

# **Multigrade bitumen for chipsealing applications**

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# Abbreviations and acronyms

**ASTM:** American Society for Testing and Materials

**EVA:** Ethylene vinyl acetate

**PI:** Penetration index

**pph:** Parts per hundred

**RCA:** Road controlling authorities

**RTFO:** Rolling thin film oven (test)

**SBR:** Styrene-butadiene rubber

**SBS:** Styrene-butadiene-styrene

**SH:** State Highway

**VTS:** Viscosity-temperature sensitivity

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# Executive summary

## Introduction

The aim of this research, undertaken in 2009–2011, was to evaluate the potential for multigrade bitumens to improve the performance of chipseals on New Zealand roads by improving chip retention, and by reducing bitumen pickup and tracking by vehicle tyres.

Multigrade bitumens are produced by chemical modification of standard bitumens, and are characterised by a desirable low-temperature sensitivity compared to standard bitumens. For example, multigrade bitumens can be manufactured with the penetration of 80/100 grade but with high temperature viscosities equivalent to 60/70 or even 40/50 grade.

In the current project, aspects of chipseal performance that are likely to benefit from the use of multigrade bitumens were investigated. Work was undertaken to:

- compare the adhesion of multigrade bitumen to tyres to that of standard materials and polymer-modified bitumens
- compare the seal cohesion (chip retention) properties of multigrade bitumen to standard bitumen and polymer-modified bitumens at high road temperatures (60.0°C).

Additionally, a trial was undertaken to demonstrate the constructability of multigrade bitumen chipseals.

The binders studied are shown in table XS1.

**Table XS1 Binders tested in this study**

Binder	Description
180/200	Penetration grade
80/100	Penetration grade
40/50	Penetration grade
M500/170	Multigrade
M1000/320	Multigrade
Styrene-butadiene-styrene modified	3.75% styrene-butadiene-styrene copolymer in 80/100 grade bitumen
Styrene-butadiene rubber modified	4.5% styrene-butadiene rubber in 80/100 bitumen
Ethylene vinyl acetate modified	5.0% ethylene vinyl acetate polymer in 180/200 bitumen
Mastic	10% crushed greywacke aggregate fines (passing a 0.075mm sieve) in 180/200 bitumen
Road	From a five-year-old chipseal on State Highway 5 (Titiokura). Sampled at the scene of a road closure triggered by seal bleed in 2008
Multigrade construction trial bitumen	Multigrade bitumen used in construction trial (before addition of kerosene)

## Bitumen-tyre adhesion

Adhesion measurements were made using a rolling tyre (400mm diameter) travelling at 1.6km/hr. The test bitumen film was formed in an aluminium tray heated from beneath by a silicon heating pad. The tyre temperature was controlled at  $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ .

Adhesion was found to occur at a well-defined temperature rather than gradually over a wide interval. Given the range of binder types studied, the adhesion temperatures were unexpectedly low, ranging from  $33.3^{\circ}\text{C}$  (180/200 bitumen) to  $54^{\circ}\text{C}$  (M1000/320 bitumen).

Although the softening point is often used as an indicator of high-temperature performance, in this case, the softening point correlated poorly with adhesion temperature ( $r^2=15\%$ ) when all the binders studied were considered. The correlation improved significantly ( $r^2 = 93\%$ ) when the polymer-modified bitumens were excluded.

A good linear relationship ( $r^2 = 96\%$ ) was found between the log of the penetration at  $25^{\circ}\text{C}$  ( $P$ ) and the log of the adhesion temperature ( $T_a$ ):  $T_a = 144 \times P^{-0.29}$ , which applied to all the bitumens studied.

It is important to note that these findings are based on a slow tyre speed (compared to traffic speeds) and work is underway to verify these results at higher speeds.

## Seal cohesion

Seals were constructed on  $130\text{mm} \times 120\text{mm}$  steel plates by pouring hot bitumen to form a  $\sim 1.4\text{mm}$  thick layer. Grade 3 greywacke chip was rolled into the bitumen layer and a 50mm square aluminium stud was glued with epoxy resin perpendicular to the surface.

The plate was heated in an oven to  $60 \pm 1^{\circ}\text{C}$  and tested on a pendulum apparatus instrumented with an optical encoder so that the height of the pendulum swing after knocking off the stud and the attached patch of chip was recorded digitally. The speed of the head of the pendulum just before impact was  $3.9\text{m/s}$  ( $14\text{km/hr}$ ), imparting an energy pulse of about 20J.

The highest cohesive energy was measured for a styrene-butadiene rubber (SBR) modified bitumen ( $-14.5\text{J}$ ), whilst the lowest was that for the 180/200 penetration grade bitumen ( $-7\text{J}$ ). Neither of the multigrade bitumens tested was exceptional, both giving results similar to that of the standard 80/100 bitumen.

The log of the shear modulus at  $5^{\circ}\text{C}$  was found to have a good ( $r^2 = 97\%$ ) correlation for five of the binders tested ( $5^{\circ}\text{C}$  moduli data for the SBR-modified binder were not available).

## Field trial

An object of the research was to demonstrate a multigrade bitumen in a trial seal. The trial was constructed on Lever Street in Napier, a straight, flat street in an industrial area near the port. The site carried about 400 vehicles per lane per day, including approximately 3% heavy vehicles. Funding limitations required that the trial form part of routine planned maintenance operations. In this respect, the timing, location and design of the trial were therefore somewhat constrained.

The trial seal consisted of a grade 4 chip with a grade 6 dry-lock coat. The bitumen application rate was  $1.8\text{L/m}^2$ . The design was to add 4 parts per hundred (pph) kerosene, but, because of an error, in practice, on the day of the trial, only 2pph were added. The seal design was that already planned for the site but with the substitution of multigrade bitumen for standard bitumen. The multigrade bitumen used had a



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25°C penetration of 72 (ie between a 60/70 and 80/100 grade) but the 60°C viscosity is close to the viscosity of a 40/50 bitumen.

The field trial showed that sealing with multigrade bitumen is straightforward with no changes to practice being necessary, except that higher spraying temperatures are required and the type of adhesion agent may possibly need to be changed. Although the most commonly used diamine type adhesion agents are not compatible with multigrade binders, alternatives are available. Some problems were experienced during construction of the trial that led to some early chip loss, mainly in high-stress turning areas. These were believed to be unrelated to the properties of the multigrade bitumen as such, but were caused by the low kerosene content used, over-chipping and the late sealing date.

## Conclusion

The field trial has shown that sealing with multigrade bitumen is straightforward, although higher spraying temperatures are required and adhesion agent choice is limited. Experimental measurements of bitumen-tyre adhesion temperatures and chip retention (albeit made at low loading rates compared to traffic loadings) did not show any advantage for multigrade bitumens compared to standard binders with similar 25°C penetration.

## Abstract

Research was undertaken in 2009–2011 to evaluate the potential benefits of multigrade bitumens in chipsealing in New Zealand. A field trial demonstrated that multigrade bitumen seals could be constructed without significant modifications to existing practice except that higher spraying temperatures are required and adhesion agent choice is limited. Experimental measurements of bitumen–tyre adhesion temperatures were made using a rolling wheel apparatus. The cohesive energy of bitumen in artificial seals at 60°C under impact loading was studied using a pendulum device. In both tests, the results for multigrade bitumens were found to be similar to standard bitumens of similar 25°C penetration even though the 60°C viscosity of the multigrade materials was 2–3 times higher.

# 1. Introduction

The aim of this research, undertaken in 2009–2011, was to evaluate the potential for multigrade bitumens to improve the performance of chipseals on New Zealand roads by improving chip retention and by reducing bitumen pickup and tracking by vehicle tyres.

Binder pickup and tracking in hot weather (particularly on new seals) is a major problem for road controlling authorities (RCAs), often leading to complaints and expensive claims for property damage from ratepayers. The problem is especially common on new seals where soft binders can also result in chip rollover, often requiring a new locking coat seal to be applied. Bleeding and tracking of bitumen on state highways from new or partially flushed seals create a hazard through reduced skid resistance and has led to the closure of State Highway (SH) 1 near Foxton in the Manawatu. Chip loss at high summer temperatures is common on both local authority roads and state highways.

Multigrade bitumens are produced by chemical modification of standard bitumens, and are characterised by a desirable low-temperature sensitivity compared to standard bitumens. For example, multigrade bitumens can be manufactured with 80/100 grade penetration but high temperature viscosities equivalent to 60/70 or even 40/50 grade. In practice, the storage behaviour, handling and application of multigrade bitumens is the same as for conventional bitumens. Multigrade bitumens have been widely used internationally, particularly in the United Kingdom and Australia (where the Austroads specification AP-T01 (Austroads 2000) has been developed) but their application has been primarily in asphalt mixtures. The usual means of multigrade bitumen production involves modification with phosphoric acid, a modifier which has become widely used in the United States to modify bitumens to meet the performance graded bitumen grading system without difficulty or performance problems arising (Chin and Oliver 2007; Baumgardner 2010; Romagosa 2010).

The behaviour of asphalt mixes manufactured with multigrade bitumens has been well studied and documented (Koole et al 1991; Robertus 1993; Nicholls 1994), and is markedly superior to that made with standard bitumens. The physical properties of multigrade bitumens (Akeroyd and Holleran 1992) indicate that high-temperature seal performance should be enhanced but, internationally, only limited experimental evidence is available to support this contention. Work carried out by Mobil Oil in Australia indicated that multigrade bitumen should reduce bleeding and stone loss in sprayed seals, but this conclusion was based on observations during and up to only two weeks after construction, and the need for longer term monitoring was indicated (Maccarrone et al 1998).

Multigrade bitumens are not currently manufactured or supplied into New Zealand, nor are they included in the NZ Transport Agency (NZTA) M/1 bitumen specification (NZTA 2007). The cost of multigrade bitumen is higher than that of standard grades; in Australia, the Shell product typically sells for about 15% more than standard grades, but much less than polymer-modified bitumens, which are about 45% more expensive (pers. comm., Mr Nigel Preston, Shell Australia, 2006). The higher cost of multigrade bitumens will prevent them from replacing standard bitumens in many applications but if their high-temperature properties translate into improved seal performance, they may potentially offer a cost-competitive alternative to polymer-modified bitumens in New Zealand.

The current project investigates aspects of chipseal performance that are likely to benefit from the use of multigrade bitumens. Work was undertaken to:

- compare the pickup behaviour of multigrade bitumen onto tyres to that of standard materials
- compare the chip retention properties of multigrade and standard bitumens at high road temperatures.

Additionally, a trial was undertaken to verify the constructability of multigrade bitumen chipseals.

## 2. Materials and test methods

The binders studied are described in table 2.1. Australian multigrade bitumens used in this study were supplied courtesy of Shell Oil Australia Ltd and are typical of the two grades currently being used in Australia. Bitumens conforming to the NZTA M/1 specification (NZTA 2007) were manufactured at the New Zealand Refining Company refinery at Marsden Point and were supplied courtesy of Higgins Contractors Ltd. Styrene-butadiene-styrene (SBS) and styrene-butadiene rubber (SBR) polymer-modified bitumens were commonly used proprietary products supplied by local contractors. The ethylene vinyl acetate (EVA) modified bitumen was prepared using a polymer (Polybilt 101) supplied by Exxon Chemical New Zealand Ltd. The road sample bitumen (see table 2.1) was recovered from a seal sample using dichloromethane at room temperature. Complete removal of the solvent was ensured by treatment in a vacuum oven at 100°C for 1 hour at <100Pa pressure (using an Edwards RV8 vacuum pump). The procedure has been shown to not affect the properties of the recovered binder significantly (Herrington et al 2006). A ‘mastic’ binder was included in the study, as fine aggregate particles are always present in binders in the field.

The multigrade bitumen used in the road trial (chapter 6) was manufactured by Technix Bitumen Pacific Ltd.

**Table 2.1 Binders tested in this study**

Binder	Description
180/200	Penetration grade (NZTA 2007)
80/100	Penetration grade (NZTA 2007)
40/50	Penetration grade (NZTA 2007)
M500/170	Multigrade (AP-T41/06) (Austroads 2006)
M1000/320	Multigrade (AP-T41/06) (Austroads 2006)
SBS modified	3.75% SBS copolymer in 80/100 grade bitumen
SBR modified	4.5% SBR in 80/100 bitumen
EVA modified	5.0% EVA polymer in 180/200 bitumen
Mastic	10% crushed greywacke aggregate fines (passing a 0.075mm sieve) in 180/200 bitumen
Road	From a five-year-old chipseal on SH5 (Titiokura). Sampled at the scene of a road closure triggered by seal bleed in 2008
Multigrade construction trial bitumen	Multigrade bitumen used in construction trial (before addition of kerosene)

The needle penetration and softening point of the binders were determined using American Society for Testing and Materials (ASTM) D5 (ASTM International 2006a) and ASTM D36 (ASTM International 2006b) respectively. Viscosity at 60°C and 135°C was by capillary viscometer according to ASTM D2171 (ASTM International 2007a) and D2170 (ASTM International 2007b) respectively. Ductility was measured according to ASTM D113 (ASTM International 2007c) and the rolling thin film oven test (RTFO) was carried out according to AS 2341.10 (Standards Australia 1982). RTFO measurements were made as a simple indicator of oxidative stability.

Shear modulus and phase angle were measured in the linear viscoelastic region using a Carrimed CSL500 rheometer. Following a standard protocol, samples were briefly (10 minutes) heated to 120°C, compressed placed between parallel plates to a thickness of 1.050mm, then trimmed and further compressed to give a 1.0mm film thickness at the test temperature. Moduli were measured at 5°C and 55°C, typical of the range of temperatures experienced in the field. The error in the measurements is approximately  $\pm 5\%$  (95% confidence limits).

### 3. Comparison of bitumen properties

The properties of the bitumens are compared in table 3.1. Not all tests were performed on all binders because of budget constraints or, in some cases, the unsuitability of the materials for particular tests (eg 60°C and 135°C capillary tube viscosity measurements of polymer-modified binders).

The penetration index (PI) is a commonly used measure of temperature sensitivity. The PI was calculated from the two penetration measurements as shown below in equations 3.1 and 3.2 (Read and Whiteoak 2003; Pheiffer and Doormal 1936):

$$PI = \frac{(20(1 - 25A))}{91 + 50A} \quad (\text{Equation 3.1})$$

$$A = \frac{(\log PT_1 - \log PT_2)}{T_1 - T_2} \quad (\text{Equation 3.2})$$

$T_1$  and  $T_2$  are the penetration test temperatures in °C; by definition,  $T_1 > T_2$ .

More temperature-sensitive bitumens have lower (more negative) PI values. The PI suffers from the insensitivity of the penetration measurement at low temperatures, which can introduce significant errors. At higher temperatures, the viscosity-temperature sensitivity (VTS), as calculated by equation 3.3, can be used as a measure of temperature sensitivity (Puzinanskas 1979).

$$VTS = \frac{(\log \log vis T_2 - \log \log vis T_1)}{(\log T_1 - T_2)} \quad (\text{Equation 3.3})$$

$T_1$  and  $T_2$  are absolute temperatures, and viscosities are in Pa.s.

The moduli ratio ( $G^*_{55}/G^*_{5}$ ) given in table 3.1 is another indication of relative temperature sensitivities.

Table 3.1 Properties of the bitumen used in this study

Property	Bitumen										
	180/200	80/100	40/50	M500/170	M1000/320	SBS modified	SBR modified	EVA modified	Mastic	Road	Multigrade construction trial bitumen
Reference number	6/07/217	6/07/216	6/07/215	6/07/147	6/07/147	6/07/22	6/07/45	6/07/217P101	6/07/217Sa	6/07/26	6/09/46-49
Penetration 5°C (dmm)	19	10	5	15	7	12	17	17	17	5	11
Penetration 25°C (dmm)	189	87	45	66	39	77	124	95	161	41	72
Softening point (°C)	40.4	47.3	54.1	54.6	60.4	70.4	55.2	67.2	41.7	54.7	52.8
Viscosity 60°C (Pa.s)	63	202	528	643	1784	nd	nd	nd	nd	nd	511
Viscosity 135°C (Pa.s)	0.274	0.399	0.680	1.00	1.631	nd	nd	nd	nd	nd	0.848
Retained penetration after RTFO (%)	56	56	54	73	70	nd <sup>a</sup>	nd	nd	nd	nd	60
Ductility after RTFO (m)	>1	>1	>1	0.253	0.204	nd	nd	nd	nd	nd	0.43
PI (5–25°C)	-1.4	-1.0	-1.1	1.5	0.5	-0.1	-0.5	0.5	-1.3	-0.9	0.5
VTS (60–135°C)	3.34	3.51	3.46	3.25	3.28	nd	nd	nd	nd	nd	3.29
G* 5°C (Pa) <sup>b</sup>	18.8E6	38.7E6	77.5E6	25.4E6	61.1E6	nd	nd	nd	nd	nd	nd
Phase angle 5°C (°)	44.6	36.1	27.4	36.6	28.3	nd	nd	nd	nd	nd	nd
G* 55°C (Pa)	5740	15,800	46,700	28,200	73,300	44,800	12,300	21,300	8950	65,300	28,200



Property	Bitumen										
	180/200	80/100	40/50	M500/170	M1000/320	SBS modified	SBR modified	EVA modified	Mastic	Road	Multigrade construction trial bitumen
Phase angle 55°C (°)	83.2	80.5	78.0	71.7	70.4	67.4	64.1	68.5	82.3	69.1	71.3
Moduli ratio ( $G_{55}^*/G_{55}^*$ )	3275	2449	1660	901	834	nd	nd	nd	nd	nd	nd

Notes to table 3.1:

- a nd = not determined  
b All moduli and phase angle data was measured at 9.042Hz.

The multigrade binders have reduced temperature sensitivity compared to the standard grade binder, as demonstrated by the higher PI and lower VTS values, and the lower moduli ratios. The PI values of the multigrade bitumens are also better than those of the polymer-modified binders.

The retained penetration RTFO test results in table 3.1 show that the multigrade bitumens have improved resistance to oxidative hardening in terms of retained penetration compared to the standard grades but the corresponding ductility values are very low. This result contradicts other findings that show increased oxidative hardening (in terms of the 60°C viscosity) after the RTFO test for multigrades (Chin and Oliver 2007). However, researchers in the United States have also reported that phosphoric acid modified binders are no more susceptible to oxidation than unmodified materials.

Using the traditional 'sol-gel' model of bitumen structure (Read and Whiteoak 2003), the low ductility data for the post-RTFO binders indicates a more gelled bitumen, which is probably related to the mechanism by which phosphoric acid acts to reduce temperature sensitivity. Consistent with this interpretation, examination of the phase angle values at 55°C shows that the multigrade bitumens are significantly more elastic than the standard materials (but less than the polymer-modified binders). The significance of the ductility test and its relationship to field performance are, however, unclear. Qualitatively, a low ductility may imply reduced resistance to fatigue cracking but actual measurements of multigrade bitumen fatigue properties suggest no change to fatigue life compared to conventional bitumens (Nicholls 1994).

## 4. Bitumen adhesion to tyres

### 4.1 Background

Under certain conditions, bitumen in chipseals can adhere to vehicle tyres and be tracked along the surface. This is more likely to occur in flushed seals but deformation of the tread means that bitumen well below the top of the chip is likely to be contacted. In extreme cases, bitumen pickup and tracking can lead to loss of skid resistance, damage to the surfacing and even road closures.

Qualitatively, the likelihood that bitumen will adhere to a moving tyre upon contact will increase as the bitumen becomes softer, and as the tyre speed decreases and the tyre load on the surface increases. Adhesion seems most likely on flushed seals for slow-moving heavy vehicles, and when road temperatures are high and the bitumen is bleeding.

Bitumen adhesion to moving tyres has received little attention in the literature (Collins et al 2008) and the relationships between bitumen properties and the likelihood of adhesion have not been determined. Harder bitumens and multigrade bitumens (because of their reduced temperature sensitivity) may have an advantage in relation to bitumen adhesion compared to standard materials.

An apparatus was designed and experiments were undertaken to measure and compare the temperature at which adhesion to a moving tyre first occurred for standard and multigrade bitumens.

### 4.2 Adhesion measurements methodology

Adhesion measurements were made on a rolling tyre apparatus. A small, treaded pneumatic tyre (400mm diameter), loaded with a 100kg dead weight, was pushed in one direction across a flat 2mm thick bitumen film by a pneumatic ram (the bitumen specimen was moved to avoid the return wheel stroke). The average tyre speed in each traverse was 1.6km/hr (the maximum the machine could achieve – see section 4.4). The tyre footprint was 26cm<sup>2</sup> with an average tyre footprint pressure of 207kPa.

The test bitumen film was formed in an aluminium tray (130 × 150mm, with 2mm high sides) and heated from beneath by a silicon heating pad (Watlow Australia Ltd). The temperature of the tray was controlled by a proportional-integral-derivative controller (Carel Australia Ltd) reading from a thermistor fixed with epoxy resin to the underside of the tray. The bitumen temperature was, however, measured directly by a separate bare thermocouple mounted just below the film surface outside the wheelpath. The wheel system and bitumen plate (firmly secured to a wooden base) was mounted inside a temperature-controlled cabinet, which was used to control the tyre temperature at 25° ± 1°C. This temperature is at the lower end of the range of temperatures likely in practice (Novopolskii et al 1993; Ebbott et al 1999). The apparatus is illustrated in figure 4.1.

The bitumen temperature was increased in 0.5–1.0°C intervals and allowed to stabilise for at least 30 minutes. At each temperature, a single traverse was made with the wheel. The process was continued until permanent adhesion of bitumen to the tyre was observed (figure 4.2). Most binders were tested in duplicate or triplicate. After completion of a test, bitumen was cleaned from the tyre using a low-boiling non-aromatic petroleum spirit ('Shellite'). The tyre was dried with a warm air gun and usually left overnight (at 25°C) to ensure all traces of the solvent were removed. Replicate measurements for the different samples tested were randomised in case repeated cleaning of the tyre affected the surface properties and adhesion over time, but no such effects were observed. It is unlikely that any residual traces of solvent affected the results. In some cases, replicates were measured after the tyre had been left for up to a week

after cleaning and were not found to be markedly different to results obtained after the tyre had been left overnight as usual.

**Figure 4.1** Adhesion testing apparatus



**Figure 4.2** Adhesion of bitumen to the tyre



## 4.3 Adhesion temperatures

For comparison to the five standard and multigrade bitumens used, samples of polymer-modified bitumens, a bitumen mastic and a sample recovered from the road were also tested (see table 2.1). Unfortunately, the multigrade bitumen used in the road trial discussed below was not available at the time of testing.

The temperatures at which permanent adhesion was first observed are listed in table 4.1. The pooled estimate of the standard deviation (calculated from those materials with replicate measurements assuming a common population variance) was 1.8°C. The procedure was thus sufficiently precise to distinguish the materials clearly. A feature of the adhesion process was that for a particular binder, adhesion occurred sharply at a well-defined temperature, rather than gradually over a wide interval. Failure in the bitumen film was ductile, producing strings of bitumen. Given the range of binder types studied, the adhesion temperatures were unexpectedly low and fell into a narrow 20°C range and mainly in a 10°C band.

The mastic sample was included in an attempt to simulate more closely the condition of binders in the field, which typically have fine material incorporated. The inclusion of aggregate fines in the bulk of the material (as opposed to a surface layer) had no significant effect on the adhesion temperature.

The road binder was taken from an 80/100 seal in which pronounced binder adhesion and tracking on a steep gradient of flushed seal had led to a severe loss of traction and closure of the road. This material was included in the study because the road surface temperature at the time of the incident could be estimated using data from a nearby weather station and could be used as an approximate verification of the laboratory procedure. The seal was more than five years old so that volatile kerosene fractions (added at the time of seal construction) which would be lost in the solvent extraction process would have largely evaporated (Herrington and Ball 1994). Fine inorganic material present, however, was lost during binder extraction. Even so, the measured adhesion temperature of 49°C agrees well with the maximum road temperature (49°C) on the day the incident occurred.

With the exception of the M1000/320 bitumen, the adhesion temperatures for the polymer-modified and multigrade binders are generally similar to that of the standard materials with similar penetration (see section 4.5).

**Table 4.1 Adhesion temperatures of the studied binders**

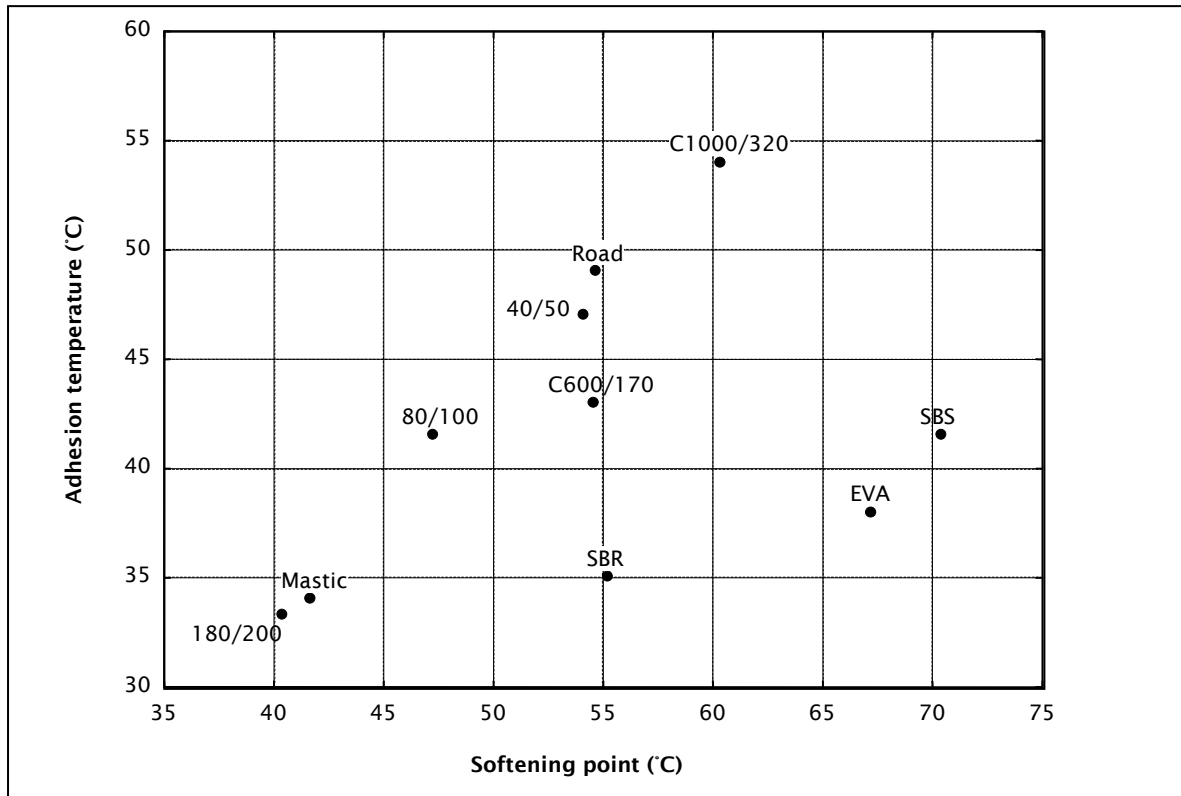
Binder	Adhesion temperature (°C)	Mean adhesion temperature (°C) ( $\pm 1.8$ )*
180/200	34.0, 32.5	33.3
80/100	39.0, 44.0, 41.5	41.5
40/50	47.0	47
M500/170	42.0, 43.5, 43.5	43
M1000/320	56.0, 52.0	54
SBS modified	41.5	41.5
SBR modified	35.0	35
EVA modified	38.0, 38.0	38
Mastic	34.0	34
Road	49.0	49

\*Pooled estimate of the standard deviation for replicate measurements.

## 4.4 Correlation with physical properties

A discussion of the theory relating adhesion to the physical properties of the binders, and correlations of those properties to the adhesion temperatures, has been published separately (Herrington et al 2010) and will not be repeated in detail here. In brief, although the softening point is often used as an indicator of high temperature performance, in this case, the softening point correlated poorly with adhesion temperature ( $r^2 = 15\%$ ), although the correlation coefficient improved substantially to 93% if the EVA, SBR and SBS polymer-modified binders were excluded (figure 4.3). The polymer-modified binders' adhesion temperatures increased as the softening increased but the trend was quite distinct from that of the other binders.

Figure 4.3 Adhesion temperature–softening point relationship for the bitumens studied



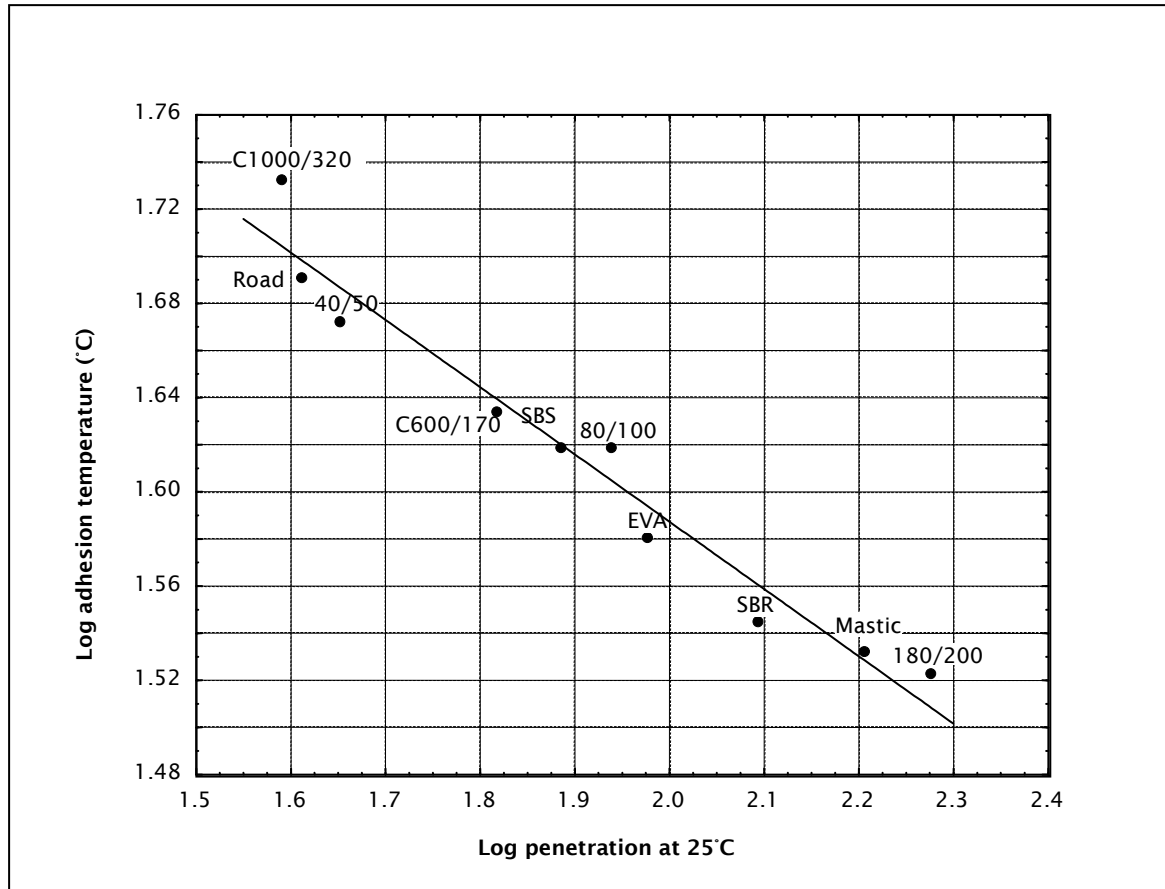
Standard viscosity measurements (capillary tube) at 60°C also did not correlate with adhesion temperature, although this property could not be measured for the polymer-modified and mastic bitumens. Viscosities measured by cone and plate viscometer at the adhesion temperature varied significantly, i.e. the adhesion temperature did not represent an equiviscous temperature (Herrington et al 2010). However, the approximate shear rate under the tyre was estimated to be about 200 per second, two orders of magnitude above that of the laboratory measurements. Measurements made over a range of shear rates at the adhesion temperature appeared to show a convergence of viscosity values (towards a possible equiviscous value) as shear rates increased, but the highest shear rates attained were still <10 per second (Herrington et al 2010).

A good linear relationship ( $r^2 = 96\%$ ) was found between the log of the penetration at 25°C ( $P$ ) and the log of the adhesion temperature ( $T_a$ ) (see figure 4.4 and equation 4.1).

$$T_a = 144 \times P^{-0.29} \quad (\text{Equation 4.1})$$

The multigrade binders have the adhesion temperatures expected from their penetration values rather than the higher values expected from their reduced temperature sensitivity, and relatively higher 60°C viscosities.

Figure 4.4 Adhesion temperature–penetration at 25°C relationship for the bitumens studied



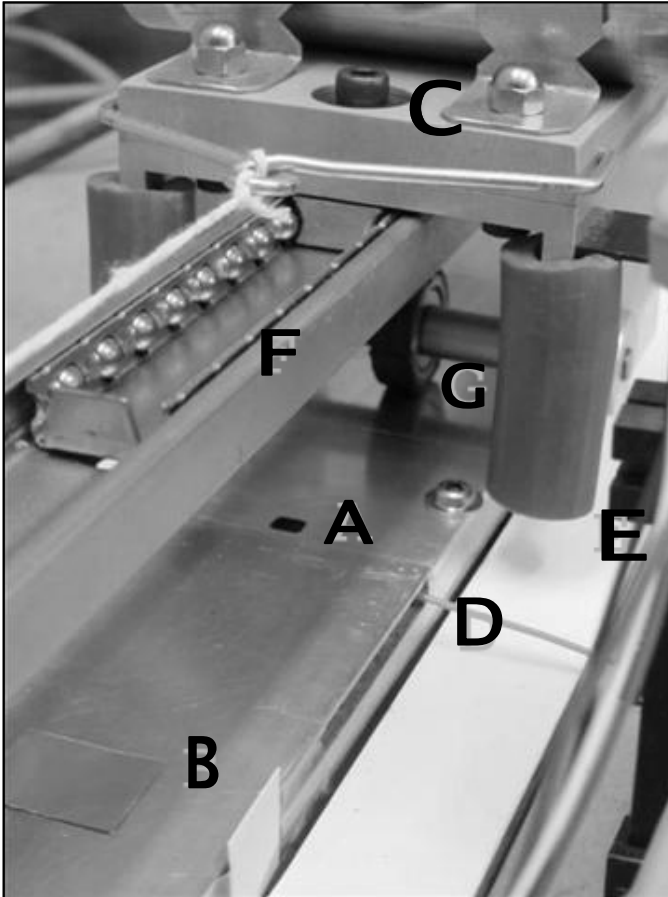
Log adhesion temperature (°C) = 2.16 - 0.29(log pen at 25°C)

$r^2 = 96\%$  Significance of the measured adhesion temperatures for the field

As discussed above, the relevance of the test method to flushed seal behaviour in the field was supported by the good agreement between the adhesion temperature measured for the road sample and the data obtained from a bitumen adhesion 'event' on SH5 in January 2008 (New Zealand Herald 2008).

The conditions under which the adhesion temperature was measured represent an 'almost worst case' scenario. In the field, solid films of bitumen, as used here are, often present in badly flushed seals; however, the surface is generally oxidised and has a fine layer of dust and other detritus which reduce tackiness. The tyre speed used in this study was only 1.6km/h, but faster speeds would also tend to decrease the likelihood of bitumen adhesion. Conversely, the load on the tyre was only 100kg, with a tyre footprint pressure of 207kPa. In practice, this would be up to 650kPa under a truck tyre, which should act to promote adhesion. To verify the adhesion temperature results given above, a new apparatus was constructed that uses a miniature wheel (2.5cm diameter) to produce tyre footprint loadings and loading times that simulate realistic traffic speeds (up to 100km/h), which is shown in figure 4.5.

Figure 4.5 Improved adhesion testing apparatus



Notes to figure 4.5:

A = bitumen patch; B = foil to record adhesion; C = moving trolley with deadweight; D = thermocouple; E = optical speed measurement device; F = running track; G = wheel.

With this apparatus, the temperature is controlled by water circulation beneath the 1.0mm deep bitumen patch. The tyre footprint area is slightly greater than that of the patch so the whole weight of the tyre is not borne by the bitumen. The wheel is attached to a moving 'cart' that is accelerated along the track by dead weights driven by gravity. The cart is fitted to the track in such a way that the weight of the cart is borne by the wheel. The speed of the cart immediately after the bitumen patch is measured electronically. The wheel is maintained at 30°C until just before the test (a test run is complete in 2-3 seconds). The rubber on the wheel was cut from a car tyre.

A full description of this apparatus and the test results obtained with it will be published separately. Initial results using a test speed equivalent to 30km/h show an increase in adhesion temperature as expected, but also appear to support the conclusions drawn above, ie that the multigrade binders do not show exceptional adhesion temperatures for their penetration despite their high 60°C viscosities. For example, the adhesion temperature for 45 penetration bitumen increases to 57°C compared to that of the 39 penetration C100/320 multigrade bitumen, which increases to 56 °C, but the 40/50 viscosity at 60°C is only 568 compared to 1784Pa.s for the multigrade bitumen. Similarly, the 66 penetration C600/170 multigrade bitumen gives an adhesion temperature of 46°C, compared to 49°C for the 87 penetration 80/100 bitumen, even though its viscosity at 60°C is more than twice as great.



## 4.5 Conclusion

The adhesion test results suggest that when seal and traffic conditions are conducive to bitumen adhesion, adhesion occurs at relatively low temperatures and the benefits of the reduced temperature sensitivity of multigrade bitumens will not be particularly apparent. Somewhat counterintuitively, the relatively high 60°C (low shear rate) viscosities of the multigrade bitumens do not appear to give rise to proportionally high adhesion temperatures compared to standard bitumens with similar penetrations.

## 5. Seal cohesion

### 5.1 Seal cohesion measurement methodology

Another potential benefit of multigrade bitumens is in improved chip retention (seal cohesion) under traffic stresses at high road temperatures. A simple test was devised to compare the ability of the binders to resist losing chip at 60°C under an impact load.

Chip retention was measured using the apparatus shown in figures 5.1 and 5.2.

**Figure 5.1** Pendulum apparatus for measuring seal cohesion (specimen in place)



**Figure 5.2 Cohesion test specimen**

A seal was constructed on a steel plate (130mm × 120mm) by pouring on 22–23g of hot bitumen to form an approximately 1.4mm thick layer. The plate had a raised (6mm) rim to contain the liquid bitumen. Crushed, washed and oven dried greywacke chip (180–182g (Pound Road quarry, Christchurch) passing a 13.2mm sieve and retained on a 9.5mm sieve (M6 grade 3 (Transit New Zealand 2004)) was warmed to about 60°C and placed onto the bitumen layer. The chip was rolled with a 50mm wide rubber lino printing roller. A metal stud (50mm square, 100mm high aluminium box section) was glued with epoxy resin (Sikadur-31, Sika (NZ) Ltd) perpendicular to the surface and allowed to cure for about 2–5 days. The epoxy extended beyond the area of the stud and covered a rectangular area of 50mm × 100mm.

The plate was heated in an oven to 60°C ± 1°C and tested on the pendulum apparatus in figure 5.1. A temperature of 60°C was selected as being representative of the upper limit of road temperatures in practice.

The pendulum was raised to a constant height (90° from the resting position) and released to strike the specimen bolted to the base of the apparatus on top of a 6mm under-layer of medium density fibreboard as thermal insulation. The plate was positioned so that the stud was struck at the lowest point of the pendulum swing. The time from removal of the test plate from the oven to pendulum impact was 15–20 seconds, so that heat losses were assumed to be negligible. The apparatus was instrumented with an optical encoder so that the height of the pendulum swing after knocking off the stud and attached patch of chip was recorded digitally. The speed of the pendulum head just before impact was 3.9m/s (14km/h), imparting an energy pulse of about 20J. Friction and air resistance losses in the system were negligible.

After the stud was dislodged from the seal surface, a piece of tissue paper was placed over the exposed bitumen, the stud and epoxy-chip patch were replaced back in position on the seal, and the energy required to move the ‘unbonded’ mass of the stud and attached patch of epoxy and chip was measured.

At the point of impact, the pendulum loses some energy in disbonding the seal patch. The remaining kinetic energy in the pendulum is converted to potential energy as it rises to its maximum height. The potential energy at that point is given by equation 5.1:

$$U = mgL(1 - \cos\theta) + \frac{1}{2}m'gL(1 - \cos\theta) \quad (\text{Equation 5.1})$$

Where:

- $U$  = the potential energy (J)
- $\theta$  = the angle the pendulum rises from the vertical position after impact (rad)
- $m$  = the mass of the pendulum bob (1.53kg)
- $m'$  = the mass of the pendulum shaft (2.04kg)
- $L$  = the length of the pendulum shaft (0.783m)
- $g$  = acceleration due to gravity (9.81 m/s).

The maximum potential energy of the pendulum attained after impacting the bonded stud was subtracted from that of the respective 'unbonded' test to give the energy absorbed by the seal (equation 5.2).

$$\Delta U = mgL(\cos\theta - \cos\theta_i) + \frac{1}{2}m'gL(\cos\theta - \cos\theta_i) \quad (\text{Equation 5.2})$$

Where:

- $\Delta U$  = the energy absorbed by the seal (J)
- $\theta$  = the angle the pendulum rises from the vertical position after impacting the unbound stud (rad)
- $\theta_i$  = the angle the pendulum rises from the vertical position after impacting the bound stud (rad).

## 5.2 Seal cohesion results

It was originally intended to modify the method described above to use cores taken from the field trial seal (see below). Because of difficulties in obtaining a suitable site, the seal type selected by the contractor and the absence of a control, this approach was abandoned. Instead, the field trial bitumen was compared to standard sealing grade binders (the properties of the field trial bitumen are given in table 3.1). This also allowed a wider range of binder types to be studied. Results from the cohesion tests are given in table 5.1; each result is the mean of 6-12 replicate tests.

**Table 5.1 Seal cohesion test results**

Bitumen	Reference number	Energy absorbed at 60°C (J) ( $\pm$ 95% confidence level)
180/200	6/97/372	-7.0 $\pm$ 0.4
80/100	6/97/371	-10.1 $\pm$ 0.5
40/50	6/07/215	-13.0 $\pm$ 0.8
M500/170	6/07/147	-9.0 $\pm$ 0.7
Multigrade trial bitumen	6/09/49	-10.1 $\pm$ 0.8
SBR modified	6/08/45	-14.5 $\pm$ 0.9

The highest cohesive energy was measured for the SBR modified bitumen, while the lowest was that for the 180/200. Neither of the multigrade bitumens tested was exceptional, with both giving results similar

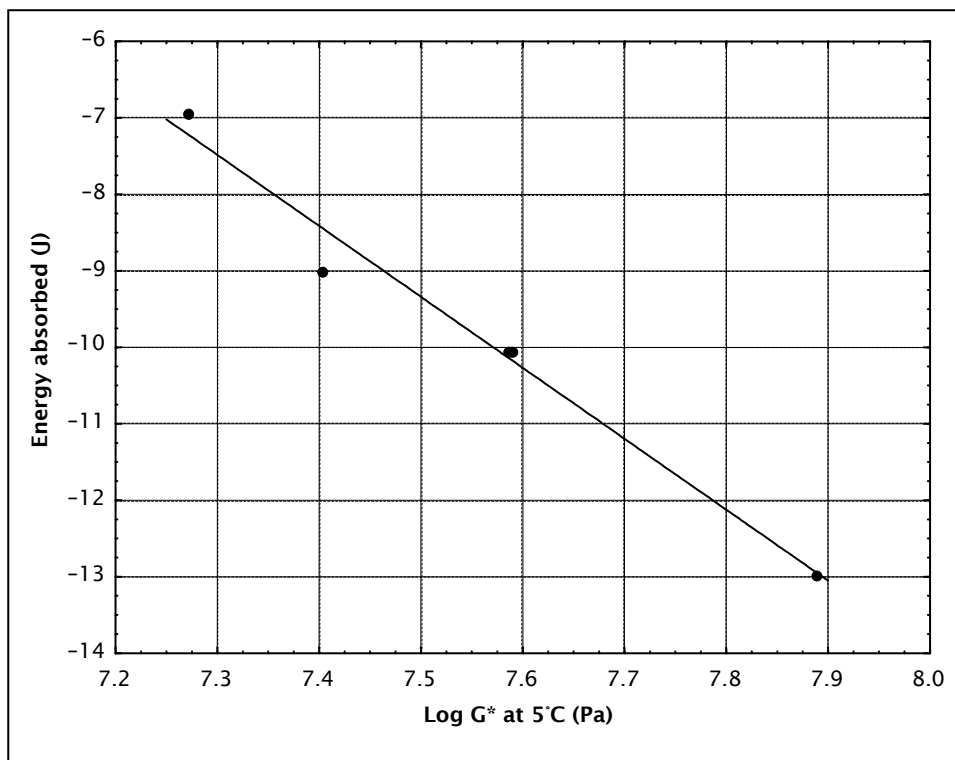
to that of the standard 80/100 bitumen. The very high result for the SBR binder is partly caused by the high ductility of the material (the same reason for the high softening points of polymer-modified binders). It could be argued that the high result is misleading in that the seal has, in effect, failed, even though threads of binder are still acting to retard the pendulum.

No significant correlations between the results in table 5.1 and viscosity at 60°C, softening point, penetration at 25°C or shear modulus at 55°C were found. The log of the shear modulus at 5°C was found to have a good ( $r^2 = 97\%$ ) correlation for five of the binders tested (5°C moduli data for the SBR modified binder were not available), as shown in figure 5.3. This correlation with a low temperature moduli measurement is qualitatively consistent with the fact that at the cohesive energy, the data was obtained under relatively high frequency (impact) loading conditions. Under such conditions, the bitumen may behave as a 'brittle' solid (as it is at 5°C) even at a temperature of 60°C (the time-temperature superposition principle).

### 5.3 Conclusion

At the loading rate employed, the 60°C the cohesive energy levels of the multigrade bitumens tested were equivalent to that of 80/100 bitumen even though the viscosities at this temperature were 2–3 times higher (table 3.1).

Figure 5.3 Relationship between cohesion energy and the modulus at 5°C for the bitumens in table 5.1



$$\text{Energy absorbed (J)} = 60.26 - 9.28(\log G^* \text{ at } 5^\circ\text{C})$$

$$r^2 = 98\%$$

## 6. Field trial

### 6.1 Trial construction

#### 6.1.1 Trial site

An object of the research was to demonstrate a multigrade bitumen in a trial seal. The trial was constructed on Lever Street in Napier, which is a straight, flat street in an industrial area near the port with an existing grade 4 chipseal surface. The site carried about 400 vehicles per lane per day, including approximately 3% heavy vehicles. Several industrial driveways opened onto the street, about halfway along which was an intersection (see figures 6.1–6.3). Funding limitations required that the trial form part of routine planned maintenance operations. In this respect, the timing, location and design of the trial were therefore somewhat constrained. It was unfortunately not feasible to construct an equivalent control section using standard bitumen.

The trial seal consisted of a grade 4 chip with a grade 6 dry-lock coat. The bitumen application rate was 1.8L/m<sup>2</sup>. The design was to add 4 parts per hundred (pph) kerosene, but, through of an error, in practice on the day of the trial, only 2pph were added. The seal design was that already planned for the site with the substitution of multigrade bitumen for standard bitumen.

#### 6.1.2 Trial bitumen

The multigrade bitumen for the trial was manufactured by Technix Bitumen Pacific Ltd in Fiji and shipped to New Zealand. The properties of the multigrade bitumen used in the trial are those given in table 3.1. The bitumen was originally intended to have a penetration in the 80/100 range. The measured penetration was slightly lower than desired but as the trial date had already been delayed several times, it was decided to proceed with the bitumen at hand. The multigrade character of the bitumen is evident from the fact that although the penetration lies between NZTA M/1 60/70 and 80/100 grades (NZTA 2007), the 60°C viscosity is close to the viscosity of the 40/50 bitumen in table 3.1. It is important to note that multigrade bitumens can, in theory, be formulated with penetrations at 25°C that are comparable to standard M/1 80/100 to 180/200 sealing grades.

The acid modification process used to produce multigrade bitumens suggests that the use of amine-based adhesion agents may be problematical (Chin and Oliver 2007). The acid modification mechanism is still under investigation (Masson et al 2009) but if free acid is present in the bitumen then reaction with the amine groups in commonly used adhesion agents may deactivate the additives, although this is not automatically the case (Orange et al 2004; Fee et al 2010 and references therein). In the present work, however, the modification process appeared to result in the deactivation of adhesion agent.

Vialit tests were carried out with a greywacke grade 3 chip and 'Diamine HBG' adhesion agent. The latter is a propylene diamine type agent of the type in common use in New Zealand. Concentrations of 0.7, 1.0 and 1.4pph all gave results of less than 10% adhesion (80% adhesion is considered a pass). However, other tests carried out using Ceca 'Polyram' agent at 1pph on a Technix multigrade made to the Australian M500/170 specification (Austroads 2006) gave adhesion values of over 90%. The differences may reflect differences in the acid concentrations used in the two different products as well as the known poor reproducibility of the Vialit test. Given the ubiquitous use of adhesion agents in New Zealand sealing practice, the effect of multigrade bitumen on their performance is an aspect that needs further investigation. Non-amine type adhesion agents can be used with multigrades (Arnold et 2009; Fee et al 2010) and precoating of chip with adhesion agent is also highly successful (pers. comm. from J. Matthews,

Technix Group Ltd). For this trial, however, these options were not available because of time constraints, so adhesion agent was not added to the bitumen.

## 6.2 Construction

The trial was constructed on 13 March 2009. The binder temperature during spraying ranged from 175°C to 180°C. The day was overcast, with the air temperature reaching a maximum of 18°C. The binder temperature was elevated slightly to allow for the higher 135°C viscosity of the multigrade. The road surface temperature varied from 15°C to 27°C during the course of the day. The seal was rolled with a vibrating steel drum roller and a five-tyre pneumatic roller. The seal was significantly over-chipped, particularly the grade 6 locking coat. The trial construction and progress are shown in figures 6.1 to 6.7.

**Figure 6.1** 13 March 2009: Lever Street before sealing



**Figure 6.2** 13 March 2009: Lever Street-Domett Street intersection before sealing



**Figure 6.3** Applying chip to the Lever Street test site





**Figure 6.4** The hand lancing stage of applying the test seal



**Figure 6.5** 24 March 2009: completed seal on Lever Street

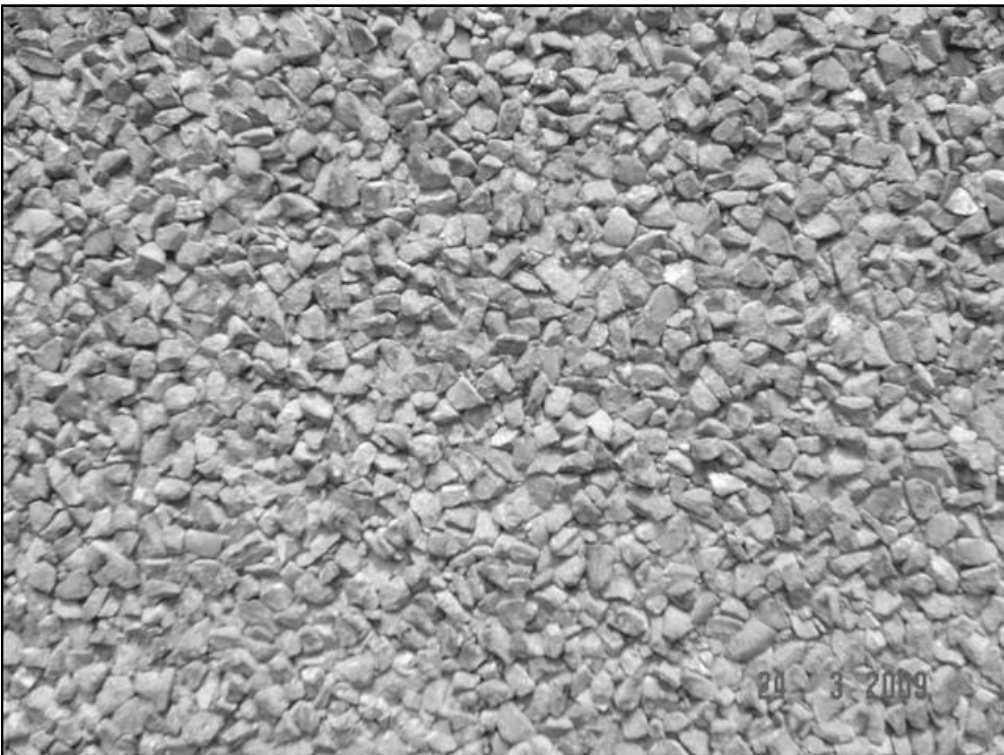


Note: this view is the same as that shown in figure 6.1.

Figure 6.6 24 March 2009: alternative view of completed seal



Figure 6.7 Completed seal, showing over-chipping



### 6.3 Progress of the trial

The principal purpose of the trial was to demonstrate the constructability of multigrade seals. The behaviour of the seal in terms of reduced bleeding bitumen adhesion, and tracking and seal cohesion in hot weather is not likely to become clear for some years beyond the scope of the current project. To this end, observation of the trial is needed for some time into the future.

In winter (August) 2009, portions of the site were found to have lost chip, particularly in areas subject to heavy turning traffic entering and leaving driveways. This was ascribed to the late date of the sealing, and the reduced amount of kerosene used, retarding chip reorientation. The severe over-chipping of the site may also have contributed to the problem. Loss of chip did not result from loss of adhesion of the bitumen to the chip (water stripping), as the chip lost carried bitumen with it. It was noted from examination of the chip lost that, in most cases, binder rise had been limited to only about the first third of the chip height. This suggests that the binder application rate should perhaps have been higher. Areas showing damage were repaired in October 2009 with a 180/200 emulsion applied, using grade 6 chip (figure 6.10). Examination in April 2011 found no evidence of further chip loss but the earlier repairs had flushed (figure 6.11). Spot flushing (by venting) was also apparent in the multigrade bitumen seal (figures 6.12 and 6.13) although binder tracking from these areas was not apparent.

**Figure 6.8** August 2009: chip loss on centre line



**Figure 6.9** August 2009: chip loss in parking lane



**Figure 6.10** July 2010: repairs at the Lever Street-Domett Street intersection



**Figure 6.11** April 2011: looking towards Domett Street, showing flushing of the repairs



**Figure 6.12** April 2011: repairs at the Lever Street-Domett Street intersection, with flushing also apparent in the multigrade bitumen seal (foreground)



**Figure 6.13** April 2011: spot flushing (venting) in the multigrade bitumen seal



## 7. Conclusion

The field trial has shown that sealing with multigrade bitumen is straightforward, although higher spraying temperatures are required. Although most diamine type adhesion agents are not likely to be compatible with multigrade binders, alternatives are commercially available. Problems experienced during construction of the trial leading to some early chip loss were not related to the properties of the multigrade bitumen as such, but were attributable to the low kerosene content used, over-chipping and the late sealing date.

Although continued observation of the trial is recommended, experimental measurements of bitumen-tyre adhesion temperatures and chip retention (cohesive energy) do not show any advantage for multigrade bitumens compared to standard binders with similar 25°C penetration (at least at the loading rates studied).

The postulated advantage of multigrade bitumens as chipseal binders is generally associated with reduced temperature sensitivity. The relatively low temperatures at which bitumen-tyre adhesion occurs appears to minimise any advantage that reduced temperature sensitivity may confer (at least for binders with similar 25°C penetration). In the draft Australian specification, temperature sensitivity is controlled through a high 60°C or 135°C viscosity limit. Compared to the conditions used to measure viscosity (usually with a capillary tube in the order of a shear rate of 0.5–3 per second), both the tyre adhesion and seal cohesion measurements made in the present work can be considered high-frequency measurements. In practice, the loading frequencies experienced by the binder in the field will be even higher than those used here. In that case, viscosity measured at low shear rates may not be a good indicator of likely performance, as appears to be the case.

## **8. Recommendations**

- The trial site should be revisited in 2–4 years to assess the performance of the multigrade seal.
- The performance benefits of multigrade bitumens used in asphalt mix (especially the effect on permanent deformation) should be investigated.



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