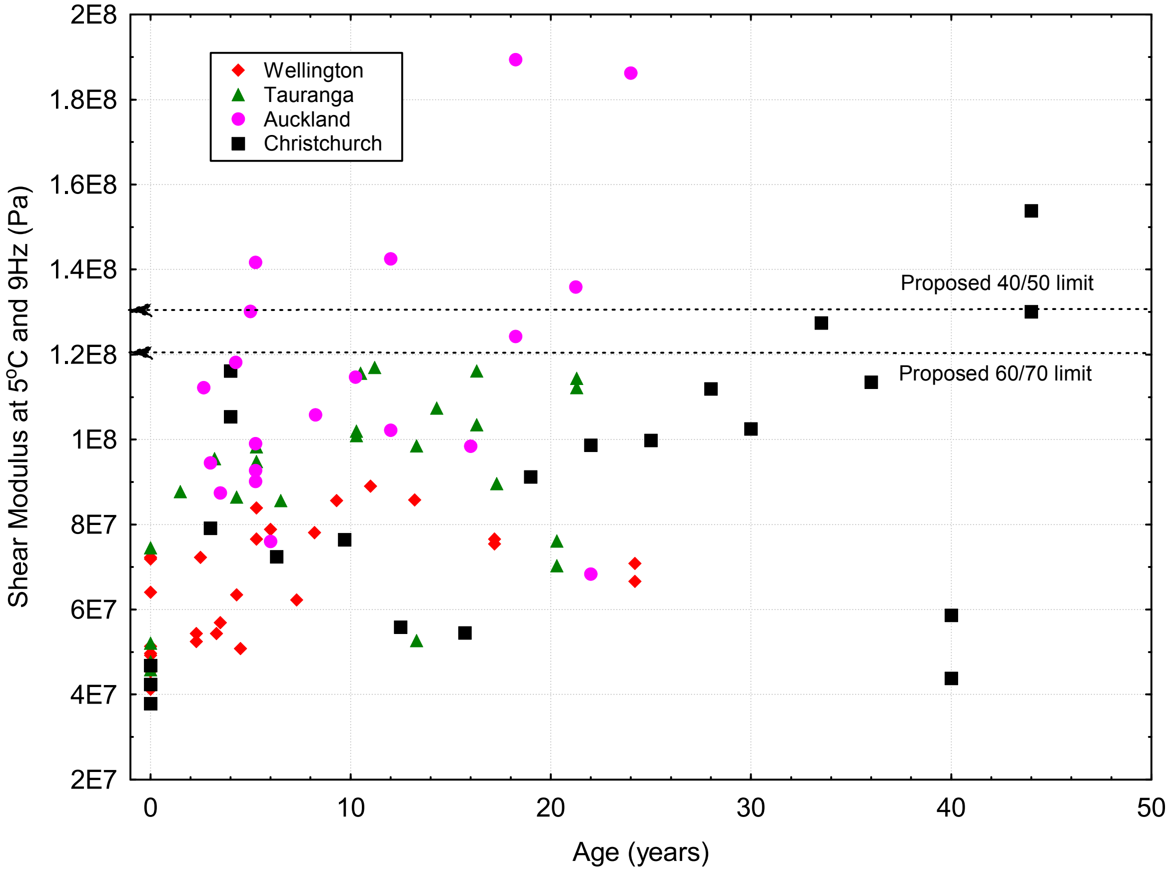


Table 3.1 Variation in moduli at 5°C for 80–100 asphalt plant samples

Plant	Project	Consecutive production day	Modulus (MPa)
A	1	1	41.23
	2	1	46.10
	3	**1**	50.53
	4	**1**	42.66
	5	**1**	49.24
	6	**1**	46.88
	8	**1**	64.10
	7	**1**	45.49
		2	71.88
		3	72.25
B	1	**1**	47.70
		2	45.87
		3	74.59
C	1	**1**	46.81
		2	42.41
		3	41.23

Figure 3.2 shows variations due to sampling location. The locations were chosen to cover a range of cooler (Christchurch) to warmer (Auckland) climates. The Auckland samples do appear to be concentrated at the upper end of the range for a given age compared with those for the other locations but the distinction is not particularly marked. This may reflect a faster oxidation rate due to the warmer climate or differences in bitumen supply source (ie differences in the initial moduli of bitumens within the same grade). Given the lack of pronounced demarcation of the 60-70 and 80-100 asphalts, the former seems more likely. Alternatively the differences may reflect differences in manufacturing and construction practices as only three or four contractors are responsible for nearly all asphalt surfacings in the country. As discussed above, available records have insufficient data to attempt to isolate particular factors.

Figure 3.2 Moduli according to asphalt location



3.1 Limitations of the durability test for asphalt bitumen grades

The current durability test was developed 20 years ago as a means of controlling variations in production or additive modifications to 180-200 Safaniya bitumen used in chip seals. An obvious short-coming with the test procedure when applied to asphalt grade bitumens, lies in the absence of a rolling thin film oven (RTFO) test step to simulate plant oxidation. In two instances samples taken from the same plant producing the same mix over three days showed a variation of up to163 % and there is no reason to believe that this effect is unusual. Such plant variations are likely to have a significant impact on the resulting life of the surfacing, all other variables being equal.

The lack of an RTFO test step is easily rectified but a more significant problem lies in establishing a correlation of test conditions to field data. The conditions in the durability test oxidise Safaniya 180-200 bitumen to a level approximately equivalent to 10 years in the field for the same bitumen in chip seals. This correlation was obtained by comparing the increase in modulus for Safaniya 180-200 bitumen from seals, with samples from the same production batch (or with an approximately equivalent initial modulus) oxidised in the test (Herrington et al 2006). The same correlation will not hold exactly for bitumens made from widely different crude oil sources, production methods or of different grades, and in the case of asphalts will be further complicated by the effect of air voids and manufacturing conditions as discussed in section 1.2 above. As a range of different bitumens have been used in New Zealand since the mid-

1990s but no record of where these different materials have been used, and no samples of the bitumen are available, then establishing test-field age correlations for asphalt grade bitumens is for all practical purposes impossible. In any case such correlations are only really feasible when bitumen supply sources are consistent over a very long period. A consistent bitumen supply source can no longer be assumed (and this situation is unlikely to change) so that revision of the durability test is needed.

Given the limitations of the durability test in relation to asphalt surfacings discussed above and the lack of correlation to field data then determining suitable specification limits for asphalt grades involves significant assumptions. Applying the test-field equivalency of 10 years to asphalt grades is the best approximation that can be made with the data to hand but the conclusions reached below must be confirmed by further research.

Although beyond the scope of the present work it should be noted that another drawback of the procedure is the lack of theoretical relationship between the modulus measurement and actual failure mechanisms. To illustrate this point, as the average life of dense asphalt surfacings in New Zealand is about 16 years (Bartley Consultants 1999) then a typical ‘modulus at failure’ of approximately 100 MPa (at 5˚C and 9Hz) can be estimated from the data in figure 3.2. However the large range of values (about 50 to 140MPa) is such as to limit the practical usefulness of this parameter and suggests that the modulus may not be a good indicator of failure in the field. This criticism also applies to use of the test method for chip seal binders.

3.2 Suggested durability test specification limits

The results show that up to about 10 years only two 60–70 samples exceeded 120Mpa. Setting the limit at this value for the 60–70 grade would thus be reasonable. It could be argued that the 80–100 grade should also be set to the higher level, but as this grade is also used in sealing operations and suppliers have no difficulty in meeting the current specification then raising it is unnecessary. No field data for 40–50 bitumens is available as use of this grade as an asphalt surfacing is currently very limited. The proposed value of 130MPa is based on durability test values found for 40–50 bitumen produced from Middle Eastern crudes at the NZRC Marsden Point refinery (currently the only suppliers in New Zealand). This value is close to the approximate upper bound of 60–70 and 80–100 data in figure 3.1 and until further information can be obtained, is an appropriate specification limit.

A variety of methods are used internationally to estimate bitumen durability unfortunately none of which are directly comparable to the New Zealand method both in terms conditions (time, temperature and pressure) or the bitumen properties measured . The closest is the US performance graded asphalt binder specification ASTM D6373-07e1 (ASTM 2007). This specification requires the value of $G^*/\sin\delta$ to be ≤5 MPa measured at 1.6Hz and temperatures ranging from 4 to 40˚C (the standard grades) for bitumens oxidised at 90 to 110˚C for 24 hours under 300psi air.

The field samples in the present work give $G^*/\sin\delta$ values in the range of about 15 to 36MPa at 5˚C. These are considerably higher than the US PG specification limit but cannot be compared directly as the oxidation time and temperature are significantly different, the US test also involves an RTFO step. For comparison a typical $G^*/\sin\delta$ value for fresh 80–100 bitumen (straight from the tin) is about 16 MPa, and for 180–200 bitumen about 7MPa.

4 Conclusions

This research has highlighted serious limitations in the current durability test procedure (developed for chip seal binders) when applied to asphalt grade bitumens, and review and modification of the procedure is required.

Based on the results presented here setting the durability test limits in the NZTA M/1 bitumen specification to 120MPa for 60-70 and 130MPa for 40-50 grades as recently proposed is considered reasonable until further information is available.

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