

Bitumen performance tests

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Abbreviations and acronyms

AASHTO	American Association of State and Highway Transportation Officials
ASTM	American Society for Testing and Materials
BBS	bitumen bond strength
BRRC	Belgian Road Research Centre
BSI	British Standards Institute
DSR	dynamic shear rheometer
ISO	International Standards Organisation
ISSA	International Slurry Surface Association
MSCR	multiple stress creep recovery (test)
RMS	Roads and maritime Services (Australia)
SBS	styrene-butadiene-styrene
Transport Agency	New Zealand Transport Agency

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Executive summary

This project was undertaken in 2015 to support development of a New Zealand performance-based bitumen specification for bitumens used in chipseals. The NZ Transport Agency (the Transport Agency) is currently working towards the development of a performance-based bitumen specification to ultimately replace the current M/1:2011 bitumen specification

A performance-based specification is one in which the bitumen properties specified are directly related to key performance properties required of road surfacings (such as chip retention), or surfacing failure modes (such as water-induced adhesion failure).

The current project was undertaken to investigate three specific aspects of bitumen performance in chipseals considered important by the Transport Agency as part of the development of a performance-based specification for chipseal binders relevant to New Zealand practice:

- 1 Compatibility with kerosene
- 2 Adhesion to aggregate in the presence of water
- 3 Chip retention- relevance of binder cohesive energy.

Compatibility with kerosene

Kerosene is added to bitumen to temporarily lower the viscosity to assist chip wetting and reorientation of the chip during and in the weeks following construction. In the context of the M/1 specification 'compatibility' means that the addition of kerosene to a bitumen results in consistent changes in viscosity equivalent to that found with a 'standard' bitumen. In practice, compatibility is determined by measuring the effect of increasing kerosene concentration on the viscosity of the binder.

An analysis of data from the last decade showed that differences due to the base (unmodified) viscosity are far greater than those produced by small differences in kerosene compatibility as defined by the slope of the viscosity-kerosene concentration plots. In theory these should be taken into account by the contractor when kerosene quantities are being calculated. The data shows that a kerosene compatibility requirement would not be required in a new performance-based specification. If it was to be retained then the overall effect of slope and base viscosity on the response to kerosene should be included in the assessment.

Adhesion to aggregate

An adequate level of bitumen adhesion to the sealing aggregate and resistance to water-induced stripping is essential for seal performance. A test to assess potential adhesion problems with chipseal binders needs to take into account both chemical affinity, potential degradation of adhesion agents at high temperatures during handling and the physical wetting of the aggregate at ambient temperatures.

At present Vialit plate adhesion tests are carried out independently of the bitumen specification by the contractor to test particular bitumen-chip-adhesion combinations. No better, practical test procedure was identified to warrant replacement of the currently used Vialit test, but suggestions were made for changes to the method to potentially improve its precision.

For a full assessment of adhesion, the entire bitumen-aggregate-adhesion system must be tested which is beyond the scope of a bitumen specification. It is proposed that a new performance-based bitumen specification include screening tests (acid number (Institute of Petroleum 1982) and a 'wetting test' based on the multiple stress creep recovery (MSCR) test carried out at 25°C according to AASHTO T350-14, be included

in the proposed performance-based bitumen specification. Such tests would provide protection against likely poorly performing bitumens and help ensure batch to batch consistency, but with requirements in place so that Vialit tests according to the M/13 approach would be undertaken with new bitumen sources by the contractor, as is currently the case when new chip sources or adhesion agents are used.

Chip retention- relevance of binder cohesive energy

One of the primary functions of the sealing binder is to retain chip under traffic loadings. Chip loss in the present context is considered distinct from chip loss possibly arising from fatigue cracking or potential loss of adhesion to the bitumen in highly oxidised seals. In the field chip loss results from cohesive failure (fracture or yielding), of the bitumen film holding the chip and tends to occur either at very low or very high road temperatures. A performance-based specification should thus include testing of bitumen properties at both low (-10°C to 0°C) and high (50°C to 60°C), temperatures.

The potential for measurement of binder cohesive energy as a binder property that could be used to control chip retention in a new performance-based specification was investigated and methods to measure the property discussed. Cohesive energy is defined as the energy expended to create two new bitumen surfaces. The only standardised method (BS EN 1388-13) for measuring bitumen cohesive energy in relation to chip retention in chipseals is the Vialit pendulum test used in the UK and Europe. A fundamental problem with the test method, however, is that the configuration of the test specimen holder means that materials failing ductility will retard the pendulum for a longer period than brittle samples, even though the film has actually passed the yield point.

It was also determined in general that because the measured cohesive energy is strongly affected by the viscoelastic properties of the binder then cohesive energy alone may not be the most suitable parameter for assessing the ability of a bitumen to resist chip loss. Potentially, at a given temperature a more ductile binder with a higher cohesive energy may still have a lower yield stress than a more brittle material and be more likely to fail under a given traffic stress. At low temperatures an alternative but related approach to measurement of cohesive energy is to measure and control the bitumen yield stress and strain (ie the stress and strain at the point of film rupture), using a simple tensile test method. A minimum yield stress and strain would be specified.

At high temperatures tensile tests on bitumen are impractical and damage to the seal through large non-recoverable deformations of the binder below the yield strain must also be controlled for. The MSCR test (AASHTO T 350-14) could be used for this purpose. A maximum value for the creep compliance and a minimum for the percent recovery would be specified.

Abstract

Aspects of bitumen performance in chipseals related to the development of a New Zealand performance-based specification for chipseal binders were investigated.

Compatibility with kerosene: Differences due to the base (unmodified) viscosity were far greater than those produced by small differences in kerosene compatibility showing that this requirement is probably unnecessary in the new specification.

Adhesion to aggregate: Acid number and a 'wetting test' based on the MSCR test (AASHTO T 350-14) at 25°C were suggested for inclusion in the new specification. Such tests provide protection against likely poorly performing bitumens and help ensure batch to batch consistency. The tests would be carried out in conjunction with Vialit plate tests.

Chip retention: Bitumen cohesive energy as a control property for chip retention was investigated. The measured cohesive energy is strongly affected by the viscoelastic properties of the binder. A tensile test at low temperatures is a better alternative with a minimum yield (rupture), stress and strain specified.

At high temperatures tensile tests are impractical and damage through large non-recoverable deformations of the binder below the yield strain must also be controlled for. Instead the MSCR test with a maximum creep compliance and a minimum percent recovery would be used.

1 Introduction

This project was undertaken in 2014 to support development of a New Zealand performance-based bitumen specification for bitumens used in chipseals. The NZ Transport Agency (the Transport Agency) is currently working towards the development of a performance-based bitumen specification to ultimately replace the current M/1:2011 bitumen specification (NZ Transport Agency 2011), which grades bitumens on the basis of the needle penetration at 25°C.

A performance-based specification is one in which the bitumen properties specified, are directly related to key performance properties required of road surfacings (such as chip retention), or surfacing failure modes (such as water-induced adhesion failure).

Research internationally has focused primarily on bitumen properties specifically related to failure modes in asphalt mixes and in particular permanent deformation (leading to rutting). The US introduced a performance grade system for bitumen in the late 1990s (ASTM 2015), which was subsequently found to have limitations particularly with respect to predicting the behaviour of polymer modified bitumens (D'Angelo 2009; Marasteanu 2007; TRB 2010). Further research has refined the earlier specification and resulted in the development of new test methods, in particular the multiple stress creep recovery (MSCR) test, AASHTO T350-14 (AASHTO 2014). AASHTO (2010) contains a draft performance-graded specification (AASHTO M320-10), based on the MSCR method.

Although there is some overlap, in general failure modes in chipseals are significantly different from those in asphalt mixes. Bitumen properties important for both surfacing types (eg adhesion to aggregate) often have to be measured using different test methods due to differences in the way the surfacings are constructed. For these reasons, although much overseas research on bitumen for use in asphalts is obviously very relevant, it cannot simply be adopted and directly incorporated into a performance-based specification for chipseal binders. Internationally, compared with asphalt surfacings, there has been relatively little work carried out on development of a chipseal bitumen performance-based specification (Bahia et al 2008; Barcena et al 2002; Hanz et al 2012; Hoyt et al 2010; TRB 2013; Vijaykumar et al 2013; Walubita et al 2004).

The current project investigated three specific aspects of bitumen performance considered important by the Transport Agency as part of the development of a performance-based specification for chipseal binders relevant to New Zealand practice:

- 1 Compatibility with kerosene
- 2 Adhesion to aggregate in the presence of water
- 3 Chip retention- relevance of binder cohesive energy.

1.1 Compatibility with kerosene

In the context of the M/1 specification, 'compatibility' means that the addition of kerosene to a bitumen results in consistent changes in viscosity equivalent to that found with a 'standard' bitumen. Ensuring consistent changes in viscosity when kerosene is added to bitumen is important for chipseal spraying operations as significant differences between bitumen sources or batches could result in too much or too little kerosene being used with potential for chip loss in cold weather or severe bleeding in warmer weather. As with adhesion this property is recognised as important in the current specification but no particular methodology is provided for assessing it. Strictly speaking this requirement is an operational one, not an on-road performance property and inclusion of the requirement in the new specification may

not be appropriate. Further, the trend to using more emulsified binders for chipsealing may render control of this property less important in the future.

1.2 Adhesion to aggregate

An adequate level of bitumen adhesion to the sealing aggregate and resistance to water-induced stripping is essential for seal performance. At present adhesion properties are not explicitly specified in the M/1 specification, which gives only a general requirement that bitumen should be 'compatible' with adhesion agents. Methods for assessing compatibility are not provided and in practice this is estimated from a measurement of the acid number, which is really only a very approximate guide to possible adhesion problems (a high acid number increases the probability that the (amine-based), adhesion agent added will react and become inactive).

1.3 Chip retention – cohesive energy

Chip retention under traffic stresses is obviously a major requirement for chipseal binders. Currently in the M/1 specification this property is controlled only indirectly. This report considers the potential for measuring binder cohesive energy as a binder property that could be used to control chip retention. Minimum cohesive energy values are, for example, specified for chipseal binders in the UK (Highways Agency 2015), and in some European countries to control chip loss.

2 Compatibility with kerosene

Kerosene is added (usually at 1–3pph) to bitumen to assist in wetting of the chip and allowing reorientation over the weeks after construction of the seal. Kerosene is no longer defined in the M/1 specification but the product most commonly used is 'Jet A1' or equivalent with a boiling range of 15°C to 300°C.

The amount of kerosene used is usually determined based on the air temperature at the site and operator experience. The amount of kerosene used can have significant effects on seal performance during and immediately after construction and potentially for much longer periods as about 20% of the kerosene added remains in the bitumen for at least the first year (Meydan 1997; Herrington et al 2006).

There is a requirement (clause 5d) in the current NZTA M/1 specification that for a bitumen to be approved for use by the Transport Agency under the terms of the specification, then evidence must be provided that shows the behaviour of the bitumen when cutback with kerosene is not 'atypical'. The intention of this requirement is that the sensitivity (in terms of viscosity change) of all approved bitumens to kerosene should be similar. In theory practitioners can thus apply their standard 'recipes' to any approved bitumen without risk of significant differences in behaviour in the field.

This requirement appears to be unique to the M/1 specification (it was introduced in 2007) and no discussion of it or similar requirements has been found in the literature.

2.1 Current method

No procedure is given in the M/1 specification for meeting the compatibility requirement. In practice the evidence usually used to obtain approval is based on an in-house method developed at Opus Research and which is discussed further below.

The Opus method for kerosene compatibility uses the equation given in the NZTA T/9 specification (NZ Transport Agency 2003) for the calculation of the kerosene content of a cutback bitumen (equation 2.1).

$$K = \frac{1}{(\log \log V + C_k)} (B - (\log \log V + C_d)(D + A) - 100 \log \log V) \quad (\text{Equation 2.1})$$

Where K is the effective kerosene content (pph v/v at 15°C).

D and A are the diesel and adhesion agent content respectively (pph by volume at 15°C).

B , C_k and C_d are constants.

In the case where no diesel or adhesion agent has been added (ie $A = D = 0$) then the effective kerosene content is the actual kerosene content and equation 2.1 can be rearranged to give equation 2.2:

$$\log \log V = \frac{B - C_k K}{(100 + K)} \quad (\text{Equation 2.2})$$

The equation provides the slope of a kerosene content–viscosity (60°C) plot for standard grades of bitumen. Values of B and C_k are given for 180–200, 130–150, 80–100 and 60–70 grades in T/9 (see table 2.1).

Table 2.1 Standard constants for kerosene estimation from NZTA T/9

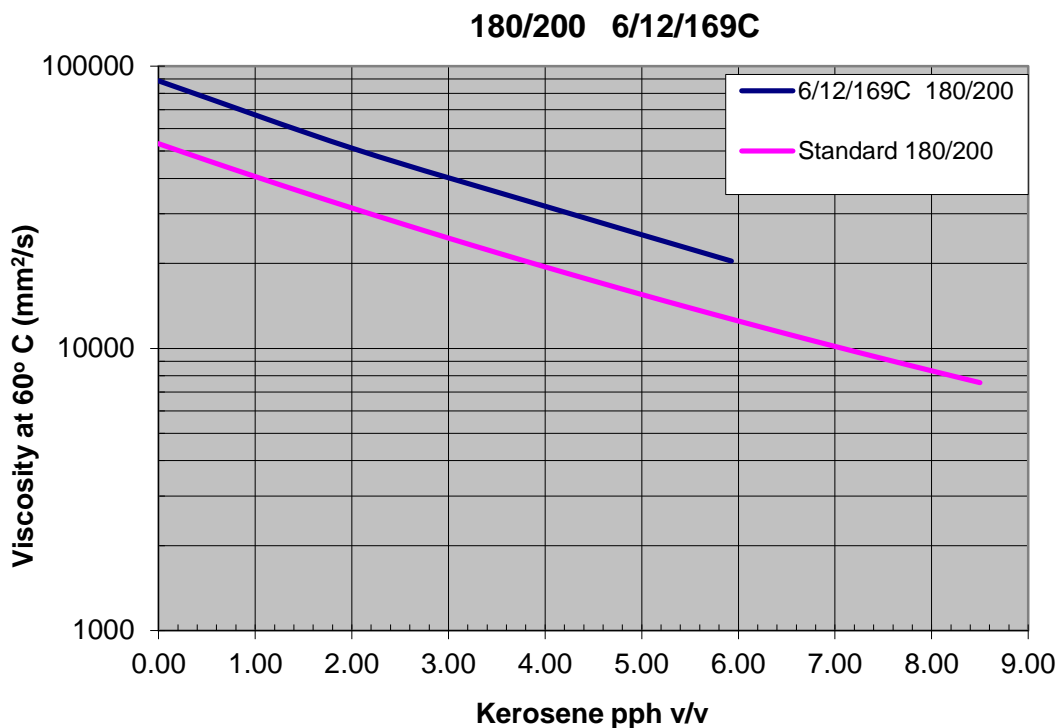
Bitumen grade	B	CK
180–200	67.44	0.42

130-150	69.39	0.42
80-100	72.29	0.42
60-70	74.51	0.42

In the Opus method for assessing kerosene compatibility the kerosene content-viscosity (60°C), plot for the test bitumen is measured (for kerosene concentrations of 0 to 6pph), and compared with that calculated for the appropriate grade of standard material. If the slopes of the curves (C_k), are significantly different then this indicates the test bitumen is more or less sensitive to kerosene (ie the amount the viscosity is lowered is different).

An example of a 180-200 test bitumen and the standard plot for 180-200 bitumen is shown in figure 2.1.

Figure 2.1 Example kerosene- viscosity plot



The offset in the curves is due to the different base viscosity (ie without kerosene added) of the materials. In effect, over the last decade of testing conducted at Opus Research it has been found that differences in behaviour between bitumens with respect to the effect of kerosene on viscosity at 60°C are relatively minor. Table 2.2 shows a sample of data from testing carried out by Opus Research over the last 10 years on bitumens being considered for approval (most of these materials were not subsequently used in New Zealand but the data provides an indication of the extent of variability encountered).

Table 2.2 Kerosene compatibility data comparisons

Bitumen grade and reference no.	C _k	B	Viscosity at 60°C (mm ² s ⁻¹)		Ratio A/B	Ratio(A/B)/(standard bitumen A/B ratio)
			0pph (v/v) kerosene (A)	3pph (v/v) kerosene (B)		
80-100						
Standard (T/9)	0.42	74.51				
6/10/26	0.48	70.85	129,101	52,404	2.5	1.00
6/10/113	0.58	71.29	145,537	54,220	2.7	1.09
6/11/71	0.60	72.57	207,686	73,291	2.8	1.15
6/11/1980	0.44	72.8	221,638	87,677	2.5	1.03
6/11/105	0.44	72.7	215,451	85,477	2.5	1.03
6/12/10	0.36	73.43	265,310	109,638	2.4	0.99
6/12/15	0.58	72.32	193,592	69,892	2.8	1.13
6/12/169A	0.37	72.88	226,725	94,414	2.4	0.98
6/12/257	0.50	73.35	259,282	96,370	2.7	1.10
13/06/316	0.42	71.4	149,992	62,663	2.4	0.98
13/06/427	0.45	73.31	256,324	99,131	2.6	1.05
13/06/448	0.57	71.26	144,348	54,220	2.7	1.08
130-150						
Standard (T/9)	0.42	69.37				
6/12/169B	0.38	70.78	126,678	55,423	2.3	1.01
6/0/00	0.43	69.56	91,485	39,867	2.3	1.01
180-200						
Standard (T/9)	0.42	67.44				
6/10/01	0.51	68.01	61,292	26,312	2.3	1.08
6/10/177	0.54	67.8	58,118	24,581	2.4	1.09
6/11/08	0.51	67.00	47,572	20,964	2.3	1.05
6/11/100	0.58	68.37	67,182	27,227	2.5	1.14
6/12/153	0.49	68.12	63,030	27,351	2.3	1.07
6/12/166	0.55	67.72	56,956	23,978	2.4	1.10
6/12/169C	0.37	69.42	88,183	40,246	2.2	1.01
6/12/170	0.53	68.36	67,010	28,112	2.4	1.10
13/06/230	0.50	67.55	54,571	23,871	2.3	1.06
13/06/294	0.40	68.18	64,000	29,501	2.2	1.00
13/06/409	0.61	66.8	45,275	18,769	2.4	1.12
14/06/210	0.50	67.44	53,086	23,287	2.3	1.06
14/06/226	0.51	67.00	47,572	20,964	2.3	1.05
14/06/290	0.51	67.86	59,006	25,430	2.3	1.07
14/06/300	0.57	67.79	57,971	24,033	2.4	1.12

The 'ratio (A/B)/(standard bitumen A/B ratio)' column in table 2.2 is a measure of the relative sensitivity of the bitumen to kerosene. In most cases the ratio is very close to one. When the data showing the largest difference (sample no. 6/11/71) from standard behaviour is examined the effect in practice is relatively small. The bitumen in question would require 2.4pph kerosene to achieve the same reduction in 60°C viscosity as 3pph in the standard bitumen. This is well within the precision of the plots as stated in T/9 (± 2 pph).

2.2 Discussion

In practice effects on cutback viscosity due to differences in the base viscosity are far greater than those produced by small differences in kerosene compatibility as defined by the slope of the viscosity-kerosene concentration plots. Anecdotally at least there is no evidence that these differences have had any significant effects (in theory these should be taken into account by the contractor when kerosene quantities are being calculated). The need for the kerosene compatibility requirement in a new performance-based specification is thus questionable but if it is retained then the overall effect of slope and base viscosity on the response to kerosene should be included in the assessment.

3 Adhesion to aggregate

3.1 Adhesion processes

Adhesion in the present context refers to the ability of the bitumen to wet the surface of the aggregate and form a bond that is resistant to the action of water. Water will act to displace the bitumen from the aggregate surface and for this reason (and New Zealand's pluvial climate) adhesion agents are routinely added at 0.5 to 1.0% concentrations. Adhesion agents or anti-stripping agents are surface active compounds (usually C18 fatty diamine or fatty amido-amines) that bond to the aggregate surface to form a hydrophobic layer that the bitumen has a greater chemical affinity to. They allow the bitumen to wet even damp aggregate surfaces.

Anecdotal evidence suggests that without adhesion agents the formation of a water resistant bond to a dry aggregate surface will still occur but only over a period of days or weeks of dry weather. This may be due to naturally occurring surface active agents in the bitumen slowly bonding to the aggregate surface or may simply indicate gradual wetting of the chip micro-texture. The latter hypothesis is also supported by the observation that adhesion agents are not routinely needed in asphalt mixes as efficient wetting is achieved at the high temperatures and shear conditions used during manufacture.

The thermodynamic work of adhesion associated with the formation or breaking of an adhesive interface between bitumen and an aggregate in water, is governed by the surface energies of the bitumen-water, aggregate-water and bitumen-aggregate interfaces, as shown in equation 3.1.

$$W_a = \gamma_{aw} + \gamma_{bw} - \gamma_{ab} \quad (\text{Equation 3.1})$$

Where W_a is the work of adhesion and γ_{aw} , γ_{bw} and γ_{ab} are the surface energies of the aggregate-water, bitumen-water and aggregate-bitumen interfaces respectively.

The work of adhesion between the bitumen and aggregate in the dry is generally positive (ie energy must be expended to break the bond). However equation 3.1 applies only to purely elastic materials; in practice bitumen is viscoelastic and additional energy must be expended, above that predicted from surface energy considerations alone, in displacing the bitumen film. In the presence of water the thermodynamic work of adhesion is usually negative and separation is favoured (Grenfell et al 2014; Hamedi and Nejad 2015). Once water gains access to the bitumen-aggregate interface separation will be spontaneous and no energy must be expended.

In practice the situation is more complex as for water to gain access to the interface the bitumen film may need to be displaced from pores or voids in the chip micro-texture which would require extra energy. Additionally the migration of polar chemical species in the bitumen to the aggregate surface may effectively alter the surface energy of the bitumen making disbonding by water less energetically favourable.

Similarly, although equation 3.1 indicates that bond formation between bitumen and aggregates is thermodynamically favoured it gives no indication of the rate of bond formation (wetting), which is dependent on the aggregate surface texture and the physical properties of the bitumen.

Binders that are highly viscous at on-road temperatures or highly elastic (as in the case of styrene-butadiene-styrene (SBS) modified bitumens), will not effectively wet the aggregate within the necessary timeframe (hours to days) after first contact.

High viscosity or highly elastic materials will only wet a relatively small area of the chip which means water is statistically more likely to completely disbond the chip. Partial wetting also means that the applied

traffic loads result in much higher than normal stresses in the bitumen film which can cause it to fail cohesively (failure in the bitumen not at the bitumen chip surface).

An added complication is that adhesion agents, depending on their chemistry and that of the bitumen, tend to lose activity after prolonged storage at temperatures used in bitumen handling and storage (>130°C). Seals can of course be constructed without adhesion agents but the risk of stripping due to rain is very high in New Zealand and in practice they are almost always added to cutback seals. Their properties and effectiveness must also be taken into account when adhesion is being evaluated. Bitumen emulsions in New Zealand are almost all made using cationic emulsifiers which although used at lower concentrations than adhesion agents in cutbacks, are generally similar chemically and are believed to act in the same manner (ie are absorbed onto the aggregate surface as the emulsion breaks); however, there is no published evidence for this. Bitumen emulsions due to their low viscosity and the water present tend to wet aggregate far more easily than cutbacks which theoretically should also aid in achieving good adhesion.

The above discussion makes it clear that obtaining satisfactory adhesion depends both on the chemical and physical material properties of the bitumen, adhesion agent *and* aggregate (and the delivery method), and cannot be satisfactorily controlled through a bitumen specification alone. This is recognised in the approaches currently taken to control adhesion properties for chipsealing bitumens as discussed below.

3.2 Control of adhesion properties

The current M/1 bitumen specification (clause 5e) requires that for a bitumen to be approved by the Transport Agency, its behaviour when mixed with adhesion agents should not be 'atypical nor likely to require changes to normal construction practices'. In effect bitumens are screened by measurement of the acid number using a titrimetric method with a visual endpoint (Institute of Petroleum 1982). If the acid number is >0.5mg KOHg⁻¹ then a flag is raised and more evidence of satisfactory behaviour is required. The 0.5mg KOHg⁻¹ limit is taken from an earlier version of the M/1 specification and was found to be typical of Safaniya and other middle-eastern crude derived bitumens (the only bitumens used in New Zealand until the late 1990s). A high acid number indicates the presence of acids that may react with and deactivate added adhesion agents.

Further evidence of satisfactory adhesion is usually gathered through a variant of the Vialit test (test method B301-89T developed by Opus Research (formerly Works and Development Services Corporation, Central Laboratories). The Vialit test is discussed further in appendix B), but essentially involves placing chip onto a bitumen film on a steel plate. After conditioning the plate is inverted and a steel ball dropped onto the back and the number of chips dislodged measured. The Central Laboratories version is designed to measure so-called 'active adhesion', in that the chips are damp when applied to the bitumen and the plates are immersed in water for a time before testing. The conditions of the test were an attempt to simulate actual field conditions where chip in stockpiles is often damp when applied.

The test procedure has not been formerly adopted as a Transport Agency test method and has not been updated since 1989. The procedure is given in the Transport Agency specification for adhesion agents TNZ M13 (Transit NZ 1989), which requires that adhesion agents and dosages are approved by the contractor testing each aggregate source to be used. An assumption inherent in the specification is that there is only one bitumen source that will be used, which is not valid today.

The Vialit test is known to be poorly reproducible and is quite operator dependent (Ball 1990; European Bitumen Association 2009). As part of this project improvements to the methodology have been suggested – see appendix B.

3.3 Role of an adhesion test in a performance-based bitumen specification

Control of bitumen-aggregate adhesion is important and it needs to be evaluated for each bitumen-adhesion agent-aggregate system. Including such a testing regime in a performance-based bitumen specification would be impracticable. No better practical test procedure has been identified to warrant replacement of the currently used Vialit test (this is not to say that the test should be improved as discussed in appendix B), and as each bitumen would have to be tested with each adhesion agent-aggregate combination, this would involve many hundreds of tests.

An alternative is to include screening tests that would provide protection against bitumens that may perform poorly and provide batch to batch consistency. However Vialit tests according to the M/13 approach would still need to be undertaken with new bitumen sources by the contractor, as is currently the case when new chip sources or adhesion agents are used.

A screening test could potentially involve a Vialit test using a standard natural aggregate or a synthetic, chemically well-defined 'chip' and a pure fatty amine compound as a standard adhesion agent. A simpler approach, however, would be to measure the bitumen acid number and a physical property related to the ability of bitumen to wet the aggregate surface.

The acid number would be a report only property that would be used by the contractor to determine if new Vialit tests were warranted.

Wetting is to some extent already controlled in a de facto way by the penetration at 25°C which is the basis for grading in the M/1 specification. It is suggested that the MSCR test (AASHTO 2014), or some variant of the procedure, would be used at 25°C to determine wetting ability in a new performance-based specification. This procedure uses a dynamic shear rheometer to apply a constant shear stress to a bitumen sample for a given time and the strain measured (ie a creep test). The stress is removed and the recovered strain measured. The process is repeated for 10 cycles at 0.1kPa loading and 10 cycles at 3.2kPa as shown in figures 3.1 and 3.2, an 80–100 grade bitumen is compared with a 180–200 bitumen in figure 3.3. The cumulative strains involved in the test are large and well outside the linear viscoelastic region (usually <0.5% strain) which makes the procedure relevant to the process chip wetting in the field.

The standard test procedure involves calculation of the average percent strain recovery for each of the two series of creep steps. A high percent recovery for the purposes of chip wetting is undesirable and a maximum value would be set. The method also calculates the average creep compliance (J_{cr}), for each set of creep tests, ie the amount of permanent deformation after time for recovery as shown in figure 3.2 and table 3.1. As the absolute value of the strains produced on each step are small, the method could be modified to improve precision by using a higher creep stress value or allowing more time for each step. Alternatively to avoid having to alter the procedure the total deformation at the end of the test could be calculated and used as a specification value. For the bitumens shown in figure 3.3 the final strains would be 16% and 89% for the 80–100 and 180–200 bitumens respectively. Current binders giving satisfactory on-road performance would be used to provide benchmark specification values. Obviously the binder requirements for chip wetting need to be balanced against other requirements such as chip retention as discussed in chapter 4.

The test procedure (carried out at higher temperatures), was developed in the US to control permanent deformation in asphalt mixes (Bahia et al 2001; Delgadillo et al 2006; D'Angelo 2009; TRB 2010; Wasage et al 2011) and forms the basis of the new AASHTO bitumen specification AASHTO M320-10 (AASHTO

2010). That specification, with minor modifications, has been adopted in the draft New Zealand asphalt bitumen specification, currently being developed by industry (Holleran et al 2014).

Table 3.1 Mean MSCR parameters at 25°C according to the AASHTO method T 350- 14

Parameter	180-200 (6/97/372)	80-100 (6/97/371)
Average % recovery @ 0.1kPa	46.15	66.75
Average % recovery @ 3.2kPa	34.14	59.68
Nonrecoverable creep compliance @ 0.1kPa ($J_{nr,0.1}$, kPa ⁻¹)	0.022	0.004
Nonrecoverable creep compliance @ 3.2kPa ($J_{nr,3.2}$, kPa ⁻¹)	0.027	0.005
Percent difference ($(J_{nr,3.2} - J_{nr,0.1}) / J_{nr,0.1} \times 100$)	23%	27%

Figure 3.1 MSCR test for an 80-100 bitumen at 25°C according to AASHTO T70.

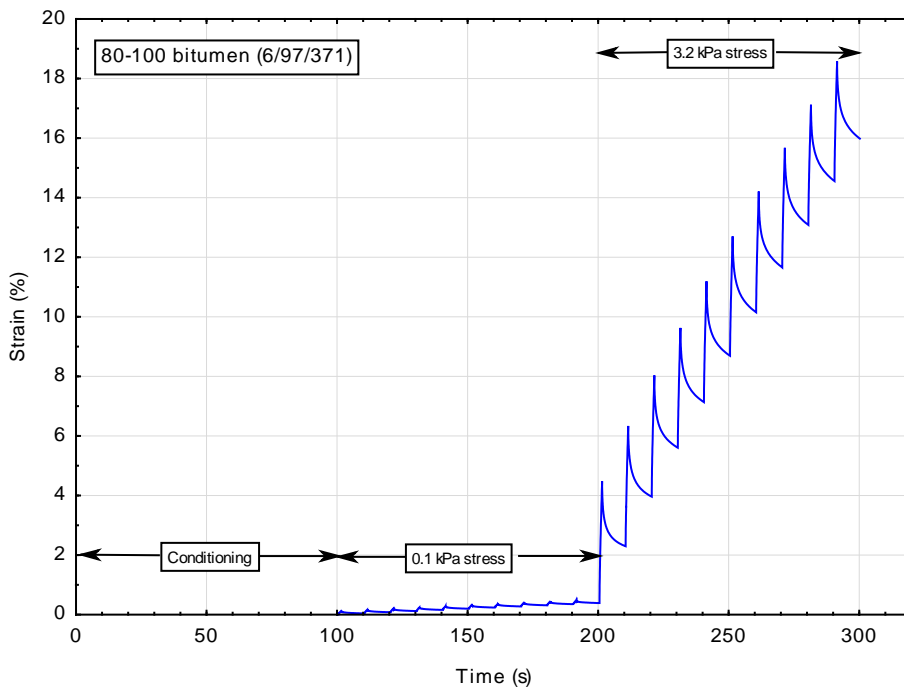


Figure 3.2 MSCR test for an 80-100 bitumen at 25°C showing a single 3.2kPa stress creep cycle

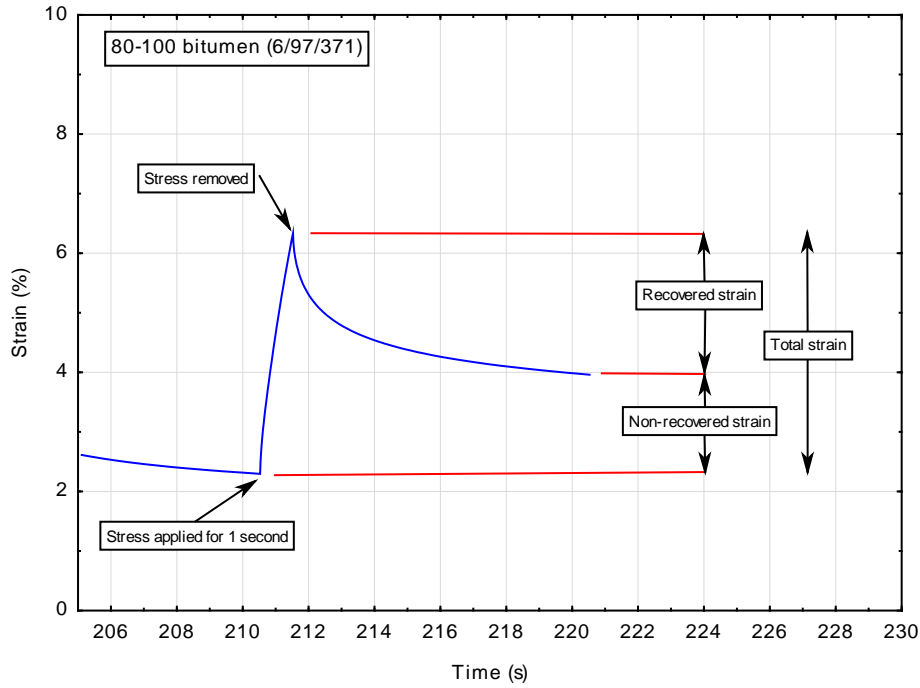
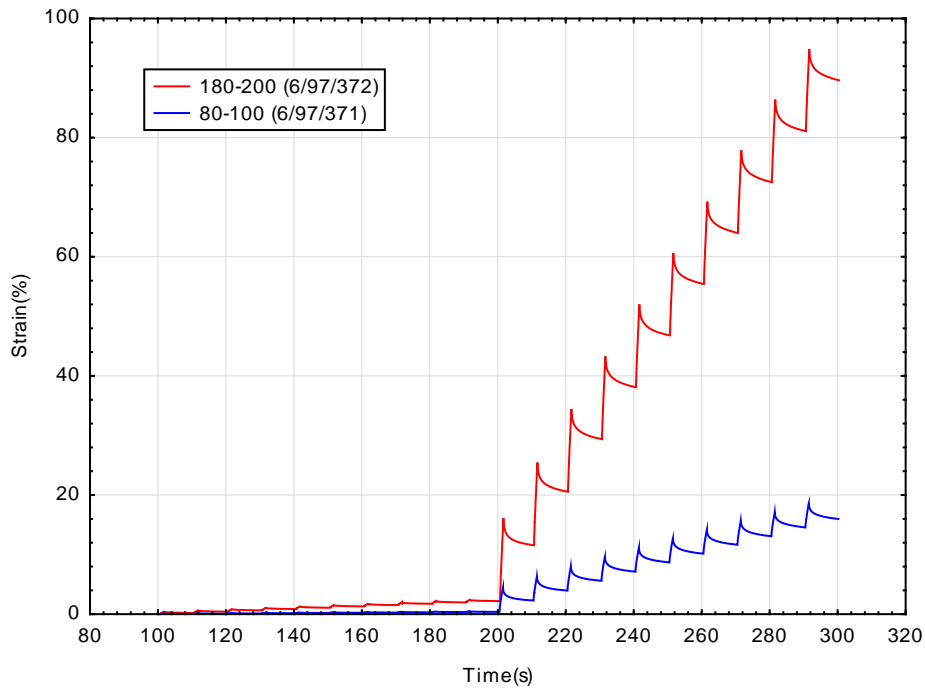


Figure 3.3 MSCR test comparison of 80-100 and 180-200 bitumens at 25°C



4 Chip retention

4.1 Chip loss processes

One of the primary functions of the sealing binder is to retain chip under traffic loadings. In the field, chip loss results from cohesive failure (fracture or yielding) of the bitumen film holding the chip and tends to occur either at very low or very high road temperatures. Establishing the temperature range over which bitumens are expected to perform is an important component of the development of a performance-based specification. Waters (2008) concluded that a range of -10°C to 55°C would cover most roads in New Zealand. An analysis based on three-day average maximum and minimum temperatures and using the model developed by Wood (1998) showed that 100% of state highways were included in the surface temperature range -7°C to 55°C . Only 12.9 km of sealed local authority road fell outside this range (Wu and Herrington 2014). Note that the latter analysis assumed all state highway surfaces were chipseals; asphalt surfacings may experience higher maximum surface temperatures.

At low temperatures chip loss occurs through brittle failure, which can be the result of a single or low number of vehicle passes. Bitumens hardened through oxidation may also lose chip through fatigue cracking of the binder film and possibly in some cases through failure of the adhesive bond to the chip, but the focus of this project was on loss that occurs early in the life of a seal. The latter type of brittle failure is often observed in seals constructed too late in the season or that experience unseasonable frost conditions soon after construction. In that case colder temperatures retard proper reorientation of the chip resulting in the aggregate 'standing up' and being more susceptible to damage from traffic. Poor orientation of the chip can increase the rotational moment and the surface area of chip wetted by the bitumen is smaller. As a result the stresses are higher resulting in rupture of the film. Brittle failure can also occur in a properly constructed seal if the bitumen is too stiff at low road temperatures, and it is this eventuality that a performance-based bitumen specification would aim to control. At present the M/1 specification uses the shear modulus (at 5°C and 9 Hz) measured after accelerated oxidation to control low temperature 'brittleness'.

A higher temperature chip loss or 'chip-roll over' can occur through ductile failure of the bitumen. In this case the chip may not be completely disbonded from the seal but may be simply substantially moved from its original position.

Chip loss in the following discussion is envisioned as a process involving one or a few vehicle passes at most, rather than arising from cumulative damage from thousands of vehicle passes. Chip loss in the present context is distinct from chip loss possibly arising from fatigue cracking or potential loss of adhesion to the bitumen in highly oxidised seals.

4.2 Binder cohesive energy for control of chip retention

The object of the current work was to investigate the bitumen cohesive energy as a specification property that may be suitable for control of chip retention in seals.

4.2.1 Cohesive energy

The thermodynamic work of cohesion (W_c), is defined as the free energy change resulting from the separation of a unit area of the medium (the formation of two new surfaces within the bitumen film) from contact to infinity in a vacuum (Israelachvili 1992), and is given by:

$$W_c = 2\gamma \quad (\text{Equation 4.1})$$

Where γ is the surface energy of the bitumen (in Jm^{-2}).

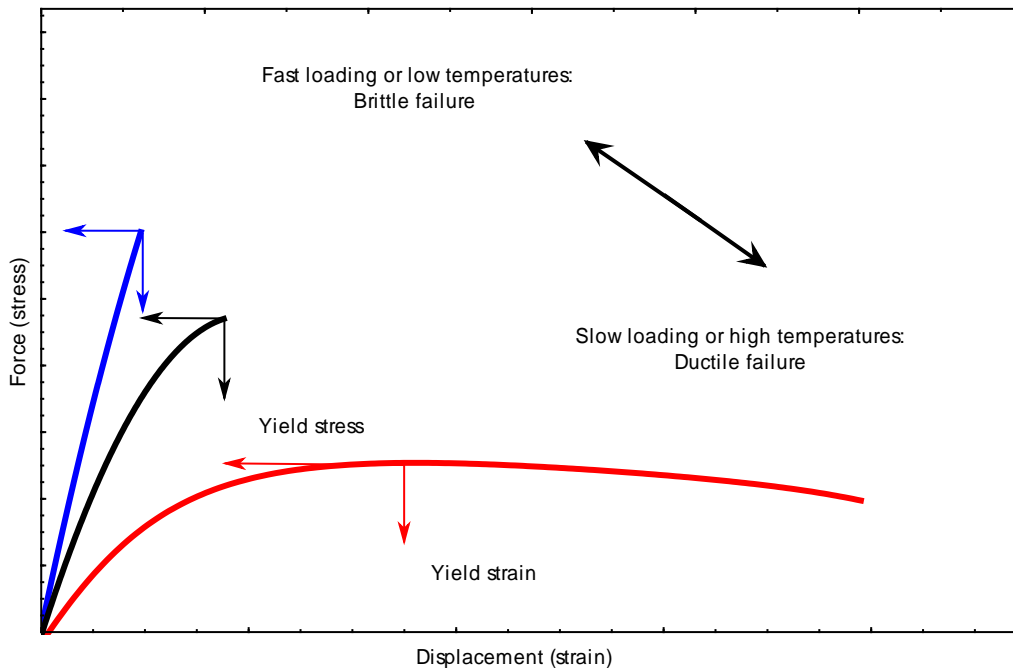
In other words the cohesive energy is the work required to create two new bitumen surfaces. However equation 4.1 applies only to ideal elastic materials, the experimentally measured cohesive energy (W_{eff}) for bitumen, is generally much greater than that predicted by the surface energy alone. Bitumen is not purely elastic at road temperatures so that in addition to W_c , additional energy will be dissipated (work done), in deformation of the film (ie the viscous flow of the binder), as the film ruptures and this is likely to dominate surface energy effects. For bitumen the measured cohesive energy will be dependent both on the temperature and the loading rate as these also affect the viscoelastic properties of the material.

For example, Howson et al (2012) measured the surface energies for several different bitumens and calculated values of W_c in the range of 0.031 to 0.068 Jm^{-2} . Values of the cohesive energy W_{eff} measured for cylindrical bitumen specimens in tension at 23°C, were approximately 200 times greater than W_c . The relationship between W_c and W_{eff} is complex and not well understood (Masad et al 2005).

4.2.2 Measurement of cohesive energy

Cohesive energy for a bitumen specimen can be calculated from the area under a stress strain curve measured at a constant rate of displacement (figure 4.1). The figure shows typical behaviour for bitumen tested under different conditions. At low temperatures (or fast loading rates), the bitumen fails in a brittle manner (blue line). The yield stress and strain (indicated by the arrows) are defined as the stress and strain at the point of rupture. At higher temperatures or slower loading rates the bitumen fails in a ductile manner (red line). The yield stress and strain are taken as the maximum point on the curve. The area under the curve to the yield stress is equal to the cohesive energy of the binder (ie the work required to rupture or yield the specimen). The measured cohesive energy depends on the temperature and loading rate used.

Some of the aggregate adhesion tests described in appendix A, for example the mini-fretting test (Fienkeng and Khalid 1996; Khalid 2000), or the Vialit plate test (Louw et al 2004; Stroup-Gardiner et al 1990; Lancaster et al 1995), have been used (without water immersion), to measure the cohesion of seals constructed with bitumen emulsions. Shuler and Lord (2000) and Shuler et al (2011) also used a test method similar to that of Fienkeng and Khalid to study emulsion curing rates. These procedures work by recording chip loss and so cannot give a direct measure of cohesive energy. Other similar methods have been developed; Santagata et al (1996) describe a method in which a shear load is applied to a seal through a loaded rubber foot. Briggs (1969) and Briggs and Croft (1970) developed a test related to the Vialit plate method employing a seal on a 25mm thick steel plate. The plate with bitumen film and embedded chips is inverted in the apparatus and struck on one side by a pendulum. The plate rebounds off a set of springs. A more even shear load is achieved across the plate than when using the steel ball impact method.

Figure 4.1 Stress- strain curves for bitumen under different conditions

A modified Vialit plate shock test (similar to that described in chapter 2 but without water immersion), is specified in BS EN 12272-3:2003 (BSI 2003) for assessing binder cohesion. A similar test, carried out at -15°C has been used by Waters in a study on New Zealand chipseal binders (Waters 2008). The same test was also used by Davis et al (1991) in the US in an attempt to correlate it with seal performance. A version of the Vialit test was also used (without water), at -5°C as part of South African guidelines for use of polymer modified binders but was found to be poorly reproducible (Louw et al 2004). The principle problem was found to be disbonding of the polymer modified bitumens used from the steel plate rather than cohesive failure occurring in the bitumen. Results were improved if a fine texture was added to the plate surface.

As an improvement over simply measuring chip loss, several authors have measured the force (yield strength) required to dislodge individual chip or a patch of chip from the seal surface (Schweyer and Gartner 1965; Woodside and Rogan 1993; Khalid and Fienkeng 1995; Herrington et al 1998).

Direct measurement of cohesive energy is normally achieved using a tensile test machine to produce a stress-strain curve for a bitumen specimen. For example various stress-strain tensile tests using notched 'dog-bone' type specimens have been developed for measurement of the fracture energy and studying bitumen properties at low temperatures (typically below 10°C), in relation to thermal cracking in asphalt (Roque et al 2013; Tabatabaee and Bahia 2014; Hoare and Hesp 2000).

For higher temperature studies Niu et al (2014) have developed a tensile test for dog-bone specimens suitable for temperatures up to 20°C . Also at 20°C Sultana et al (2014) have studied the effect of confinement ratio and loading rate on cylindrical bitumen films tested in tension. Similar work has been reported by Howson et al (2012) and Masad et al (2005) and much earlier by Marek and Herrin (1958).

A 'toughness-tenacity' test was developed by Benson (1955) and is standardised in ASTM D5801-12 (ASTM 2012). The test measures the area under the tensile stress-strain curve produced pulling a partially submerged a spherical ball from a pool of bitumen. The test was found to have poor repeatability at low temperatures or fast loading rates and is now not widely used (Thompson 1964; Read and Whiteoak 2003).

The bitumen bond strength (BBS) test discussed in appendix A, is another tensile test used to evaluate the cohesion of bitumen films. The force required to pull off a circular metal stub bonded to a polished aggregate surface with a 0.8mm thick layer of bitumen is measured. The yield strength or stress is measured not the displacement, so that the work done cannot be easily calculated.

Johnson et al (2009) and Bahia et al (2010) have developed a 'binder yield energy test' to predict the propensity for fatigue cracking of binders in asphalt mix. This method makes use of the dynamic shear rheometer to measure a (shear), stress-strain curve at 19°C and a constant strain rate of 0.0075 s⁻¹ beyond the point of apparent film 'yield'. The area under the curve up to the point of maximum stress (typically at 100–200% strain), is designated the yield energy and was found to correlate with fatigue cracking rankings for asphalt mixes obtained on a full-scale accelerated loading facility. The method has been further explored by Choi (2014; 2015), to characterise bitumens as part of a new Australian durability test. A drawback with the test as a general method of measuring bitumen cohesive energy is that it is restricted (by DSR instrumental limitations), to intermediate temperatures. At low temperatures (<0°C), the torque of a typical DSR instrument is insufficient and at higher temperatures (>50°C), experiments under the current project with 180–200 grade bitumen, showed that the yield stress is too low to measure accurately.

The only test method currently used to measure bitumen cohesive energy in relation to chip retention in chipseals is the Vialit pendulum test which is discussed further below.

4.2.3 Vialit pendulum test

The Vialit pendulum test chip, BS EN 13588, *Bitumen and bituminous binders- determination of cohesion of bituminous binders with pendulum test* (BSI 2008), is used in Europe to assess the ability of a seal binder to retain chip. This method does not produce a stress-strain curve but measures the energy lost from a swinging pendulum bob (travelling at about 4.4ms⁻¹), as it fractures a 1mm thick, 1cm² film of bitumen between steel blocks as shown in figure 4.2 (Widyatmoko et al 2002; Airey et al 2004; Rowe et al 2014). The test procedure is similar to that of the Charpy and Izod impact strength tests widely used to determine the fracture energy of metals and other materials. Methods for these tests are described in various standards such as ISO 179-1 2010 (ISO 2010) and ASTM D256-10 (ASTM 2010) for the Charpy and Izod tests respectively. The standard test procedures require a notched self-supporting specimen so are only suitable for bitumen testing at low temperatures (Zaic-Kubatovic 2000).

The Vialit pendulum method is used in the UK and Europe particularly to specify polymer modified bitumens and standardised instrumentation available from commercial suppliers. Tests are carried out at a range of temperatures and the maximum value recorded. A maximum cohesion value of greater than 0.7Jcm⁻² is accepted in the UK as corresponding satisfactory cohesion performance for an unmodified binder (Airey et al 2004).

There does not appear to be any published data on the correlation of the test to actual chip retention in seals and the origin of the 0.7Jcm⁻² limit is unclear (R Elliott, Technical Director, URS Infrastructure & Environment, UK Limited, pers comm, 2014).

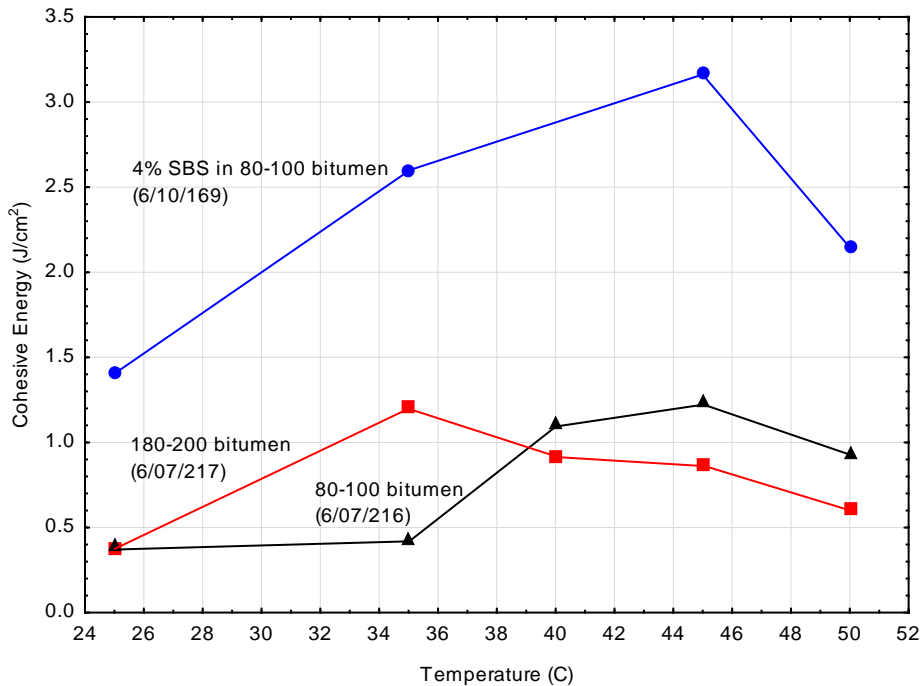
The Vialit pendulum test was used by Waters (2008), as part of a study on climate-based binder selection for chipseals in New Zealand. Various polymer modified (SBS), and sealing grade binders (including kerosene and adhesion agent), were tested at low (-5 to -10°C), and high (45 to 55°C), temperatures. The binder results at low temperatures were all very similar but showed significant differences at the higher temperatures.

Figure 4.2 Vialit test specimen holder showing the point of impact by the pendulum



The standard test method requires that tests are conducted at a range of temperatures to identify the maximum cohesive energy. Tests were carried out for the current project using standard New Zealand bitumen penetration grades (80–100 and 180–200) and a 4%wt SBS polymer modified 80–100 following BS EN 13588 and the results are presented in figure 4.3.

The maximum cohesive energy for the three materials ranges from approximately 35°C to 45°C and is lower at both the low and high ends of the temperature range. This type of behaviour is typical of results reported in the literature (Widyatmoko et al 2002; Airey et al 2004; Rowe et al 2014). The drop in cohesive energy at low temperatures is due to the bitumens failing in a brittle manner (ie less energy is lost in deformation of the bitumen as discussed in section 4.2.1).

Figure 4.3 Effect of temperature on cohesive energy using the Vialit pendulum method

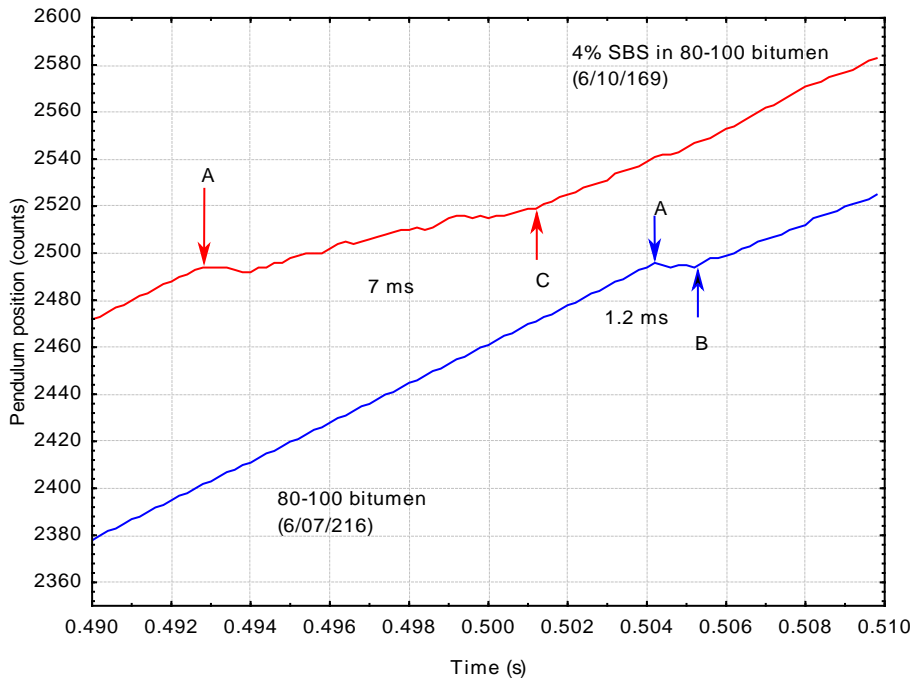
A fundamental problem with the test method is that the configuration of the test specimen holder means that materials failing ductility will retard the pendulum for a longer period than brittle samples even though the film has actually passed the yield point.

To illustrate this point another pendulum test machine was instrumented with an optical encoder (Hengstler model RI58-0/10000EK.47KB), with a resolution of 0.036 degrees per count. The output from the encoder was logged at 5kHz over the time of the impact. The test specimen was formed as a 1mm film between two serrated steel blocks and the top block sheared off by the pendulum – as in the standard test method, except the specimen surface was larger than that of the standard test (1cm in the direction of pendulum travel and 4.9cm wide). The pendulum speed was also slightly slower (3.9m s^{-1}), than that of the standard apparatus (4.4m s^{-1}).

Comparative results for an 80–100 and 4% SBS in 80–100 binders are shown in figure 4.4. In both cases contact of the pendulum with the specimen occurs at point A. For the 80–100 binder the film has ruptured at time B and the motion of the pendulum is no longer impeded. For the polymer modified binder pendulum motion is still affected up to point C; the top block was clearly detached from the bottom but was still connected by long threads of binder. The key point is that the duration of the impact is much longer for the polymer modified binder material; the pendulum is being retarded beyond the yield point of the material and the measured cohesive energy is thus higher than it should be.

Another difficulty with the standard Vialit test method is that at low test temperatures ($<5^{\circ}\text{C}$), or with very hard binders, partial loss of adhesion to the steel substrates is sometimes observed. This was the case when 40–50 grade bitumen was tested even at 25°C . Herrington and Bagshaw (2014) modified the sample holder to produce a dog-bone specimen that eliminated this problem.

Figure 4.4 Pendulum motion over the impact period showing the effect of material ductility (see text for description)



4.2.4 Limitations of cohesive energy measurements for control of chip loss

In the current M/1 specification the ability of a bitumen to retain chip both at low and high temperatures is not directly measured. The cohesive energy is a parameter that could be used to control that property and a standard method (BS EN 13588), and instrumentation is available commercially (albeit with serious drawbacks).

The use of cohesive energy data alone, however, may not be sufficient on its own to predict chip retention.

For properly constructed seals chip loss and seal damage tends to occur at high road temperatures in summer under high shear conditions, especially after two or three days of hot weather. Significant chip loss in cold weather (apart from the exceptional case of late seals or unseasonable ‘cold-snaps’ immediately after construction), is uncommon. This observation is inconsistent with the fact that the measured cohesive energies for bituminous binders, are lowest both at high and low temperatures (as discussed above), and suggests that other factors need to be considered.

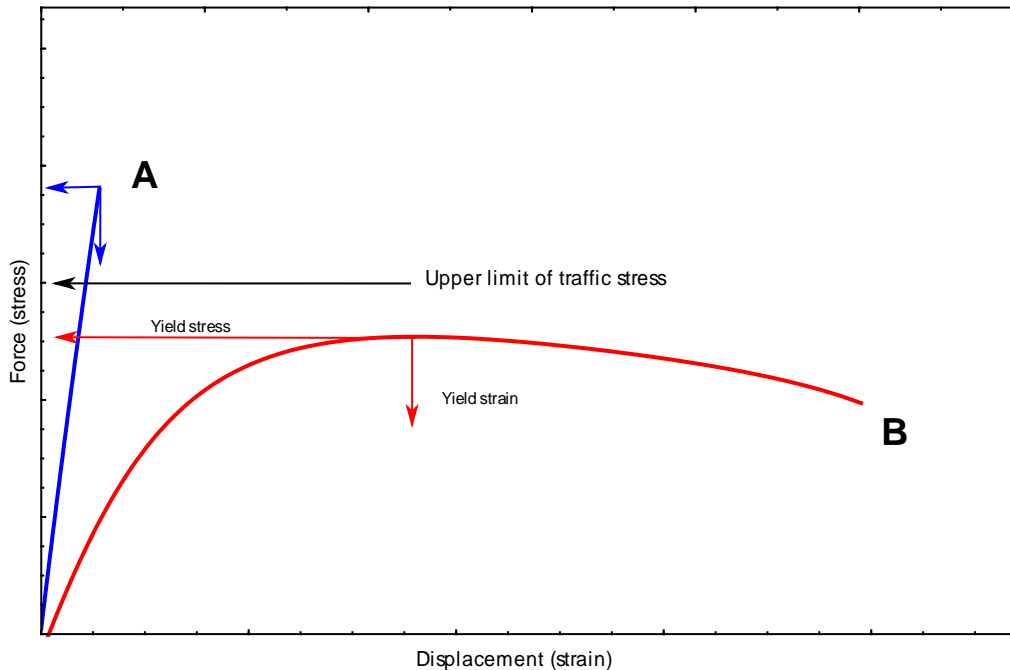
The problem is illustrated in figure 4.5 which shows schematically the stress–strain behaviour of identical films of two different bitumens (A and B), at the same temperature and loaded at the same rate of force increase (the difference in behaviours is exaggerated to illustrate the discussion). This is the situation in a chipseal where the traffic load rises from zero to some maximum value over a period of milliseconds, depending on the speed of the vehicle and the tyre footprint length. A distribution of maximum stress levels (and loading rates), will be applied to any given point in the bitumen film depending on the tyre pressure and the particular resultant angle at which the force is applied and the configuration of the chips beneath the tyre footprint.

The cohesive energy of bitumen B (the area under the curve), is greater than that of A but the yield stress is much lower (red and blue arrows). If the bitumen films were present in chipseal surfacings with an upper bound of the traffic stress distribution as shown, at the same temperature and the same loading

rate, then bitumen B would potentially fail but bitumen A would not. Under these particular conditions chip would be lost from a seal constructed with bitumen B but not that using bitumen A, even though the cohesive energy predicts the opposite.

On this basis the key parameter for chip retention would be the *yield stress* rather than simply the cohesive energy.

Figure 4.5 Schematic diagram showing the potential for error associated with using cohesive energy to rank bitumens in terms of chip retention



4.2.5 Control of chip loss through yield stress and strain

The simplest approach is to conduct a tensile test at the temperature of interest and (in light of the discussion in section 4.2.4), specify a minimum fracture or yield stress and strain at a given test temperature. The performance of satisfactorily performing bitumens would be used as benchmarks to set specification limits which then need to be validated using accelerated laboratory testing and ultimately field data.

Ideally the tests would be conducted at loading rates comparable to traffic loadings. An estimate of the loading rate can be made by assuming a truck tyre pressure of 700kPa and a tyre footprint of 150mm × 200mm (0.03m²), and that the tyre patch area is completely filled with square pyramidal shaped chips. Using data reported by Waters (2008), for the telegraph road site it can be calculated that a typical grade 3 seal has approximately 8,000 chip m⁻² on first construction after loose chip are removed. This corresponds to about 240 chip under the truck tyre footprint with each chip having an effective average base area of 0.000125m⁻². The average load on each chip is thus 0.0875kN. For a truck tyre travelling at 100km/h⁻¹ the time from first contact until the tyre reaches the centre of a chip (a distance of about 0.006m), is 2×10^{-4} s giving an approximate average loading rate of 438kNs⁻¹ or 3.5×10^6 kPa s⁻¹. This load is then maintained for about 0.0072 s as the footprint passes.

Such a loading regime is impractical to achieve without highly specialised and expensive equipment. For a practical test lower loading rates would need to be specified. However, at temperatures below zero

bitumens behave more elastically than at higher temperatures so loading rate effects are likely to be less significant in determining rankings of different bitumens.

4.2.5.1 Low temperatures

At low temperatures a tensile test on bitumen is relatively straight forward. At probable test temperatures (-10 to 0°C) bitumen is brittle and relatively easily handled.

Existing ASTM or European standard test methods for tensile testing of dog-bone shaped specimens using commonly available electro-mechanical test machines could be adapted for the testing. Bitumen specimens would be prepared using silicon rubber moulds. Metal t-shaped end pieces would be cast with the bitumen specimens to provide a gripping surface. A fluid bath or environmental chamber would be needed to control the specimen temperature.

4.2.5.2 High temperatures

Conducting tensile tests on bitumen at high road temperatures (>50°C), is difficult, especially for 180–200 grade bitumen. At such temperatures the bitumen behaviour is largely viscous and specimens are not self-supporting. The situation is also more complex in that at high temperatures specifying yield properties alone would not be sufficient to prevent seal damage.

For a given load, the strains in the bitumen film at high temperatures are likely to be much greater than those at sub-zero temperatures. Even without reaching the yield point of the film, large strains could be said to constitute damage to the seal as the chip may have moved significantly, even without technically being 'lost'. Such damage may also be repaired if the binder shows sufficient elastic recovery.

To minimise the risk of damage the resistance to deformation and elastic recovery of the binder need to be optimised. To cover these variables it is suggested that the MSCR tests described in section 3.3 be adopted in the proposed performance-based specification. The advantage of the MSCR test compared to measurement of viscosity is that elastic recovery data can be readily obtained. The specification would set maximum J_{nr} and a minimum percent recovery values.

Tests on an 80–100 and a 4% SBS polymer modified 80–100 bitumen are compared in figure 4.6. Results of three to five replicate tests for each binder are given in table 4.1. The 180–200 and 80–100 bitumens show greater J_{nr} values than the polymer modified binder, ie greater permanent deformation. Some of the 3.2kPa stress creep steps for the 80–100 and SBS modified bitumens are shown in figure 4.7 illustrating the much greater elastic recovery of the polymer modified binder.

Table 4.1 Mean and standard deviation (in brackets), MSCR parameters at 55°C according to the AASHTO method T 350- 14

Parameter	180-200 (6/12/160)	80-100 (6/97/371)	4% SBS in 80-100 (6/10/169)
Average % recovery @ 0.1kPa	0.78 (1.18)	8.45 (1.03)	72.97 (7.81)
Average % recovery @ 3.2kPa	-5.29 (0.08)	-0.50 (0.07)	36.07 (6.10)
Nonrecoverable creep compliance @ 0.1kPa ($J_{nr 0.1}$, kPa ⁻¹)	6.57 (0.07)	1.44 (0.04)	0.27 (0.10)
Nonrecoverable creep compliance @ 3.2kPa ($J_{nr 3.2}$, kPa ⁻¹)	8.31 (.26)	1.74 (0.04)	0.73 (0.18)
Percent difference, $((J_{nr 3.2} - J_{nr 0.1})/ J_{nr 0.1}) \times 100$	27% (4)	21% (2)	180% (58)

Figure 4.6 Comparison of MSCR test (AASHTO T 350- 14), behaviour at 55°C

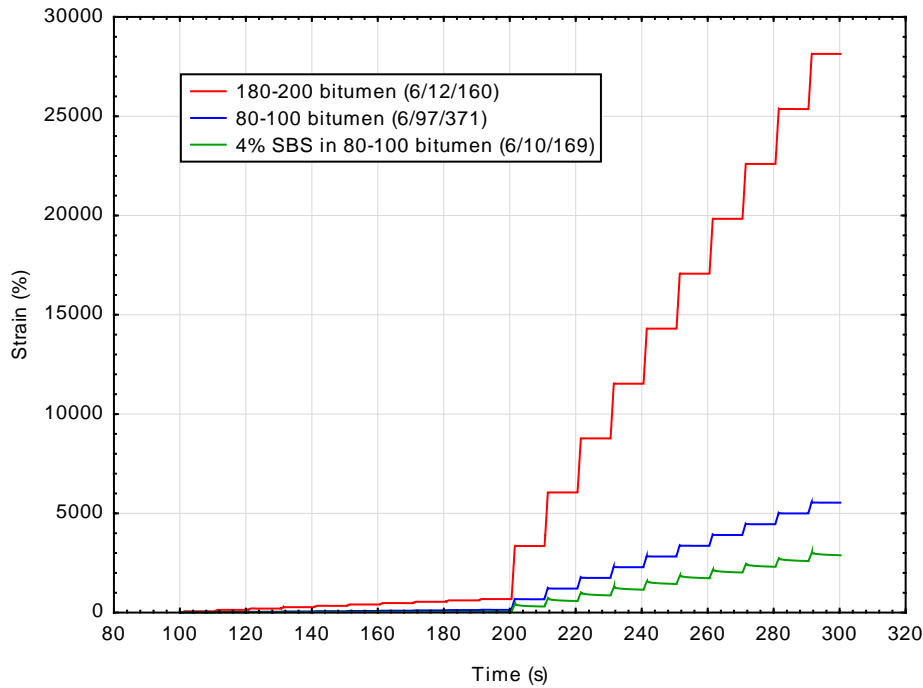
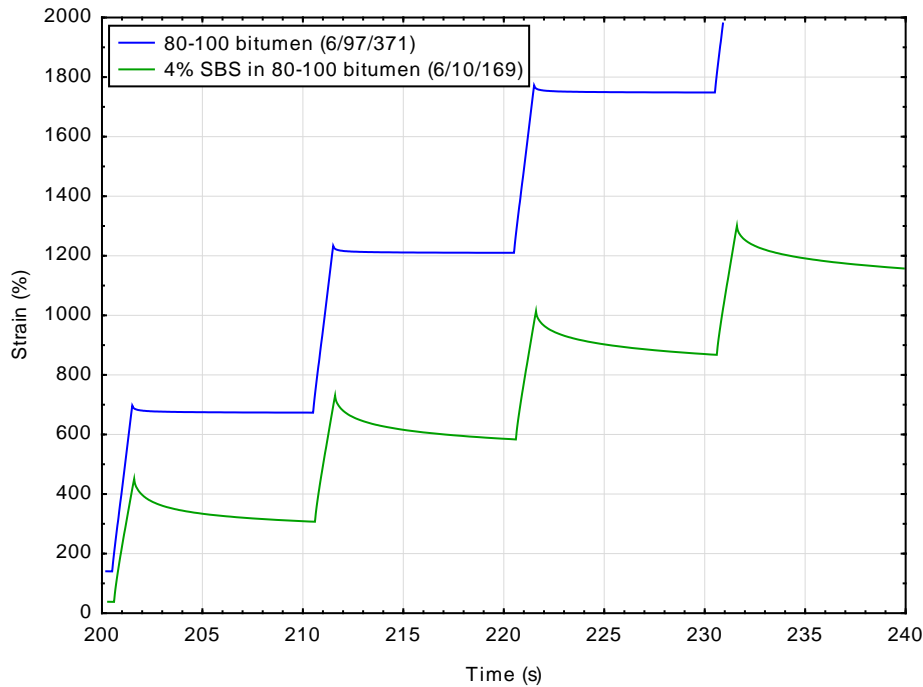


Figure 4.7 Comparison of MSCR test (AASHTO T 350- 14), behaviour at 55°C showing comparative elastic recovery



4.2.6 Bitumen pick-up and tracking

Adhesion of bitumen to vehicle tyres ('pick-up'), and tracking along the surface is a problem related to chip loss and roll-over at high temperatures. Freshly exposed bitumen due to chip loss or disturbance can

adhere to tyres sometimes carrying the chip as well. Pick-up also occurs on new two-coat and sandwich seals where bitumen is exposed to tyres.

This process can accelerate damage to the surface and the tracked bitumen can potentially reduce the skid resistance of the adjacent seal. Research has shown that at realistic loading rates and contact pressures, standard penetration grade and SBS polymer modified bitumens readily form a strong adhesive bond to the tyre rubber at temperatures well within those found in the field (Herrington et al 2010; Herrington 2015). The pick-up of bitumen by the tyre is governed by the yield strength of the bitumen not the strength of the tyre-bitumen interface. The use of the high temperature MSCR test in the specification to minimise the bitumen compliance will thus also help prevent pick-up.

5 Conclusions

5.1.1 Kerosene compatibility

- In practice, differences in kerosene compatibility have been found to be insignificant compared with changes in the base bitumen viscosity at 60°C (in theory these should be taken into account by the contractor when kerosene quantities are being calculated).
- Anecdotally, at least, there is no evidence that these differences have had any practical impacts. The data shows that a kerosene compatibility requirement would not be required in a new performance-based specification. If it was to be retained then the overall effect of slope and base viscosity on the response to kerosene should be included in the assessment.
- It is important to note that the specification limits ultimately set in the new performance specification for chipsealing bitumens must make allowance for the fact that kerosene will be added during seal construction. This will affect the properties of bitumen, especially in the early life of the seal.

5.1.2 Adhesion

- A test to assess potential adhesion problems with chipseal binders needs to take into account chemical affinity, potential degradation of adhesion agents at high temperatures during handling and the physical wetting of the aggregate at ambient temperatures.
- No better, practical test procedure has been identified to warrant replacement of the currently used Vialit test (this is not to say that the test should be improved as discussed in appendix B).
- Control of bitumen-aggregate adhesion is important but it needs to be evaluated for each bitumen-adhesion agent-aggregate system and such a testing regime is beyond the scope of a bitumen specification and would be impracticable to implement in that context.
- Screening tests (acid number and 'wetting test' based on the MSCR test (AASHTO T 350-14) at 25°C, could be included in the proposed performance-based bitumen specification. Such tests would help provide protection against likely poorly performing bitumens and help ensure batch to batch consistency of adhesion properties, but with requirements in place so that Vialit tests according to the M/13 approach would be undertaken with new bitumen sources by the contractor, as is currently the case when new chip sources or adhesion agents are used.

5.1.3 Chip retention

- Chip loss resulting from cohesive failure (fracture or yielding), of the bitumen film holding the chip tends to occur either at very low or very high road temperatures. A performance-based specification should thus include testing of bitumen properties at both low (-10°C–0°C), and high (50°C–60°C) temperatures.
- Measured cohesive energy is strongly affected by the viscoelastic properties of the binder so that cohesive energy alone may not be the most suitable parameter for assessing the ability of a bitumen to resist chip loss.
- An alternative at low temperatures is to measure and control the bitumen yield stress and strain. This can be achieved by a simple tensile test.

- At high temperatures tensile tests are impractical and damage to the seal through large non-recoverable deformations of the binder below the yield strain must also be controlled for. The MSCR test appears useful for this purpose.
- Both the tensile and MSCR test approaches, however, need additional research to quantify their relationship to seal performance.

6 Recommendations

6.1 Kerosene compatibility

- The requirements for kerosene currently in the M/1 bitumen specification should not be included in a performance-based specification for chipseals.

6.2 Adhesion test

- To exclude bitumens with potentially poor adhesion characteristics the proposed performance-based chipseal specification should include tests for acid number (Institute of Petroleum 1982) and chip wetting (the MSCR test at 25°C (AASHTO 2014)).
- Physical adhesion tests on aggregate-bitumen-adhesion agent combinations should continue to be carried out but not as part of the bitumen specification. The effectiveness of the Vialit plate test for this purpose and its current application in the industry needs to be reviewed. The improvements to the methodology suggested in this report may be useful to improve the precision and consistency of the test.

6.3 Chip retention

- Control of chip retention in the proposed performance-based specification should be through measurement of tensile strength at low temperatures. A minimum yield stress and strain would be specified.
- Control of chip retention and permanent binder deformation causing seal damage at high temperatures should be through the MSCR test. Maximum values of the creep compliance and minimum percent recoveries would be specified.

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Appendix A: Aggregate- bitumen adhesion and resistance to water induced stripping tests for chipseals

The literature on various aspects of bitumen-aggregate adhesion theory and testing have been reviewed by Khoo 2014; Airey and Choi 2011; FEHRL 2006; Hefer 2004; Ball and Herrington 1995. The following discussion will focus mainly on methods used to test for adhesion relevant to chipseals rather than asphalt mixes.

Many tests have been developed to assess the breaking rate of bitumen emulsions in contact with aggregate. These tests are related to adhesion tests but are primarily intended to measure the cohesive strength of the binder not the binder-stone interface. This review has been limited to methods in which the role of water in displacing bitumen from the aggregate surface can be measured or calculated.

A1 Contact angle (surface energy measurements)

Several authors have investigated surface energy measurements as a means of ranking the ability of bitumen to wet flat polished aggregate surfaces (Liu et al 2014) or resist water stripping from natural aggregate surfaces (Arabani and Hamadi 2014; Boulange et al 2013; Cuadri et al 2015; Grenfell et al 2014; Hamedi and Nejad 2015; Hefer et al 2007). Although a theoretically sound approach to rank bitumen-aggregate in terms of the thermodynamic favourability of wetting and resistance to disbonding, surface energy measurements are complex to make and the viscosity of the bitumen will affect the time taken to reach equilibrium. Wistuba et al (2012) also concluded that the method was poorly reproducible.

In theory the surface energies calculated will rank bitumens in terms of their thermodynamic resistance to displacement by water for a particular aggregate. Bhasin et al (2007) showed there was a correlation between surface energy measurements and resistance to water stripping as determined by physical tests on asphalt mix specimens. However the rate at which bond formation takes place (an important consideration for chipseals more so than asphalt mix because of the temperature at which wetting takes place), and the actual energy required to disbond will also depend on the physical properties of the bitumen (see section 3.1).

A2 Calorific measurements

The heat transferred when bitumen wets the aggregate surface has been used in the study of aggregate – bitumen affinity by Ensley and Sholtz (1972) and Nowell and Powell (1991). In such experiments the aggregate is finely ground to increase its surface area and increase the heat transfer to a measurable level. The method ranks bitumens based on the amount of heat released when the aggregate is wetted. Measuring the energy changes as water displaces bitumen from the aggregate surface is probably feasible but has not been investigated.

Podoll and Irwin (1990) developed a flow-cell micro-calorimeter technique using a continuously flowing bitumen solution over an aggregate bed. Heat is evolved when the bitumen interacts with the adsorbent (ie aggregate) surface. The process is then reversed so that clean solvent passes over the bed and the amount of adsorbate left on the surface can be determined by the differences between the heats of adsorption and desorption. This method, however, does not take into account the physical properties of the bitumen and how they affect the rate of wetting of the aggregate.

A3 SHRP M-001: Net adsorption test.

This procedure was developed in the 1990s as part of the US SHRP research programme (Curtis et al 1993). A solution of bitumen in toluene is passed through a column packed with small aggregate particles. As bitumen is adsorbed from solution onto the aggregate, the light absorbance of the solution changes. Water can be introduced in small quantities to the solvent to determine its effect on desorption of the bitumen (light absorbance increases). The principle limitation of the procedure is that it does not take into account effects of the physical properties (viscosity) of the bitumen and the aggregate micro-texture on wetting (Senadheera and Yazgan 2008; FEHRL 2006; Walsh et al 1995).

A4 Stone coating tests

There are a large number of test procedures that can be grouped under the general heading of stone coating tests. They typically involve coating aggregate particles with hot bitumen or emulsion, immersing the coated aggregate in water (often hot or boiling water) and visually assessing the amount of bitumen disbonded by the water (eg BS EN 13614:2011 (BSI 2011)); ASTM D3625/D3625M (ASTM 2012)).

Tests of this type are widely used in Australia and have been reviewed by Khoo (2014). The Australian standard method AS 2341.28, *Determination of stone coating ability and water resistance* (Standards Australia 2013) determines the stone coating ability (adhesion) and water resistance of emulsified binder using a standard aggregate (dry and damp) mixture: dolerite and two 'indicator' aggregates (white quartz and limestone). Test results on binder adhesive performance and resistance to stripping are applicable to the standard aggregate mixture only.

Related tests involve pushing stone chips into a film of bitumen followed by soaking and visual assessment of the chips after removal from the film. One example is that used in New South Wales, RMS *Test method T230: Resistance to stripping of aggregates and binders* (RMS 2012a). This method is similar to RMS test method T238 (RMS 2012b) and AS 1141.50 (Standards Australia 1998). The test is used to assess stripping resistance of aggregates (with or without treatment with pre-coating materials) from bituminous binders (with or without the addition of binder adhesion agents). Fifty aggregate particles (10mm) are embedded by hand onto a metal plate which has been coated with the binder under test. The plate is kept in warm water (50°C for four days followed by 24°C for an hour) and then the aggregate is pulled out vertically using pliers and examined. The percentage of stripped or partly stripped particles is determined by visual assessment.

A commonly used coating test is the rolling bottle test, BS EN 12697-11:2012 part A (BSI 2012) in which coated aggregate is rotated in a flask for six hours at 60rpm at room temperature. The amount of stripping is assessed visually. The repeatability of this test is $\pm 20\%$ (FEHRL 2006). A variant of this test involves boiling the coated aggregate in water (FEHRL 2006; Cuciniello 2013). The partially stripped aggregate is treated with excess hydrochloric acid solution to react with carbonate minerals in the exposed stone surface following BS EN 12697-11:2012 part E (BSI 2012). The amount of acid that has reacted is determined by back titration with sodium hydroxide. The test must be calibrated for a given aggregate by a blank titration on uncoated aggregate. The repeatability of this test is $\pm 15\%$ and is generally limited to aggregates with significant levels of carbonates. A similar procedure using acid titration has been developed by the Belgian Road Research Centre (BRRC 1991) to replace visual assessment after a boiling test similar to ASTM D3625.

Gronniger et al (2010) have used software analysis of digital images to attempt to quantify bitumen coverage after the rolling bottle test as a means of improving the speed and precision. The method developed gave results somewhat higher than those determined by visual assessment but ranked

aggregates in the same order. The precision of the method was better than visual assessment but not markedly so. A similar approach has been described by Velasquez et al (2012).

A5 Loading tests

Another group of tests involve applying a load to the bitumen-aggregate bond and measuring the force required to break the bond or measuring the number of adhesive bonds broken (chips lost) for a given load.

A5.1 Bitumen bond strength (BBS) test

The procedure is based on the pneumatic adhesion tensile testing instrument. This device uses compressed air to measure the pull-off strength of a coating from a rigid substrate. Moraes 2011; Kunawee and Bahia 2005; and Copeland et al 2007 have used this device to develop a test method that can be used to assess bitumen disbonding from a flat polished aggregate surface. The binder is used to attach a circular metal stub to the aggregate surface (0.8mm thick bitumen film), which can then be soaked with water. The force required to remove the stub using compressed air is measured. Loading rates in the order of 10^3 kPa s^{-1} can be achieved but the results obtained were imprecise above about 700 kPa s^{-1} (Miller et al 2010). A draft AASHTO specification for this method has been produced (Greyling 2012; Greyling et al 2011). Drawbacks with the method are the potential for partial cohesive failure of the bitumen near the aggregate interface and the use of a polished surface which removes surface microtexture effects on wetting and subsequent bond strength. Jenkins et al (2013) also found that the method needed to be modified to improve precision.

A5.2 Mini-fretting test

This method measures the curing performance and stripping resistance of binder by simulating the fretting and shearing action of traffic on seal surface (Fienkeng and Khalid 1996; Khalid 2000). A seal is produced on an aluminium test plate using the test bituminous emulsion and standard aggregate particles (retained on a 2mm sieve). After curing, the seal surface is abraded in a rotational movement, using a length of rubber tubing attached to a Hobart mixer. Abrasion is carried out in the dry condition, then the abraded test plate is immersed in water for three days and again abraded. The percentages by mass of water loss and loss of aggregate (dry/wet) are recorded.

A5.3 Vialit plate shock test

This method consists of applying chip to a film of bitumen on a steel plate. The plate is then immersed in water, removed and inverted onto a test frame and a steel ball dropped on the back to dislodge disbonded chips. The number of chips that are lost is recorded. The standard method used in New Zealand is B301-89T, a method developed by Works Consultancy Services (now Opus Research) in 1989 and is given in appendix C. The reproducibility of the test is generally considered poor (part of the reason for this may relate to the variable chip wetting obtained using the specified roller and lack of temperature control during that stage). A feature of this particular method is that the aggregate is applied damp to the bitumen. The intention was to measure the active adhesion properties of adhesion agents, ie the ability to assist in the binder wetting and adhesion to a damp aggregate surface (as is often necessary in practice).

Internationally the Vialit method is most often used to assess binder cohesive strength at different temperatures (ie without immersion in water) as described in BS EN 12272-3:2003 (BSI 2003) and discussed in section 4.2.2.

There are numerous variations to this test, for example to reduce problems of loss of adhesion of the binder to the steel plate (rather than the aggregate), Senadheera and Yazgan (2008) modified the plate and holder to reduce the effects from the test plate bending under impact.

A5.4 Discussion

There are a large number of different test procedures currently in use to assess bitumen-aggregate adhesion and resistance to water disbonding in chipseals but there is no general consensus in the literature on the best method of testing. Many test procedures are qualitative which can make them imprecise.

Most methods involve both the bitumen and aggregate as both materials have an influence on the strength of bond formed. The bond depends on both the physical properties (viscosity, micro-texture) and the chemical composition of both bitumen and aggregate, and the effect of all these factors should be included in any test.

The BBS test has been the focus of considerable recent development in the US. A drawback of this procedure though when used for assessing adhesion is that it makes use of a polished aggregate surface. The effect of the micro-texture of the aggregate is not considered in the test. The same criticism can be made of some of the other procedures described above.

All the above procedures make use of natural aggregate. However, conclusions drawn from the results of a particular aggregate-bitumen pair are not necessarily applicable in general. For the development of a New Zealand performance-based specification this is problematic given the logistics of bitumen supply. It is necessary to be confident that a given bitumen is suitable for use with the range of aggregates used in the country before importation.

The rainfall patterns in New Zealand dictate that adhesion agents must be used to assist in achieving good adhesion. These compounds are almost exclusively amine or amido-amine based. None of the test procedures reviewed appeared to consider the effect of bitumen acids reacting with the added adhesion agents during storage at high temperatures.

An ideal method must be quantitative and take into account the effects of bitumen viscosity (or other properties governing wetting rate), aggregate micro-texture, surface chemistries and potential for bitumen reaction with adhesion agents.

Appendix B: Suggested improvements to the Vialit plate test (B301- 89T)

The adhesion test currently used in New Zealand is known as the 'Vialit test', and is presented in appendix C. The test is known to have poor precision, although it is comparable to other commonly used adhesion test methods (Ball 1990). A round robin of five laboratories in 1990 showed that the between laboratory reproducibility of the test method was 54 percentage units at the 80% chip retention level, ie results from two laboratories testing the same materials would only differ by more than 54 percentage units in only one case in twenty. In other words if the true result from the test was 80% chip retention, then one laboratory could obtain a result of 100% and another 46% and both would be valid.

A review of the Vialit test method has highlighted various aspects of the procedure which could be improved to reduce the complexity and potentially improve the precision of the method.

B1 Test temperature

The test recommends that different grades of bitumen are tested at a temperature that gives a needle penetration value of 235 (equivalent to a 180–200 bitumen with 0.7pph of adhesion agent). This is approximately 32°C for an 80–100 but the temperature will vary depending upon the source of the bitumen and the type and amount of adhesion agent used. A simpler approach is to conduct the test at a single standard temperature, 25°C for example, which is close to that at which adhesion will have to take place in the field. The physical properties of the bitumen must be such as to attain some minimum level of adhesion at that temperature. Potentially different temperatures could be used depending on the climate of the region in which the bitumen will be used.

B2 Bitumen plate preparation

The galvanised plates specified in the current method do not have a lip and for some operators are relatively difficult to evenly coat with bitumen, especially near the edges. A plate of the same dimensions but with a raised lip (5mm), would enable much easier and more uniform preparation of the bitumen test film. The stiffer plate may act to increase aggregate loss so that the test pass/fail criteria may need to be changed.

B3 Chip addition

The current process for addition and rolling of the chip is unsatisfactory. The rolling process does not apply uniform pressure to all the chips. A mechanical device could be designed to attempt to remedy this problem or an alternative is to allow the chip to 'sink' into the bitumen and for wetting to occur under the action of gravity only, ie with no rolling. The chip would be placed on the film and left for a set period to allow bond formation to occur and the binder temperature of the chip. The method makes use of a damp chip so moisture would evaporate from the chip surface while the binder was wetting the surface. This may introduce variability so use of a dry chip would probably be necessary (see section B4).

B4 Chip pre-treatment

In the current method the chips are treated in a water saturated atmosphere before application to the plate but during that process they are drying at a rate dependent on the laboratory air humidity and temperature which is variable and use of a dry chip may help reduce this. The concept behind the use of a saturated atmosphere is that it simulates the fact that sealing chip in stockpiles is often damp but other aspects of the test (the way the stress is applied to the plate to dislodge chip for example) are very different to field conditions and the benefit of trying to simulate a damp chip may not warrant the potential variability introduced. The effect of the humidity at which the chips are conditioned needs to be examined to determine if it is a significant variable.

B5 Lack of a control

The current method could be enhanced by use of a 'control stone' as used in the polished stone value test (BS EN 1097-8:2009), to control the friction properties sealing chip (BSI 2009). Additionally a control bitumen and 'adhesion agent' could be specified. The latter would be a pure, chemically well-defined compound similar to commonly used adhesion agents, such as octadecylamine. Control tests would be run in parallel with the test materials and the results used to correct for variations. A complication with this approach, however, is the fact that bitumen properties are known to change gradually over time (even without significant oxidation) and recalibration of the control test results would be needed possibly every four to five years.

B6 Heating of the binder

Adhesion agents can degrade through reaction with acids in the bitumen. This is not modelled in the current method. A heating interval (eg two hours) at 140°C should be introduced to allow reaction to occur.

Better control of the hotplate temperature and time spent in spreading the binder onto the plate should be introduced by specifying ranges for both parameters.

B7 Assessment of cohesive failure

Chips that are dislodged but retain more than '5% binder coverage' are not counted as an adhesive failure. Guidance as to what this means in practice should be provided. Photographs showing examples of chips that should or should not be counted as adhesive failure should be provided to produce greater consistency between operators.

B8 Potential changes to the Vialit plate test

Some preliminary experiments were conducted to explore the effects of modifying the test plate with a raised lip and removing the rolling step. The tests were conducted in a constant temperature room which is operated (for other purposes), at 23°C.

Grade 3 greywacke sealing chip (Belmont quarry, Lower Hutt), were washed and dried at per the standard method but then left at 23°C at 43% humidity for 20 to 24 hours (ie dry conditions). Four bitumen films were prepared by addition of 40g of 180-200 bitumen at 140°C to standard size Vialit plates but with a 5mm high raised lip braised at the corners. The lip made the plates stiffer than those used in the standard

test. Both unmodified and bitumen with 0.5%wt Fentamine BE102 brand adhesion agent were prepared. These plates were then left at 23°C for 20 to 24 hours before 100 chips were applied as in the standard test but without rolling. The plates were left for 17 hours at 23°C and then placed in a water bath (23°C) for 24 hours, removed and tested as in the standard method. Results are shown in table B.1.

Table B.1 Modified Vialit test using raised- lip plates and without chip rolling

Treatment	% chips retained
180-200 test 1	34, 26, 27, 28
180-200 test 2	27, 17, 24, 28
Mean (± 95% confidence limits)	26 ± 4
180-200 + 0.5% adhesion agent, test 1	100, 100, 100, 100
180-200 + 0.5% adhesion agent, test 2	100, 100, 100, 100
Mean	100

A more extensive study, beyond the scope of the current work, would be required to make any general comments about the variability of the modified procedure but the results do show a clear distinction between the two treatments. By altering the bonding and water soaking times the percentage chips retained could be adjusted. The results show that rolling is not required to achieve adequate adhesion. Such a procedure would take several days to complete but would require similar or less labour.

Appendix C: Method used to assess aggregate – bitumen adhesion (Vialit plate test)

B301-89T

CENTRAL LABORATORIES
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DRAFT METHOD

METHOD OF MEASURING THE ACTIVE ADHESION BETWEEN A
BITUMINOUS BINDER AND A ROADING AGGREGATE MATERIAL

1.0 AIM OF TEST

The purpose of this test method is to assess the ability of a given combination of bitumen binder and anti-stripping agent to form a bond with a graded chip sealing aggregate in the presence of water.

2.0 GENERAL DESCRIPTION

In the test, the binder and anti-stripping agent are mixed and poured on to four test plates to give a uniform layer. After cooling to test temperature, a fixed number of pieces of preconditioned aggregate are applied to each plate. Then, after a further period at the test temperature, the plates are inverted and three standard blows applied to the back of each one. The average value for the proportion of stones retained on the plates, expressed as a percentage, is the binder aggregate adhesion for that particular combination of aggregate, binder, and adhesion agent.

In a variation for water-dispersible adhesion agents, the diluted agent is sprayed on to the binder on the test plates immediately before placing the aggregate.

The method can be used to compare the effects of different amounts and/or types of adhesion agent, using a standard aggregate, or, alternatively, to compare different aggregates using a fixed amount of a standard adhesion agent.

3 0 APPARATUS

3.1 Drop-Test Unit (Fig.1) and Steel Ball-Bearing

The unit consists of a three-point mount for holding the test plates, attached to a square table possessing screw feet for levelling. A rod is attached to one side of the table; on the top of this rod is a shallow slightly sloped trough. The ball-bearing is placed on this trough to roll off and fall a known distance on to the test plate, thus providing a standard impact force. The slope of the trough is set so that the ball has a sideways component of motion, and thus strikes the test plate once only.

The ball bearing should be a standard two-inch diameter bearing of weight 534.5 ± 1.0 grams.

The unit must be placed on a rigid, non-flexible, surface (see note 1). It should be adjusted, using the levelling screws, so that when the ball rolls off the trough it will strike any test plate (item 3.3 in this list of apparatus) placed in position on the unit within 15 mm of the centre of the plate (see note 2)

3.2 Catching Tray

This is optional, as the ball-bearing can, with experience, be caught by the operator after bouncing off the test plate. The tray should be large enough to ensure that the ball will strike it after bouncing off the plate, and should have a soft rubber pad at least 10 mm thick covering its base inside.

3.3 Test Plates

These should be made of galvanised sheet steel cut into squares of side 200 ± 2 mm. The original ungalvanised plate should be of thickness 1.85 ± 0.11 mm and the coated plate of thickness 1.90 ± 0.12 mm with minimum average zinc coating of mass (total both

sides) 300 g/m² (New Zealand Steel Limited markets a suitable material). At least four plates are required for each test.

The plates should be as flat as possible. A straight rule aid parallel to any one of the plate edges across either face of the plate should at no point be more than 0.5 mm from the plate surface as measured with a feeler gauge or other suitable equipment

3.4 Roller

A roller with attached handle is required to press the test aggregate into the test binder. The roller cylinder should be covered with a layer of rubber at least 13 mm thick and of hardness such that it satisfies the requirements listed in the annex

The diameter of the covered roller should be 250 ±3 mm. Its weight, including that of any axle required to secure it to the handle, should be 25.0 ±0.2 kilograms.

The distance from the roller axis to the unattached end of the handle should be 1.1 ±0.1 metres, and the weight of the handle 5.45 ±0.10 kilograms.

3.5 Aggregate Conditioning Equipment

This consists of a cylindrical sealable plastic conditioning vessel of minimum internal diameter and height 140 mm and 150 mm respectively. The vessel contains a flat circular corrosion-proof perforated or wire mesh plate whose diameter is not less than 10 mm below the internal diameter of the vessel. The plate should be supported in the conditioning vessel at least 20 mm above the inside base and at least 100 mm below the bottom of the vessel lid. When in place in the vessel, the plate must be sufficiently strong to support a kilogram of aggregate spread over its surface.

A piece of absorbent paper, such as blotting paper, is placed against the inside curved surface of the conditioning vessel so as to completely cover the surface from the base of the vessel to the lid.

3.6 Conditioning Bath(s)

The temperature of the liquid in the bath(s) should be held to $\pm 0.3^{\circ}\text{C}$ of nominal test temperature. Total bath volume available should be sufficient to hold four aggregate-covered test plates and the conditioning vessel immersed to within 25 mm of its top. The bath liquid, in the case of the test plates, shall be distilled or deionised water. Sufficient additional bath volume to accommodate a fog-spray bottle (item 3.24) should also be available if water dispersible agents are to be tested.

3.7 Thermometer to read bath temperature to $\pm 0.3^{\circ}\text{C}$.

3.8 Oven for sample preparation, set at $140 \pm 5^{\circ}\text{C}$.

3.9 Balance to weigh 1000 g to 0.01 g.

3.10 Half-litre cans with press-lid tops.

3.11 Milkshake mixer or similar high shear rate stirrer.

3.12 Sieves for aggregate sorting. For grade 3 aggregate (as defined by New Zealand National Roads Board Standard NRB M/6), sieves of mesh 9.5 and 13.2 mm are required.

3.13 Aggregate washing sieves, nominal diameter 200 mm minimum, depth 50 mm minimum (a 4.75 mm mesh is suitable for grade 3 materials).

3.14 Electric hotplate, area 200 x 200 mm at least, with variable temperature control up to at least 120°C . The effective area of a hotplate may be increased if necessary by placing on top of it a thick (approximately 13 mm) metal plate of appropriate area.

- 3.15 Device to measure hotplate surface temperature to $\pm 1^{\circ}\text{C}$. A digital thermometer with a surface probe is satisfactory for this purpose.
- 3.16 Flexible filling knife with blade width approximately 100 mm.
- 3.17 Glass or brass binder-stirring rod, length 200 mm or greater.
- 3.18 Open-top metal can with outward-curved lip and loose fitting top, volume approximately $\frac{1}{2}$ litre.
- 3.19 Heat resistant gloves.
- 3.20 Board of low thermal conductivity non-metallic rigid material, eg pine wood, with minimum width and length at least 245 and 590 mm respectively, thickness at least 6 mm.
- 3.21 Brass aggregate stirring rod, approximate diameter 6 mm, length 200 mm or greater.
- 3.22 Glass or plastic beaker, nominal volume 1 litre, for aggregate washing.
- 3.23 Distilled or deionised water supply.
- 3.24 Non-absorbent wide-mesh material to make bags to hold test aggregate during conditioning. Nylon stocking/pantihose material has been found suitable.
- 3.25 Fog-spray plastic bottle (for water-dispersible agents only).
- 3.26 Plastic beaker, 400 mL capacity or greater (for water dispersible agents only).
- 3.27 Square polystyrene pad, thickness 12 mm minimum, square side 190 ± 5 mm.

4.0 MATERIALS PREPARATION

4.1 Binder Samples

Unless otherwise agreed the test binder shall be pure 180/200 grade bitumen satisfying NRB specification M/1, of the type used in sealing work in New Zealand (see note 3). The binder should be heated, and samples of 300 grams or more poured into the half-litre cans, which should then be sealed and stored at room temperature until needed for the test. The minimum possible amount of heating should be employed for sample preparation, and exposure of the binder to air while hot should also be minimised. These precautions minimise binder hardening.

4.2 Aggregate Preparation

Sieve the aggregate, if necessary to obtain material of the desired size range. Unless otherwise specified, grade 3 aggregate shall be sieved to pass a 13.2 mm sieve and be retained on a 9.5 mm one

From the (if necessary sieved) material obtain a fully representative sample of the test aggregate of approximately 500 stones (approximately 1 kilogram weight for typical grade 3 chips). Quartering, performed either before or after sieving, is a suitable means of ensuring the sample is representative.

4.3 Agent Preparation

For water dispersible adhesion agents, proceed as follows. Shake the agent container, if necessary, to ensure uniformity of the product, and then prepare approximately 200 grams of water/adhesion agent mixture by mixing the agent with distilled or deionised water in an approximately 400 mL plastic beaker. Agent concentration shall be that to be used in sealing practice. Quantities of water and agent used shall be measured to the nearest 0.01 g and values noted. Mix the solution in the beaker with a clean brass stirring rod, and then transfer it to a clean plastic fog-spray bottle

For solid agents in particulate (eg, pellet) form for which average particle weight is greater than 0.01 grams, grind together at least 30 grams of material, and mix into a uniform paste. Place in a sealed container.

Agents which are liquid or paste, or which consist of very small solid particles, require no special preparation.

5.0 TEST PROCEDURE

5.1

Bring the liquid/s in the conditioning bath^s to the specified test temperature (see note 3). BATHS WHICH WILL HOLD TEST PLATES MUST BE REFILLED BEFORE EACH TEST WITH FRESH DISTILLED OR DEIONISED WATER TO PREVENT CONTAMINATION OF THE TEST BINDER SURFACES.

5.2

Pour sufficient water into the conditioning vessel to give a saturated atmosphere when the vessel is sealed at the conditioning temperature. The water level should be at least 5 mm below the circular plate.

5.3

Unless otherwise specified, clean the aggregate as follows:

Place the approximately 500 stones representative aggregate sample (see Section 4.2) in the aggregate washing sieve, and agitate thoroughly for at least 20 seconds in tap water 100 mm or more deep (a laboratory sink is suitable). Repeat this procedure four times using fresh water each time. Drain the stone. Transfer it to an approximately one litre aggregate washing beaker and wash three times using at least 500 mL of distilled water for each wash, whilst stirring the stone with a clean brass aggregate stirring rod. Rinse the washing sieve with distilled water, upend the aggregate on it, and leave approximately 20 seconds to drain. Do not allow the stone to air dry before carrying out step 5.4.

5.4

Immediately make up four lots of 100 stones each from the washed aggregate, and place each of these lots in bags made from the non-absorbent wide-mesh material. Fasten the bags shut (rubber bands can be used for this), place them on the plate in the conditioning vessel and seal the vessel. No bag should rest on top of any other bag.

5.5

Place the conditioning vessel in a conditioning bath so that all but the top 25 mm (or less) is immersed in the liquid (use a weight to hold in place if necessary). Also place the fog-spray bottle (see Section 4.3), if prepared, into a conditioning bath so that the top surface of the adhesion agent mixture is level with or below the bath liquid surface, with the spray mechanism above the liquid to prevent mixing of both liquid and adhesion agent solution. Leave in the bath/s for 25 ± 2 hours immediately prior to starting to place aggregate on the test binder (step 5.21) (see note 4).

5.6

Place the drop-test unit on a rigid, non-flexible, surface (see note 1). Using the levelling screws, adjust the unit until the steel ball-bearing will roll off the sloped trough to strike any test plate placed in position on the unit within 15 mm of the centre of the plate (see note 2). Do not move the drop-test unit after this adjustment has been made.

5.7

Bring the oven to $140 \pm 5^\circ\text{C}$ and the hotplate to a temperature just sufficient to melt and spread evenly any binder on a test plate placed on it. The hotplate surface temperature should be measured and recorded. Unless otherwise agreed, the plate temperature shall not exceed 85°C for a binder containing 180/200 bitumen or 95°C for a binder with 80/100 bitumen.

5.8

Choose a half-litre can of test bitumen and estimate the approximate weight of bitumen in it by weighing. Loosen the lid on the can, but do not remove it. Place the can in the oven for 2-2½ hours (see note 5). Ensure that the test plates are clean, using an appropriate solvent to remove any material adhering to them (note that new plates may have an undesirable oil film on their surfaces).

5.9

Decide on the weight of bitumen that will be transferred to the open-top can in step 5.10, using the estimate of the weight of bitumen available obtained in step 5.8. Let this amount be W grams. It is suggested that W be as high as practicable to obtain maximum accuracy in doping, but in no circumstances shall it be greater than 400 or less than 250.

Calculate

- (a) The weight of any water dispersible agent solution, prepared as in Section 4.3, that will be required to cover a test plate area of 0.040 m^2 .
- b) The weight of any adhesion agent and/or diluent required to be added to W grams of bitumen to give the test concentrations.

5.10

Place the empty open-top can on the balance and add adhesion agent and diluent(s) in amounts as close as possible to those appropriate to a W gram sample. Note the actual quantity of each material added to the nearest 0.01 grams. Cover the can with a loose fitting lid (see note 6).

5.11

After the bitumen has been 2-2½ hours in the oven, take it out, using the heat resistant gloves, and thoroughly stir the bitumen with the binder-stirring rod. Place the open-top can containing appropriate

amounts of agent and diluent(s) on the balance and immediately transfer W grams of bitumen, measured to the nearest 0.2 grams, into the open-top can. Note the actual mass of bitumen transferred, W' grams, to the nearest 0.1 grams (see note 6). Immediately mix for 2 minutes ± 10 seconds with the high shear rate stirrer, keeping the container away from any heat-conducting surface during this process. Cover the open-top can with a loose fitting lid and replace it in the oven for 15 ± 1 minutes.

5.12

Remove the open top can from the oven and thoroughly stir the binder with the (cleaned) binder-stirring rod.

5.13

Place a test plate on the balance, and pour approximately 40 grams of binder from the open-top can on to the plate. The test plate may be preheated to aid spreading of the binder, but its temperature must at no time be greater than that of the hotplate.

5.14

Cover the open-top can and replace it in the oven until required for the next plate.

5.15

Place the binder-covered plate in the hotplate and, using the flexible filling knife, spread the binder over the test plate as uniformly as possible. Leave the test plate on the hotplate until the binder surface just levels, as indicated by reflection of light off the surface. Do not proceed to step 5.16 until levelling is complete.

5.16

Remove the binder-covered test plate from the hotplate and reweigh it to ensure total binder cover is 40.0 ± 1.0 grams. If not, reject the plate and prepare another.

5.17

Steps 5.18 to 5.29 inclusive shall be carried out under an ambient temperature between 20°C and 30°C inclusive.

5.18

Place the covered test plate on a level surface for 20 ±0.5 minutes in air.

5.19

Repeat steps 5.13 to 5.18 until four satisfactorily coated plates have been obtained.

5.20

Twenty minutes after taking each test plate off the hot plate, immerse it in the conditioning bath to a depth of at least 25 mm for 20 ±0.5 minutes.

5.21

At the end of the 20 minute period remove the test plate from its bath, hold it binder surface downwards, and shake vigorously to remove water. Ensure that as much water as possible has drained from the test binder surface, BUT DO NOT WIPE THE SURFACE.

5.22

Carry out this step only for a water dispersible adhesion agent.

Otherwise proceed to step 5.23.

Place the test plate, binder surface up, on the polystyrene pad on the balance. Ensure that the plate completely overlaps the pad. Remove the plastic fog-spray bottle from the conditioning bath and wipe it dry. Apply the adhesion agent solution to the binder surface, using the fog-spray bottle, moving the nozzle parallel to one edge of the test plate and alternatively at right angles to this, to give an even coating of the solution on the binder surface of weight as close as

possible to that calculated in Section 5.9. The final weight of the coating should be recorded to 0.01 grams. Take care to ensure that no adhesion agent solution is sprayed on to the balance pan. Return the fog-spray bottle to the conditioning bath.

5.23

Place the test plate, binder surface up, with the polystyrene pad beneath, on a flat level surface. Remove a bag of aggregate from the plastic conditioning vessel. Reseal the vessel and leave it in the conditioning bath. Place all the stones from the bag on the binder, adjusting their position by hand so that no stone touches any other. Do not press any stone into the binder to any extent whatsoever.

5.24

Place the low thermal conductivity board on the floor with the test plate on top, binder surface up, taking care not to move any of the stones. Roll the rubber-covered roller over the plate three times in one direction (eg, twice forward and once reverse), then turn the plate through 90° and repeat.

5.25

Replace the plate in the conditioning bath for the required adhesion set-up period. The total period the plate has been out of the bath shall be less than 5 minutes. Unless otherwise specified, the set-up period shall be 2½ hours ±5 minutes.

5.26

Repeat steps 5.20 to 5.25 for the other three plates.

5.27

At the end of the set-up period remove the plate from the bath, invert so that stones and binder are underneath, and place on the three point support on the drop-test unit.

5.28

Place the 2 inch diameter ball-bearing on the trough, and allow it to roll off to strike the back of the test plate. Repeat two more times. The three impacts should occupy a total time of less than 10 seconds and the time between removing the plate from the bath and completion of the impacts should be no greater than 20 seconds.

5.29

Remove the tray from the drop-tester and count the number of stones still adhering to the binder. Inspect the stones which have been detached to see if they retain any significant amount of binder (see note 7).

5.30

Calculate the adhesion value:

$$\text{Adhesion Value} = 100 - \left\{ \begin{array}{l} \text{Number of stones that fall off and do not} \\ \text{retain a significant amount of binder on} \\ \text{a face (see note 7).} \end{array} \right\}$$

5.31

Repeat for the other three trays, to obtain four adhesion values, A_1 , A_2 , A_3 , and A_4 .

5.32

Calculate the binder aggregate adhesion, \bar{A} .

$$\bar{A} = \frac{1}{4} (A_1 + A_2 + A_3 + A_4)$$

5.33

If \bar{A} is not equal to zero or 100 continue as follows; otherwise proceed to step 5.37.

5.34

Calculate the standard deviation of the adhesion value:

$$S_A = \sqrt{1/3 \{(A_1 - \bar{A})^2 + (A_2 - \bar{A})^2 + (A_3 - \bar{A})^2 + (A_4 - \bar{A})^2\}}$$

Calculate the test criterion:

$$5.35 \quad T = \frac{S_A}{\sqrt{\frac{\bar{A}(100 - \bar{A})}{100}}}$$

5 36

If T is greater than 2.0 repeat the test. If the repeat test has $T < 2.0$ accept the repeat value. Otherwise quote both binder aggregate adhesions and their respective T-values and report "abnormally large spread of results".

5.37

Calculate actual dose rates of adhesion agents and cutting agents used in the test from weights recorded during the above test procedure.

5.38

Report (1) Agent type and dose rate in g/m² of agent (excluding diluting water) for water dispersible agents or parts per hundred by mass or volume of bitumen for other agents.

(2) Bitumen crude oil source, refinery of origin, and grade.

(3) Any cutting agents used, in parts per hundred by mass or volume of bitumen.

(4) Aggregate information including:

(a) source;

(b) geological name of parent stone;

(c) original grading, with a description of any sieving done in the laboratory to prepare the test sample;

- (d) washed or unwashed;
 - (e) any other information on aggregate condition which might be considered capable of affecting the result of the test.
- (5) Any departure from the standard methods and quantities specified in this text.
- (6) Test temperature in degrees Celsius.
- (7) Adhesion set-up period.
- (8) Binder aggregate adhesion and its T-value.

5.39

If the test aggregate is too large for 100 stones to fit on a test tray without touching one another, proceed as follows:

5.39.1

Determine the maximum number of stones that can, without undue difficulty, be fitted on to a test plate without the stones touching one another. Let this number be called N.

4.39.2

Carry out materials preparation as indicated above.

5.39.3

Carry out the test procedure as indicated above, with the following modifications.

Step 5.4: Make up four lots of N stones, rather than 100.

Step 5.30: Adhesion value = $100 - \{100/N\}x$

$\left. \begin{array}{l} \text{number of stones that fall off and do not retain a} \\ \text{significant amount of binder on a face (see note 7)} \end{array} \right\}$

Step 5.35: Calculate the test criterion:

$$T = \frac{S_A}{\sqrt{\frac{A(100 - A)}{N}}}$$

Step 5.37: Report also the initial number of stones per test plate (N).

Notes:

1. If the drop-test unit is placed on a flexible surface, increased and erratic adhesion values can result. Avoid, in particular, flexible thin metal bench or sink surfaces and wooden bench surfaces without support immediately below the test unit.
2. The place at which the ball bearing strikes the test plate can be determined by placing a thin piece of adhesive plasticine or a similar material on the upper side of the plate at the centre, and noting the position of the indentation the ball produces. Other methods which will determine the strike point with sufficient accuracy are acceptable.
3. Unless otherwise agreed, under the usual test situation with 180/200 bitumen the test temperature shall be 25.0 ±0.3°C.

In general, reduction of the binder viscosity, all other factors being equal, will increase the adhesion value. Hence if large dosages of agent and/or use of diluents is contemplated, to obtain a useful comparison with other tests it may be considered appropriate to adjust the test temperature so that the physical binder properties approximate those of a 180/200 binder with a typical adhesion agent dosage (say approximately 0.7 pph). It should be noted that this will only give an indicative comparison because chemical

reaction rates, which will affect the adhesion value after any given time, vary with temperature. A 180/200 bitumen containing approximately 0.7 pph of kerosine has a penetration at 25°C of approximately 235 dmm (test time 5.0 ± 0.1 seconds and needle and spindle weight 100.0 ± 0.5 grams), when measured according to the ASTM method D5-83, Standard Test Method for Penetration of Bituminous Materials. It is suggested that for highly diluted or non-standard binders the test temperature be chosen to give this penetration value. A plot of log (penetration) versus temperature is almost linear, and penetrations at two temperatures can therefore be used to locate the adhesion test temperature. It should be noted that:

- (a) The sample preparation method of D5 must be varied to allow doped samples prepared under the conditions of B301-86 to be poured straight into the penetration cans (temperature requirements are altered).
- (b) The larger penetration ("6-ounce") container is required for this test.
- (c) The inherent variability of the penetration test means that a suitable adhesion test temperature can only be determined with limited precision. Hence the repeatability of the adhesion test when the temperature must be varied to allow for changes in binder viscosity will be larger than for the standard test.

4. The aims of this procedure are:

- (a) To condition the stones in a water-saturated atmosphere at the test temperature for 25 ± 2 hours before testing.
- (b) To bring the diluted water dispersible agent to the test temperature.

Any alternative arrangements which do this are acceptable, but the stones must be divided up into lots of 100 (or N - see Section 5.38) before conditioning.

5. The time of the warming procedure is specified so that all samples receive the same treatment, and different degrees of hardening which would affect the adhesion results are avoided.
6. Anti-stripping agent and/or diluents may, as an alternative, be added to the open-top can after the bitumen, rather than before.

In this case the material(s) should be weighed in a small container or on a flat metal plate (the plate for solid or paste agents only) to the nearest 0.01 grams, and added to the bitumen immediately prior to the high speed stirring. If necessary, to ensure that all the weighed material is added, the container or plate should be placed in the bitumen with the materials. Any container so used must be of a form such that all the material in it will thoroughly and uniformly mix with the bitumen during the high speed stirring process.

7. Of bond failures occurring in the impact test, only those at the interface of stone and bitumen are of interest, since it is these that indicate the failure of an adhesion agent. Thus, stones dislodged by rupture within the bulk of the binder (and thus still having bitumen adhering to a face) are not counted.

A "significant amount of binder" shall be binder which has, in the view of the operator, remained adhering to more than 5% of that face of a detached stone which was formerly in contact with the binder layer on the test plate. Binder adhering to the edges of this face shall not be considered in this assessment, as such binder may be there because it caught on protrusions as the portion of the stone near the bonded face was pulled through the binder upon detachment.

ANNEX A

METHOD FOR ASSESSING THE HARDNESS OF THE RUBBER ON THE TEST ROLLER

A1.0 APPARATUS

A1.1 Two steel or brass rods, diameter 6.0 ± 0.1 mm length at least 580 mm.

A1.2 One pair of Vernier calipers.

A1.3 One meter rule.

A1.4 White blackboard chalk

A2.0 TEST METHOD

A2.1 Rule four parallel lines at least 590 mm long on a flat horizontal surface. The distances of the left-most line from the other three should be 60, 200, and 260 mm respectively, each within ± 2 mm.

A2.2 Cover the two rods with chalk to within 50 mm of each end, first of all roughening the rod surfaces with emery cloth if this is necessary to make the chalk dust adhere to them.

A2.3 Lay the two rods along the two inner lines.

A2.4 Using the two outer lines as guides to the position of the edges of the roller surface, draw the roller by its handle slowly along the rods, for a distance of at least 400 mm over the chalked surfaces. Ensure that the rods do not move during this process and that both rods are as near as possible equidistant from the respective nearer edges of the roller.

- A2.5 Measure the widths of the chalk lines produced on the rubber surface of the roller by the rods. Take three readings for each line at three different positions, each position at least 100 mm from either end of the line. Average the six readings.
- A2.6 The rubber on the test roller shall be accepted as being of a satisfactory hardness provided the average chalk line width is between 1.9 and 3.6 mm inclusive.

Note:

Rubber of approximately 80 International Rubber Hardness Degrees is expected to pass the above test. This information is provided only as a guide to roller manufacture. The roller must satisfy the test criterion described above to be acceptable for use in the binder aggregate adhesion test method.

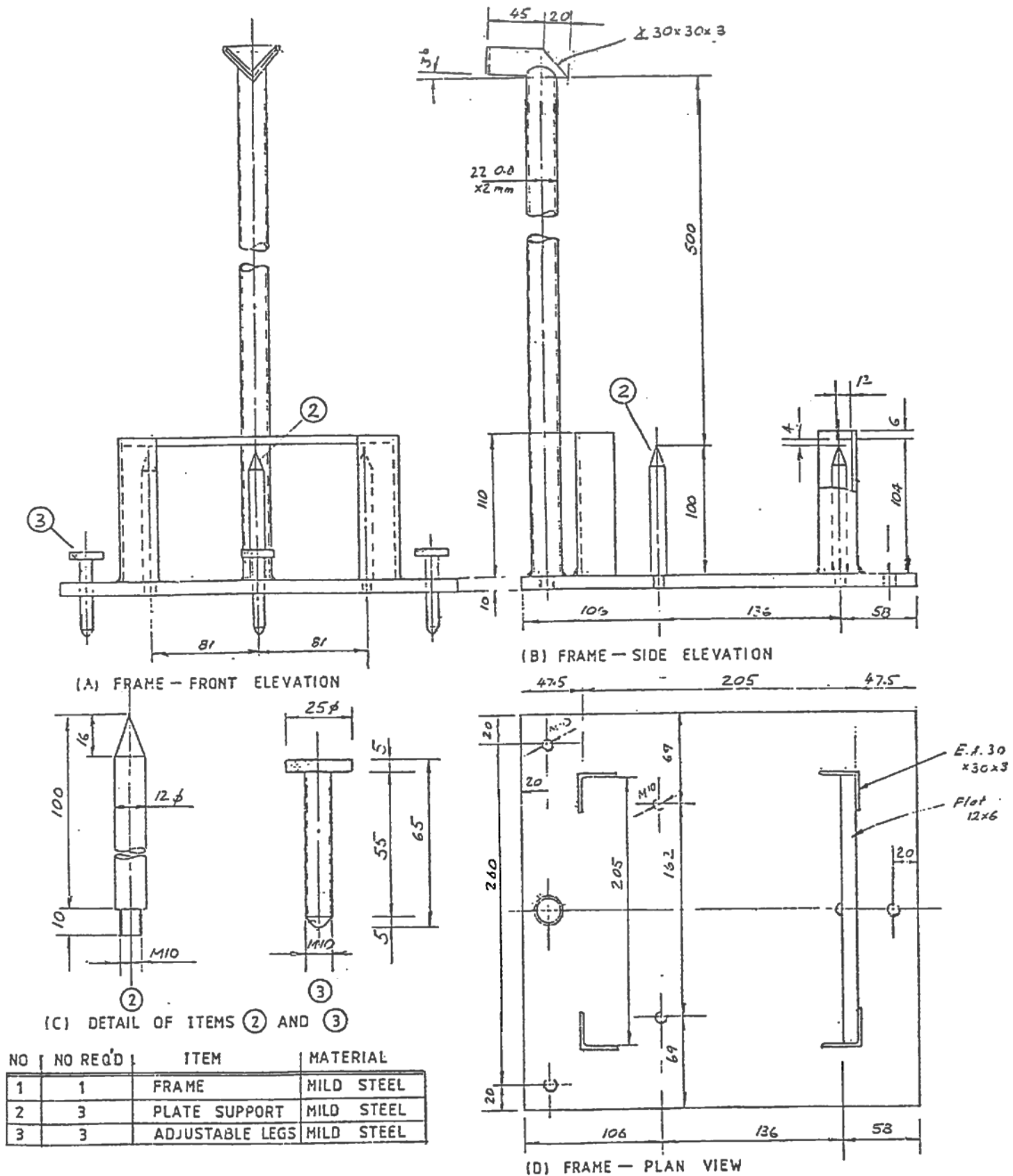


FIGURE 1 VIALIT DROP-TEST UNIT
 DIMENSIONS: MM
 TOLERANCES: $\pm 0.5\text{MM}$ ⁶⁹