ASSESSMENT OF A TEMPERATURE-SENSITIVE BITUMEN FOR CHIPSEALING ON NEW ZEALAND ROADS

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EXECUTIVE SUMMARY

Introduction

The New Zealand Refining Company (NZRC) has been reviewing their refining practice and a range of bitumens of different temperature sensitivities is likely to be available, some of which may not be able to meet the requirements of the TNZ Specification for asphaltic bitumens, TNZ M/1:1995. Economic benefits may be possible if the requirements of TNZ Specification M/1 could be relaxed to permit a more temperature-sensitive bitumen.

Most road bitumen available in New Zealand is used in chipseal surfacings, but the effect of allowing a wider range of temperature sensitivities in them is not clear. The design for these chipseals is based on evolved experience with bitumens whose properties have varied little over decades, rather than on any precise understanding of the relationships between bitumen properties and seal performance.

Experience of the effects on chipseals that might occur with bitumen that is softer at high temperatures and harder at low temperatures is limited. Because of this investment in chipsealing, a better understanding of these relationships is needed.

At low temperatures two phenomena have to be considered:

1. Chip loss during the first winter after sealing can occur if there has been insufficient time before winter for enough traffic compaction to have made the seal stable.
2. Long-term seal failure associated with oxidative hardening of the bitumen.

At high temperatures, the associated reduced cohesive strength of the binder can result in early chip loss under traffic stresses.

Stage 1 Research

Stage 1 of this report describes the development, in 1993-94, of a laboratory test methodology, and the results of these initial tests on a trial bitumen produced by NZRC. They are compared with results from a normal 180/200 Safaniya bitumen.

Factors affecting seal stability were investigated using the pendulum adhesion/cohesion impact tester and chipseal surface stability tester.

Stage 1 research included carrying out additional laboratory tests that go beyond the TNZ M/1 requirements on the initial trial 180/200 bitumen produced by the NZRC (Appendix 1), and on a 180/200 trial bitumen produced from Heavy Iranian crude oil (Appendix 2). They are compared with a normal Safaniya bitumen. These extra tests were performed to check that chipsealing would not be adversely affected if these trial bitumens were used.

In the event, the initial trial bitumen was found to be similar to the current Safaniya bitumen, and it was not used in the further trials in Stage 2 concerning temperature-sensitivity. The trial Heavy Iranian bitumen was however sufficiently different to warrant further investigation and field trials.

Stage 1 Conclusions

Low temperature testing

1. Using the low temperature test as it was set up for this project, significant chip loss only occurs at temperatures lower than those experienced on New Zealand roads.

2. Variability of the test results, as measured for one chip size and one binder thickness at one temperature, is large enough to require multiple testing to be done. Possibly this variability alters significantly with variation of binder thickness and proportion of chip lost. This method is not practicable for routine use.

3. As expected, greater binder thickness gives better chip retention at a lower temperature. The decrease in retention accelerates at lower temperatures, so that an approximate critical temperature where loss becomes significant can sometimes be defined.

4. Larger chips give poorer retention than smaller chips for equivalent percentage binder rise in the seal.

5. These two items above suggest that first winter sealing chip loss may commence only above a certain binder hardness, dependent on both chip size and degree of compaction.

High temperature testing

6. Repeatability of the high temperature test is good, as the critical temperature above which chip loss becomes important can be determined to within 5 °C, and possibly even to within 3 °C.

7. Less compacted seals have lower critical temperature, and tend to lose chip at lower temperatures.

Stage 1 Recommendations

1. Comparison of a limited set of results for two bitumens of significantly different low-temperature penetrations would be of considerable value. This comparison would determine whether binder penetration is a suitable quantity to define susceptibility to early low temperature chip loss.

2. A comparison should be carried out on the effect of equally rolling seals of different chip size and equivalent binder thickness (as a proportion of ALD). Data from this would allow the determination of whether or not equally rolled seals of different chip sizes are equally stable against chip loss.

3. Repeat testing with binders of different temperature sensitivities is recommended, to determine if the presumed correlation between viscosity and tendency to chip loss with changes in temperature is justified.

4. The test programme should be extended to check the effect of different binder spray rates, and chip shapes and sizes. These factors may result in seals of different stability at higher temperatures. A field trial would need to incorporate these possibilities, and further laboratory work in this area may be useful before finalising trial details.

Stage 2 Research

As a result of the Stage 1 research, and in response to NZRC producing another bitumen using a Heavy Iranian crude, further laboratory tests and field trials to assess its responses to low and high temperatures were carried out between 1994 and 1996. This bitumen was slightly outside the TNZ M/1 specification. Both laboratory tests and field trials were performed to determine if the early life (first year) of chipseals constructed using this bitumen would be expected to be significantly different from the currently used bitumen that is produced from a Safaniya crude.

The standard physical tests (viscosity and penetration) showed that:

1. at low pavement temperatures (approximately 0 °C) the Iranian bitumen is harder than Safaniya by a factor equivalent to a change in temperature of approximately 4 °C;

2. at high pavement temperatures (approximately 60 °C) the Iranian bitumen is softer than Safaniya by a factor equivalent to a change in temperature of approximately 5 °C.

A laboratory-based simulation of performance was performed by constructing chipseals on a steel plate and then subjecting them to an impact designed to simulate the effect of a braking tyre. Tests on the seal strength were carried out at low temperatures (approximately 0 °C) and high temperatures (up to 60 °C) using the Iranian and the current Safaniya bitumens.

The failure temperature was found to be related to the penetration at low temperatures and viscosity at high temperatures for both bitumens. The Iranian bitumen failed at temperatures approximately 4 °C above the current Safaniya bitumen at low temperatures, and at 5 °C below the Safaniya bitumen at higher temperatures.

The field performance of the bitumen was assessed through the construction of comparative trials on roads in Canterbury, New Zealand. A range of bitumen application rates were used with both bitumens. With the Iranian bitumen, two kerosene contents were used - the same as used with the Safaniya (3 pph), and the other to obtain a similar 60 °C-viscosity (1 pph).

The field trial was monitored for a year (1995-96) and, although the sections with the normal design application rate all performed well, the Heavy Iranian bitumen at low application rates (20% below design) and 1 pph kerosene lost chip at the beginning of winter. The control Safaniya bitumen at the same application rate performed well.

Stage 2 Conclusion
This investigation has shown that chipseal performance could be affected by the use of a more temperature-sensitive bitumen in that there is an increased risk of chip loss in the first winter, and of chip rollover during periods of high pavement temperatures.

ABSTRACT

Refining practice for bitumens in New Zealand has been reviewed over the years, and a range of bitumens of different temperature sensitivities may become available for roadmaking in New Zealand. Some of these bitumens may not be able to meet the requirements of the Transit New Zealand Specification for asphaltic bitumens, TNZ M/1:1995. Economic benefits may be possible if the requirements of TNZ M/1:1995 could be relaxed to permit a more temperature-sensitive bitumen.

Because of New Zealand's investment in chipsealing, an assessment of the sensitivity of chipseal performance to changes in the rheology (flow properties) of the binder was made. The bitumens studied between 1993 and 1996 were a 180/200 bitumen that is slightly outside the TNZ M/1:1995 specification and a 180/200 Heavy Iranian, and they were compared with a normal Safaniya bitumen used in chipseals. Both laboratory tests and field trials were performed to determine if the early life (first year) of chipseals constructed using these bitumens was significantly different from the Safaniya bitumen currently used.

The results show that chipseal performance could be affected using a more temperature-sensitive bitumen with an increased risk of chip loss in the first winter, and of chip rollover during periods of high pavement temperatures.

Stage 1
INITIAL EXPERIMENTAL TESTS

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1. INTRODUCTION

Expected changes in the availability of different crude oils, and the limited lifetime of the section of plant that is currently producing bitumen at the NZ Refining Company (NZRC), Marsden Point, near Whangarei, mean that the properties of the bitumens used on New Zealand roads over the last 25 years or so may change significantly. A range of bitumens of different temperature sensitivities is likely to be available, some of which may not be able to meet the requirements of the Transit New Zealand (TNZ) Specification for asphaltic bitumens, TNZ M/1:1995.

Most road bitumen available in New Zealand is used in chipseal surfacings but the effect of allowing a wider range of temperature sensitivities in them is not clear. The design for these chipseals is based on evolved experience with bitumens whose properties, when just manufactured, have varied little over decades, rather than on any precise understanding of the relationships between bitumen properties and seal performance. Experience of the effects that might occur with bitumen that is softer at high temperatures and harder at low temperatures is limited and, given the investment in chipsealing, a better understanding of these relationships is needed.

At low temperatures two phenomena have to be considered:

1. Chip loss during the first winter after sealing can occur if there has been insufficient time before winter for enough traffic compaction to have made the seal stable.
2. Long-term seal failure associated with oxidative hardening of the bitumen.

At high temperatures, the associated reduced cohesive strength of the binder can result in early chip loss under traffic stresses. The effect on a chipseal's performance of a reduction in binder cohesive strength is not clear, as other factors contribute to seal stability such as chip size, degree of compaction, substrate texture, seal design (single or two coat), and spray rate. The relative importance of these factors, and in particular the sensitivity of seal stability to binder strength (which correlates closely with viscosity), can be examined by field trials and by comparative tests on artificial seals prepared in the laboratory.

In mid 1993, plans were drawn up for field trials to examine the results of sealing with bitumen of increased temperature sensitivity. A 180/200 bitumen that was temperature-sensitive was to be manufactured by the NZRC at Marsden Point. Observations and testing were carried out to assess initial chip adhesion, initial seal strength against trafficking, chip retention during the first winter, and macrotexture change over the first year.

However, the physical properties of that bitumen did not differ significantly from those of the normal 180/200 bitumens that are used on New Zealand roads. Consequently, to meet the urgent need to provide guidelines for choosing bitumens, a laboratory investigation was carried out in 1994 of the effect of temperature sensitivity on seal performance, using the current 180/200 bitumen stock. The results of the testing could be applied to bitumens of various temperature sensitivities by noting bitumen viscosity and penetration values at the test temperatures.

This Stage 1 of the report describes the development, in 1993-94, of a laboratory test methodology, and the results of initial tests on a trial bitumen produced by NZRC, and comparing them with the properties of a normal 180/200 Safaniya bitumen.

It also includes, as Appendices, the results of additional laboratory tests that go beyond the TNZ M/1 requirements, carried out on the initial trial bitumen produced by NZRC (Appendix 1), and on a 180/200 trial bitumen produced from Heavy Iranian crude oil (Appendix 2). These bitumens are compared with a normal Safaniya bitumen. These extra tests were performed to check that chipsealing would not be adversely affected if these trial bitumens were used.

In the event, the initial trial bitumen was found to be similar to the Safaniya bitumen, and it was not used in the further trials of Stage 2 concerning temperature-sensitivity. The trial Heavy Iranian bitumen was however sufficiently different to warrant further investigation and field trials, to assess responses to low and high temperatures. The results of these trials are recorded in Stage 2 of this report.

2. INVESTIGATION OF FACTORS AFFECTING CHIPSEAL STABILITY

2.1 Experimental Approach

The proposed approach to this investigation was to use the pendulum adhesion/cohesion impact tester and chipseal surface stability tester, developed at Opus Central Laboratories, Lower Hutt, in 1992.

This tester is a modified Avery hardness testing machine (Figure 2.1). It had been modified to fulfil two tasks: as an adhesion/cohesion impact tester, and as a chipseal surface stability tester, described below.

2.1.1 Adhesion/Cohesion Impact Test (Figure 2.2)

This is a development of the adhesion test equipment developed by DKH Briggs (Briggs & Croft 1970, British Carbonisation Research Association 1975).

In the original method a 25 mm-thick steel plate is coated on one side with a layer of bituminous binder. One hundred stones are pressed into the binder surface by a standardised method, and the plate is held at the test temperature for an hour. It is then inverted, slid onto guiding rods, and pushed against a steel bar attached by pre-tensioned springs to the test frame.

Figure 2.1 Impact tester used for both adhesion/cohesion impact and chipseal surface stability tests.

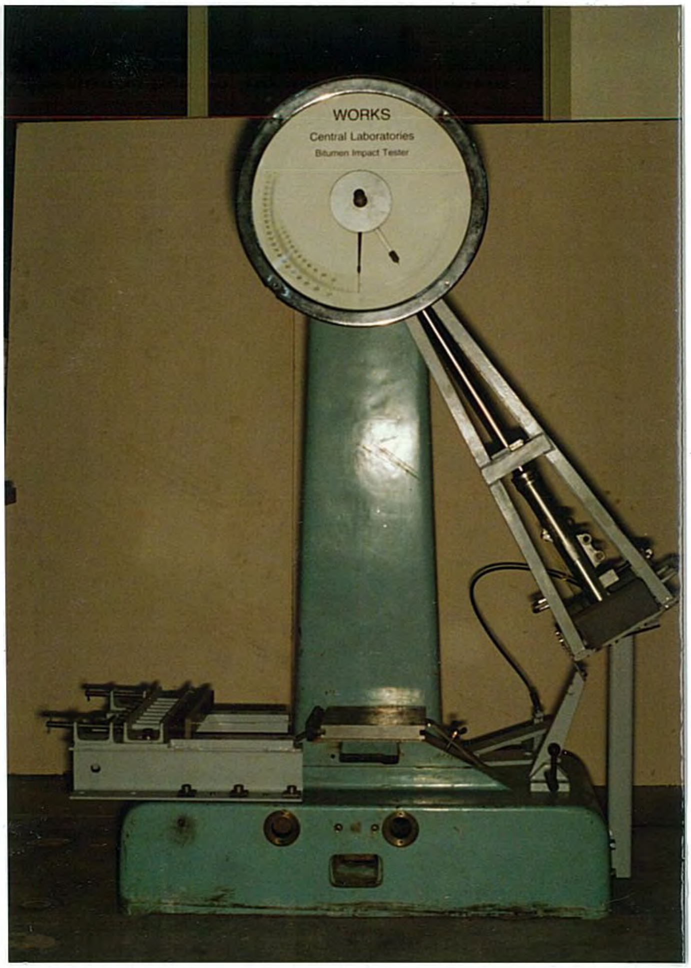
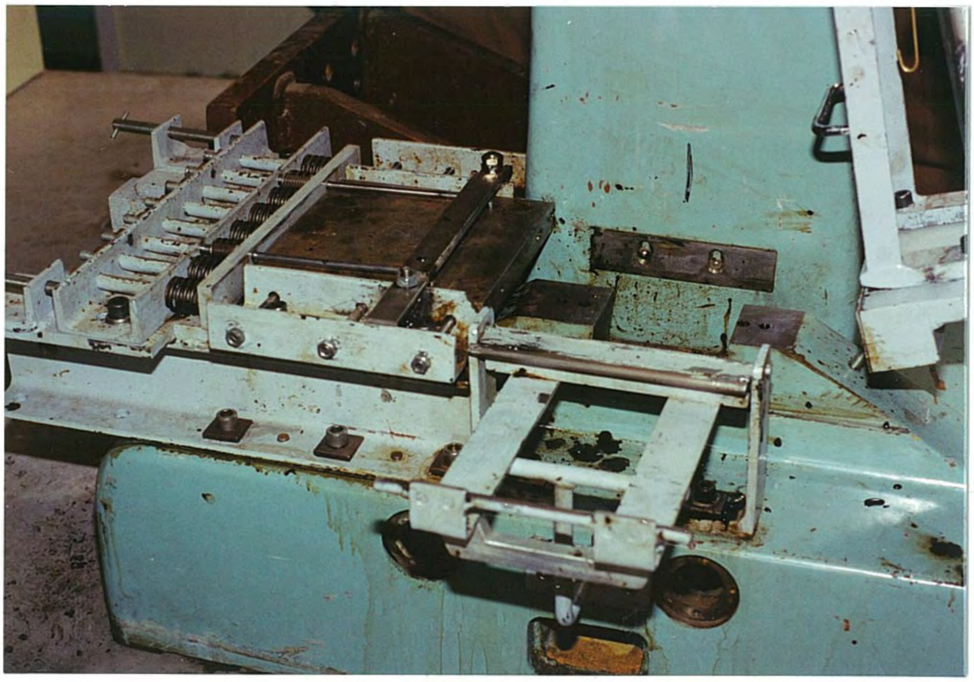


Figure 2.2 Adhesion/cohesion impact test equipment.



The test consists of a single impact by a standardised pendulum on the side of the plate opposite the bar, with the percentage of stones being retained taken as a measure of the adhesion of binder to stone. By controlling the spring tension and having a side-on impact, Briggs & Croft sought to reproduce the duration and direction of traffic stresses causing chipseal stripping.

Briggs & Croft standardised their test further by:

- placing the stones in the binder at the same temperature as planned for the test;
- fixing upon a standardised binder thickness (1 mm).

By doing this they were able to pass from 100% adhesion to almost zero adhesion over a very small change of test temperature, and so define a "critical adhesion temperature" for 50% chip loss within ±1 °C. The actual critical temperature varied with the binder type, but generally corresponded to a binder viscosity of around 10,000 Pa.s for clean chip. The researchers concluded that chip wetting should not occur at all if the binder viscosity was above a critical value.

Use of this type of apparatus was planned to study chip loss occurring at low temperatures. Tests would be carried out for different chip sizes and different binder thicknesses (simulating the effect of different amounts of traffic compaction by the start of winter). It was hoped that, because the thick test plate and side-on impact of the test apparatus results in all test chips experiencing very similar shearing forces, the temperatures at which chip loss occurred might be defined to within a few degrees Celsius.

The starting point for the work recorded in this Stage 1 was the study by Houghton & Hallett (1987). This suggested that stripping of chip from single coat seals during the first winter after construction is not likely to occur if, by the time winter conditions set in, trafficking has compacted the seal to the point at which at least 35% of the total volume of voids is filled by binder.

Two grades of chip were selected and impressed at 40 °C onto test plates (thickness 25 mm, surface 198 x 204 mm) coated with 180/200 bitumen binder of different thicknesses corresponding to different degrees of compaction. After a period in an oven at 40 °C to allow good bonding to the chips, the plates were cooled to test temperatures and tested on the impact test apparatus.

A circular scale with uniform divisions (10.8 °) was used to set the pendulum position so that a standard impact could be applied to the side of the test plates. The test procedure was standardised, aiming at approximately 50% chip loss around 0 °C for grade 2 chip and a binder of thickness around 25% of the chip ALD[1]. Three blows from a pendulum scale setting of 6 (64.8 ° from the vertical) were found to give the desired result.

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[1] ALD Average Least Dimension (mm) of chip

2.1.2 Chipseal Surface Stability Test (Figures 2.3, 2.4)

For this test the apparatus was modified to simulate the effect of a tyre skidding on a chipseal surface to induce chip rollover at relatively high temperatures. Model seals were produced by rolling chip into a bituminous binder coating one side of a steel plate, 6 mm thick, 400 mm long and 280 mm wide. A motorcycle front-fork leg was incorporated in the original pendulum, with a pivoting steel foot attached to the lower end. The steel foot had a rubber pad (75 mm wide, 100 mm long) bonded onto the base. In the test the rubber was dragged across the surface of the test chipseal by the momentum of the released pendulum.

Figure 2.3 Chipseal surface stability test equipment.

The fork spring within the fork leg was pre-loaded, the aims of this being:

- the slight upward movement of the pad as it moved across the seal surface to produce a relatively small change in the downward force on the seal, making the dislodging drag on the stones effectively constant;
- the downward force on the seals to be of the same order as that experienced by chips on the road.

Under standardised testing conditions the seal test temperature was progressively increased until significant chip rollover occurred.

2.1.3 Details of Equipment

Details of the two pieces of equipment (seal compaction and surface stability test equipment) used are as follows.

1. **Seal compaction equipment**. The complete apparatus is shown in Figure 2.4. The wheel is weighted to simulate the effect of a loaded car tyre, and is driven back and forth over two test plates (400 x 280 mm each) via a pneumatic cylinder. This is preferred to a crank attached to an electric motor because it is much more compact given the large stroke (1 m) involved and because it gives a more uniform motion.

   The plates themselves are driven slowly back and forth at right angles to the wheel motion, so that the full plate is rolled, and a slight twisting action on the chip assists compaction. Rates of traverse are approximately 0.4 m/s (1.44 km/h) for the wheel and 0.5 mm/s for the transverse plate motion.

   Bitumen (180/200) coverage of the test seals is calculated by the TNZ *Bituminous Sealing Manual* (1993) method for a zero texture surface carrying 1000 vehicles per day.

   Chip quantities were set at a coverage of 995/ALD m^2/m^3. This is a lower rate of spread than the approximately 725/ALD m^2/m^3 recommended by the TNZ *Bituminous Sealing Manual*. For the higher chipping rate, no more stone was incorporated into the seal, and similar ultimate texture depths were obtained.

   Degree of seal compaction was measured using standard sand circle sand (TNZ Test Method T/3:1981). Details of the seals are listed in Table 2.1.

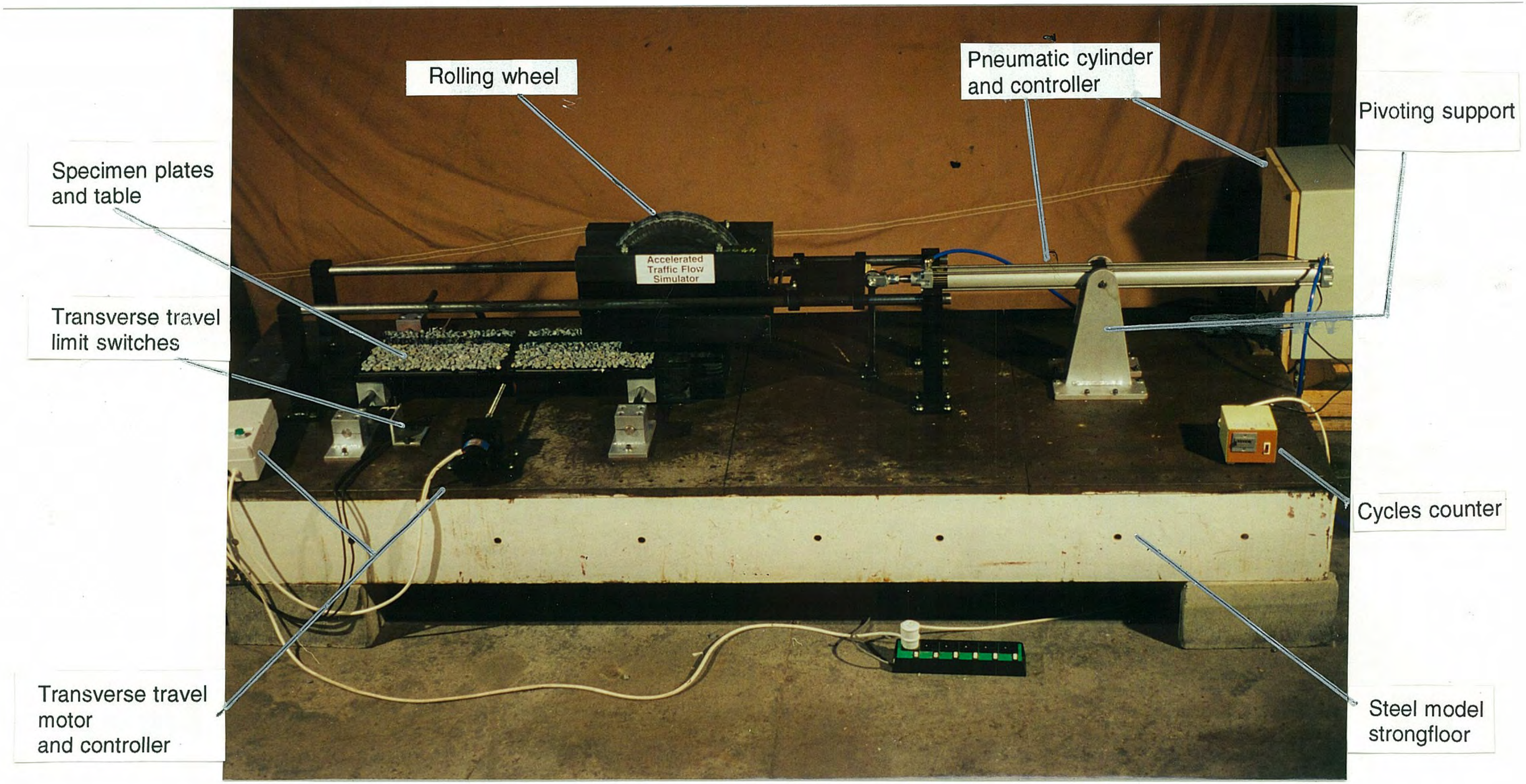
Table 2.1 Details of pre-test settings.

Materials: Binder - 180/200 Safaniya bitumen (ex NZRC)
Chip - greywacke (ex Winstone's, Lower Hutt)
Test Plate Dimensions: 240 x 400 mm

Property	Grade 3 chip	Grade 5 chip
ALD	8.66 mm	5.04 mm
Bulk density (air dry)	1400 $kg.m^{-3}$	1390 $kg.m^{-3}$
Binder coverage	1.30 lm^{-2} (149.1 g)	0.755 lm^{-2} (149.1 g)
Aggregate coverage	1.364 kg	0.818 kg

Before testing,
Grade 3 chip was sieved to pass a 13.2 mm sieve and be retained on a 9.5 mm sieve.
Grade 5 chip was sieved to pass a 9.5 mm sieve and be retained on a 4.75 mm sieve.

Figure 2.4 Seal compaction test equipment.



2. Surface stability test equipment

Pendulum deflection is measured on the circular scale as for the adhesion/cohesion impact test. The pendulum is weighted to increase kinetic energy and thus allow the pad to fully traverse the seal test area. Spring pre-tensioning is 400 N; deflection of the rubber pad increases it to 511 N for the first millimetre, after which spring response is linear with a spring constant of 67 N/mm. Thus typical downward forces on the trial chipseals are of the order of 450 N, and of the order of those that may be experienced under the inside wheels of a cornering car.

The degree of contact of the rubber pad with the chipseal surface was standardised using a grade 5 chipseal with an epoxy binder. Contact was adjusted until the pendulum, when dropped from a scale reading 4 (43.2° from the vertical), lost half its energy.

A flat aluminium plate was then placed on the seal surface and marked to indicate the position where the rubber pad leading edge just tracked in its downward swing. This marked plate was then placed on any seal to be tested, and the equipment adjusted to give the standard degree of contact of rubber pad and seal surface.

3. ADHESION/COHESION IMPACT TEST RESULTS

3.1 General Observations

In all cases chip which had fallen off the plates during testing had their detached surfaces covered in bitumen. Thus detachment was always caused by cohesive failure rather than failure at the aggregate/binder interface, as found by Briggs & Croft. Results are therefore independent of the source of the test aggregate, and controlling the aggregate–binder bond strength by accurately standardising the time in the 40 °C oven during which chip/bitumen adhesion takes place is not necessary.

As the test temperature is altered, results for retained chip percentages do not change nearly as rapidly as for Briggs & Crofts' work, where the change from 100% to zero adhesion usually occurred over a 2° to 3 °C temperature range. It was therefore necessary to define the detachment temperature as that at which a set percentage of chips are lost.

3.2 Test Parameters

In all cases the test binder used was 180/200 Safaniya bitumen, with penetration values of 180 dmm and 18 dmm at 25°C and 5°C respectively. The plot of log (penetration) versus temperature is closely linear below 25 °C for this bitumen, so that these data can be used to calibrate test results against binder penetration value.

Chipseal thicknesses on the road are typically of the order of 1.1 x ALD (Potter & Church 1976, Dickinson 1990). The relationship of filled void content versus binder height is not linear, but depends on both the shape of the chip and the closeness of chip packing in the particular seal. In view of this variation cohesion was initially measured as a function of binder height up the stone rather than void content. (If the chips are approximately cubic the relationship of binder height to void content will be effectively linear.) Target binder thicknesses were approximately 25, 35 and 45% of 1.1 x ALD. Details of chip sizes and binder thickness for the tests are listed in Table 3.1. All aggregate was greywacke from Winstone's quarry, Lower Hutt.

Table 3.1 Sealing chip and binder thickness for low temperature cohesion impact tests.

Chip grade	ALD (mm)	Binder thickness (mm) (% of 1.1 x ALD)		
2	11.6	2.85 (22.4)	4.50 (35.4)	6.10 (47.9)
5	5.0	1.24 (22.4)	1.96 (35.4)	2.66 (47.9)

Before testing: Grade 2 chip was sieved to pass 16.0 mm and be retained on 9.5 mm; Grade 5 chip was sieved to pass 9.5 mm and be retained on 4.75 mm sieves.

3.3 Test Repeatability

Four plates, each with 81 grade 2 chips and binder thickness 0.224 ALD, were tested at 1 °C. The numbers of retained chips were 38, 37, 31 and 51 respectively. This gives an average chip retention of 48.5% with a standard deviation of 10.4%.

3.4 Variation of Chip Retention with Temperature and Binder Penetration

Retained chip quantities at various temperatures for the two chip sizes and different thicknesses of binder are listed in Tables 3.2 and 3.3. Results are plotted as a function of binder penetration in Figure 3.1.

Table 3.2 Impact test results for grade 2 chip, by % chip retained.

Temperature (°C)	Binder penetration (dmm)	Binder thickness (mm) (% of 1.1 x ALD)		
		22.4	35.4	47.9
		% chip retained		
1	11.4	48	–	–
– 4	6.4	50	55	72
–10	3.2	22	58	84

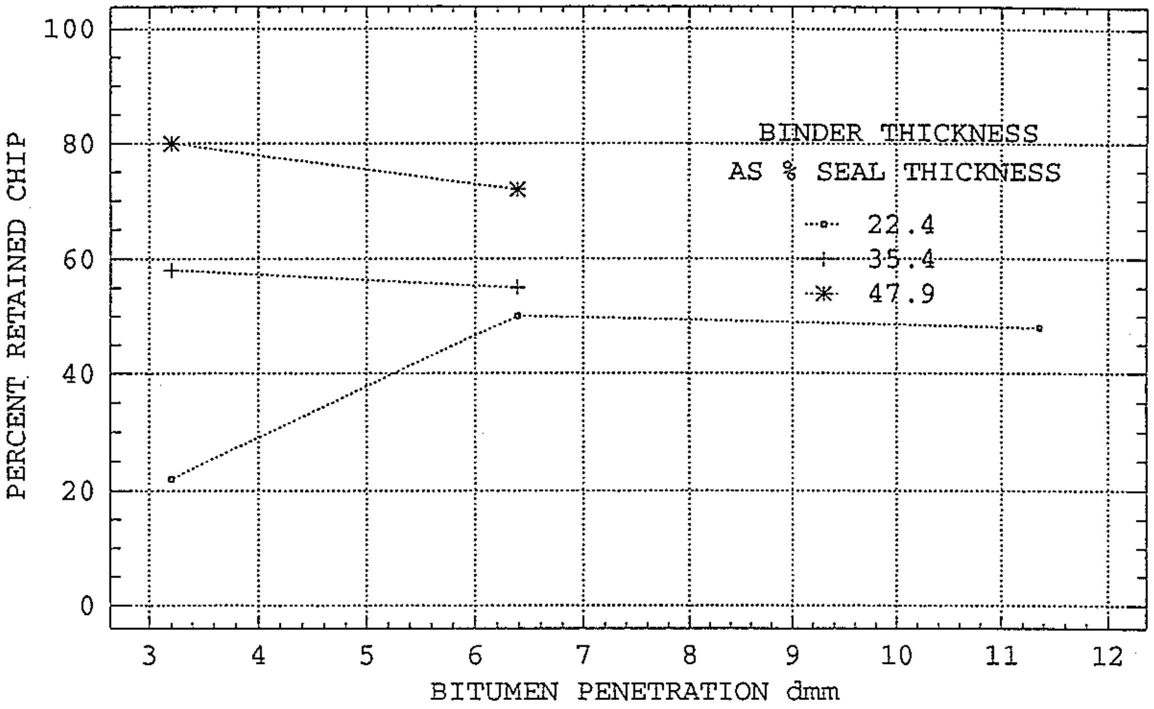
Table 3.3 Impact test results for grade 5 chip, by % chip retained.

Temperature (°C)	Binder penetration (dmm)	Binder thickness (mm) (% of 1.1 x ALD)		
		22.4	35.4	47.9
		% chip retained		
– 5	5.7	–	98	–
–10	3.2	84	91	–
–17	1.4	42	32	74
–24	0.9	2	60	83

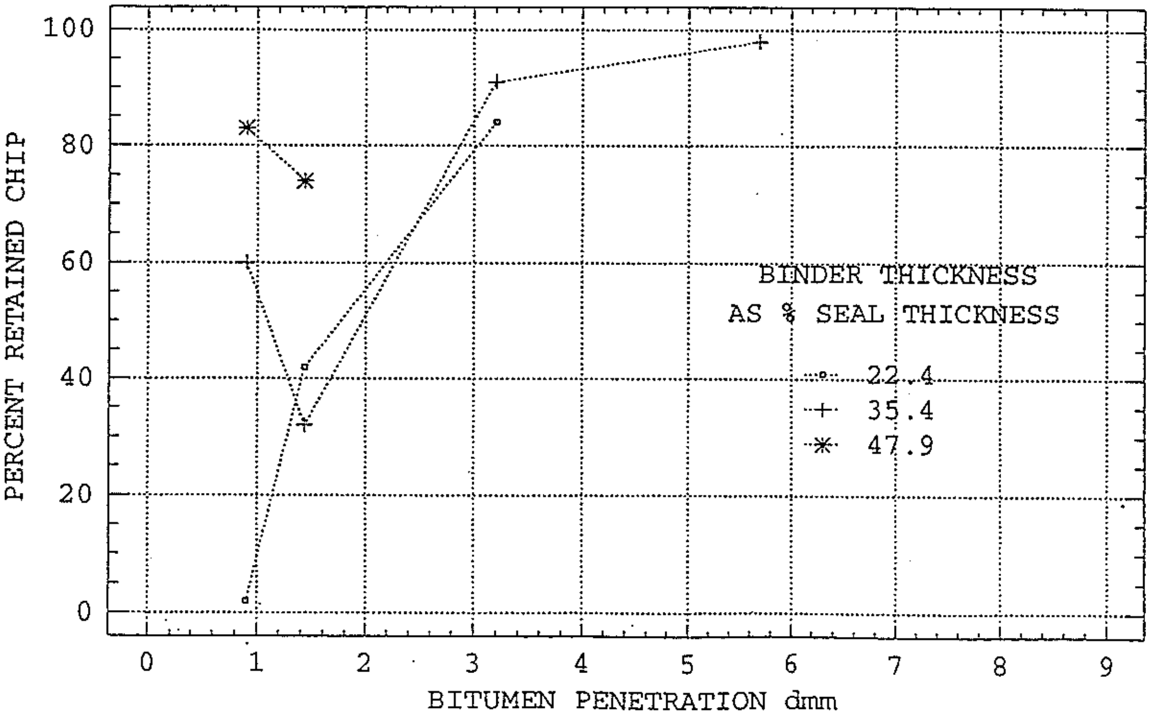
Except for the grade 2 chip result at 1 °C (four test plates), all results are for a single plate and, given the repeatability of the results (Section 3.3 above), repeat testing is needed to establish adhesion values accurately. Certain trends are apparent, however.

Figure 3.1 **Variation of chip retention with binder penetration value, at different temperatures (from adhesion/cohesion impact test results).**

Grade 2 chip at -10°C (left), -4°C, +1°C (right)



Grade 5 chip at -24°C (left), -17°C, -10°C and -5°C (right)



- For a given grade of chip, the greater the binder thickness the greater the chip retention at a given temperature. The only apparent exception is the result for grade 5 chip and maximum binder thickness at -17°C. Given the repeatability of the test, this result needs to be checked by further testing.

- The drop in percent chip retention with either decreasing penetration or temperature is slow at first and then accelerates. If the disbonding mechanism parallels chip loss on roads, this suggests that, for a given compaction, there may be a critical temperature below which stripping occurs.

- Grade 2 chips give lower retained chip percentages than grade 5 for equivalent binder thicknesses (as % of chip ALD).

  If this translates to performance on the road, it indicates that seals with small chip would be less liable to low temperature chip loss in the first winter after sealing than seals with large chips. The difference in critical temperatures may be of the order of 10°C.

4. SURFACE STABILITY TEST RESULTS

4.1 General Observations

4.1.1 Chip Rolling

For each chip grade, seals were compacted to three levels, corresponding to one traverse of the plate (87 back and forth cycles of the wheel, taking approximately 7 minutes) for minimum compaction, a compaction to refusal (taking typically two days), and a medium compaction corresponding to a sand circle texture depth approximately 1.3 times the ultimately attained texture (approximately 30 minutes).

The effectiveness of the rolling depends on the ambient temperature, and so a fixed number of traverses to obtain a given level of compaction cannot be defined. Thus repeated sand circle testing is necessary.

4.1.2 Stability Testing

For the testing the chipsealed plate is first placed in the test apparatus and the degree of contact between the chip and the rubber test pad is standardised as described in Section 2, in this Stage 1 of the report.

The plate is then placed in an oven and heated to the test temperature. On removing, it is tested immediately.

If no stone is lost, the plate is replaced in the oven for heating to a higher test temperature. If a few chips are lost, the contact between chip surface and rubber pad is re-adjusted to the previous level (in this case the chips lost have often been protruding, and the adjustment is necessary for contact with the surface to continue), and the pendulum test is performed again. This process is repeated until either no further chip loss occurs, in which case the plate is heated to a higher temperature and re-tested, or catastrophic chip loss occurs, when the test temperature (hereafter called the critical temperature) is recorded. The onset of catastrophic chip loss is sudden and unmistakable, accompanied both by the movement of several adjacent chips (though not necessarily their detachment from binder) to leave a bare patch of bitumen, and also by a significant change in the energy loss of the pendulum.

The testing has been performed in 5°C steps so it could be carried out within a practicable time. The principal constraint on performing a series of measurements is the time taken to heat the test plates.

4.2 Test Parameters

The test binder used was 180/200 Safaniya bitumen with viscosity levels of 57.70 Pa.s and 20.11 Pa.s at 60°C and 70°C respectively. To convert critical test temperatures to equivalent critical binder viscosities these values were extrapolated to lower temperatures using the relationship that Heukelom (1973) derived to construct the Shell bitumen test data chart. The conversion equation is:

$$\log \eta = \frac{46.07}{0.0437076T + 3.8234241} - 5.38606$$

where η is the viscosity in Pascal seconds (Pa.s)
T is the temperature in degrees Celsius

4.3 Test Results

The results are listed in Tables 4.1 and 4.2.

Table 4.1 Grade 3 chipseal surface stability test results.

Rolling category	Seal texture depth (mm)	Texture depth ALD		Critical temperature at seal failure (°C)	
		Individual	Mean by rolling category	Individual	Mean by rolling category
Minimum	3.82	0.441	0.449	45	45
	3.82	0.441		45	
	3.91	0.452		45	
	4.00	0.462		45	
Medium	3.26	0.376	0.370	55	52.5
	3.14	0.363		50	
Maximum	2.63	0.304	0.304	60	62.5*
	2.63	0.304		60+	

* Calculated assuming the last plate would fail at 65°C.

Table 4.2 Grade 5 chipseal surface stability test results.

Rolling category	Seal texture depth (mm)	Texture depth ALD		Critical temperature at seal failure (°C)	
		Individual	Mean by rolling category	Individual	Mean by rolling category
Minimum	3.81	0.756	0.714	40	40
	3.39	0.673		40	
Medium	2.82	0.560	0.533	45	45
	2.55	0.506		45	
Maximum	1.83	0.363	0.377	60	62.5*
	1.97	0.391		60+	

* Calculated assuming the last plate would fail at 65°C.

Figure 4.1 High temperature seal stability testing: critical temperature v texture.

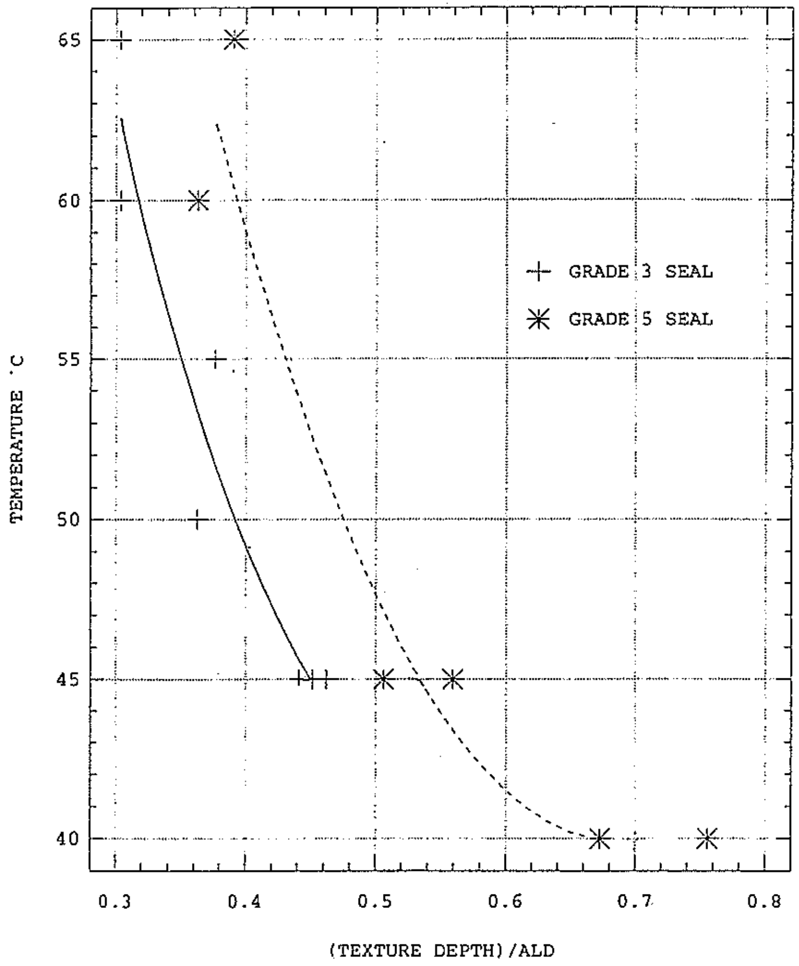
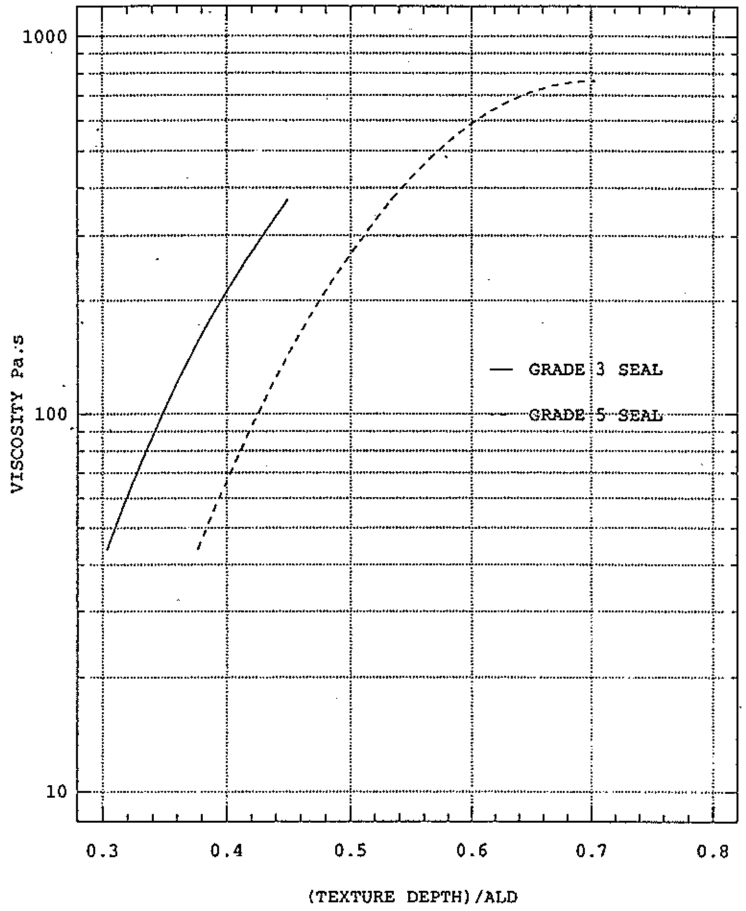


Figure 4.2 High temperature seal stability testing: critical viscosity v texture.



Testing was not continued above 60°C as chipseals on New Zealand roads do not exceed this temperature. Given the observed repeatability of the results (see Tables 4.1 and 4.2, and Section 4.4), the seals which have not failed at 60°C are likely to fail at 65°C. The data have been plotted in Figures 4.1 and 4.2 assuming this to be so.

The critical temperature versus texture depth to ALD ratios are plotted in Figure 4.1. Curves are drawn through the mean values for each rolling category. For a given texture depth/ALD ratio, the critical temperature for a grade 3 seal is about 10°C below that for a grade 5 seal. It is premature, however, to deduce from this that grade 5 seals are significantly more stable against high temperature chip loss than grade 3 seals, as the grade 3 seals proved to be capable of compaction to a greater relative amount (texture depth/ALD ratios for grades 3 and 5 seals compacted to full extent are 0.304 and 0.377 respectively). Thus it is possible that, for equivalent amounts of rolling (rather than attained compaction as measured by the texture depth/ALD ratio), the stability of the two grades of seal may be similar.

The comparative effect of rolling on the two seal types cannot be deduced from the results available, as grade 3 and grade 5 seals were compacted on different days under probably different laboratory temperature regimes. A comparison could be readily carried out, however, by compacting a grade 5 seal and a grade 3 seal simultaneously on the seal compaction equipment, and noting progression of texture depth against time. A plot of the existing critical temperature data versus compaction for the two grades would then give a clear indication of whether or not one seal type is more stable than another.

The curves of Figure 4.1 are re-plotted in Figure 4.2 to indicate the dependence of seal stabilities on binder viscosity.

4.4 Test Repeatability

The data in Tables 4.1 and 4.2 indicate that, for a given seal grade and rolling category, the critical temperatures do not vary by more than 5°C. Since measurements were carried out at 5°C intervals, it is possible that the critical temperature may be able to be defined to a greater accuracy.

5. CONCLUSIONS

Low temperature testing

1. Using the low temperature test as it was set up for this project, significant chip loss only occurs at temperatures lower than those experienced on New Zealand roads. The obvious adjustment to the test would be to increase test impact force. However this practice may be near the safety limit of the equipment.

2. Variability of the test results, as measured for one chip size and one binder thickness at one temperature, is large enough to require multiple testing to be done. Possibly this variability alters significantly with variation of binder thickness and proportion of chip lost. This method is not practicable for routine use.

3. As expected, greater binder thickness gives better chip retention at a lower temperature. The decrease in retention accelerates at lower temperatures, so that an approximate critical temperature where loss becomes significant can sometimes be defined.

4. Larger chips give less retention than smaller chips for equivalent percentage binder rise in the seal.

5. Items (3) and (4) above suggest that first winter sealing chip loss may commence only above a certain binder hardness, dependent on both chip size and degree of compaction.

High temperature testing

6. Repeatability of the high temperature test is good, as the critical temperature above which chip loss becomes important can be determined to within 5 °C, and possibly even to within 3 °C.

7. Less compacted seals have lower critical temperatures and tend to lose chip at lower temperatures.

6. RECOMMENDATIONS

1. Comparison of a set of results for two binders of significantly different low temperature penetrations would be of considerable value. This comparison would determine whether binder penetration is a suitable quantity to define susceptibility to early low temperature chip loss.

2. A comparison should be carried out on the effect of equally rolling seals of different chip size and equivalent binder thickness (as a proportion of ALD). Data from this would allow the determination of whether or not equally rolled seals of different chip sizes are equally stable against chip loss.

3. Repeat testing of binders of different temperature sensitivities are recommended, to determine if the presumed correlation between viscosity and tendency to chip loss with changes in temperature is justified.

4. The test programme should be extended to check the effect of different chip sizes and shapes, and binder spray rates, These factors may result in seals of different stabilities at high temperatures. A field trial would need to incorporate these possibilities, and further laboratory work in this area may be useful before finalising trial details.

Stage 2
FURTHER TESTS & FIELD TRIALS

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1. INTRODUCTION

Since at least the 1950s, bitumen used in New Zealand has been very consistent in terms of its viscosity-temperature relationship. Although bitumen was originally imported and then refined at NZ Refining Company's (NZRC) Marsden Point refinery near Whangarei using a number of crude oils, the different bitumens have been indistinguishable in terms of the standard tests (TNZ Specification for asphaltic bitumens, TNZ M/1:1995) of penetration, softening point, and viscosity at 70°C and 135°C.

The last major change in crude oil used for producing bitumen at Marsden Point was in 1975 when Safaniya crude was introduced.

New Zealand therefore has had no experience with use of bitumens that may be more temperature-sensitive than those currently used, and the chipsealing techniques that have evolved are based on the properties of a very consistent bitumen.

The NZRC has, over a number of years, proposed changes to their refining methods. These have been discussed with Transit New Zealand (TNZ) and the bitumen industry. The NZRC considered that there would be economic benefits if the requirements of TNZ M/1 could be relaxed to permit a more temperature-sensitive bitumen.

The current TNZ M/1 Specification controls the physical properties of bitumen at 25°C through a penetration requirement, and at 70°C and 135°C by a viscosity range. As the "grade" of bitumen is controlled at 25°C, all bitumens used must be of the same consistency at this temperature. The NZRC proposal was to allow a lower viscosity at 70°C. This would result in a more rapid change in consistency with temperature, thus the bitumen would be regarded as more temperature-sensitive.

The effect of this change in temperature sensitivity would be that, at high pavement temperatures (up to 60°C), the bitumen would be softer. Thus the risk would be that chipseals may be more sensitive to chip rollover. At low pavement temperatures (0°C) the bitumen would be harder, thus incurring the risk that chip loss could occur.

This research report covers the assessment of the bitumen produced by NZRC using a Heavy Iranian crude that is slightly outside the current TNZ Specification. In response to NZRC producing this bitumen and as a result of the Stage 1 research, laboratory tests and field trials were carried out, between 1994 and 1996, to determine if the early life (first year) of chipseals constructed using this bitumen would be significantly different from the 180/200 Safaniya bitumen currently used.

2. ASSESSMENT METHODS

A range of techniques were used to compare the properties and performance of a 180/200 penetration grade bitumen produced from the Heavy Iranian crude (Iranian bitumen) and that of the current 180/200 grade Safaniya bitumen used throughout New Zealand on chipsealed roads.

The objectives of the tests were to compare the properties of the bitumens in a chipseal at high pavement temperatures, where the lower viscosity of the Iranian bitumen could lead to increased incidence of chip displacement and rollover under traffic. At low pavement temperatures there was considered to be an increased risk of chip loss when the Iranian bitumen is harder than traditionally used.

The following testing and assessment methods have been used.

1. Standard Physical Tests: these include penetration, softening point and viscosity.

2. Laboratory Simulation: chipseals were constructed in the laboratory using a rolling wheel compactor, and then the two temperatures (low and high) at which chips were displaced, when subjected to an impact designed to simulate a vehicle, was measured.

3. Field Trials: comparative field trials were performed using a range of bitumen application rates to compare the field performance of the Iranian bitumen with that of the Safaniya.

3. COMPARISON OF STANDARD PROPERTIES

A comparison of the properties of the Iranian bitumen with a typical Safaniya bitumen is given in Table 3.1.

Table 3.1 Properties of the two bitumens.

Test	Iranian	Safaniya	TNZ M/1 Limits
Penetration 25°C, 100 g, 5 sec	186	190	180-200
Penetration 5°C, 100 g, 5 sec	13	19	N/A
Softening point, °C	41.2	41.0	–
Viscosity 70°C, mm/sec	12,200	19,000	14,000 min
Viscosity 135°C, mm/sec	186	260	–

These results are illustrated in the bitumen test chart in Figure 3.1, and the low temperature properties in Figure 3.2. At high pavement temperatures the Iranian bitumen has a viscosity at 60°C equivalent to that of Safaniya at 65°C. At low temperatures the reverse occurs in that at 0°C the Iranian bitumen has a penetration value equivalent to the Safaniya at –4°C.

Figure 3.1 Comparison of the properties of the two bitumens (from Heukelom 1973).

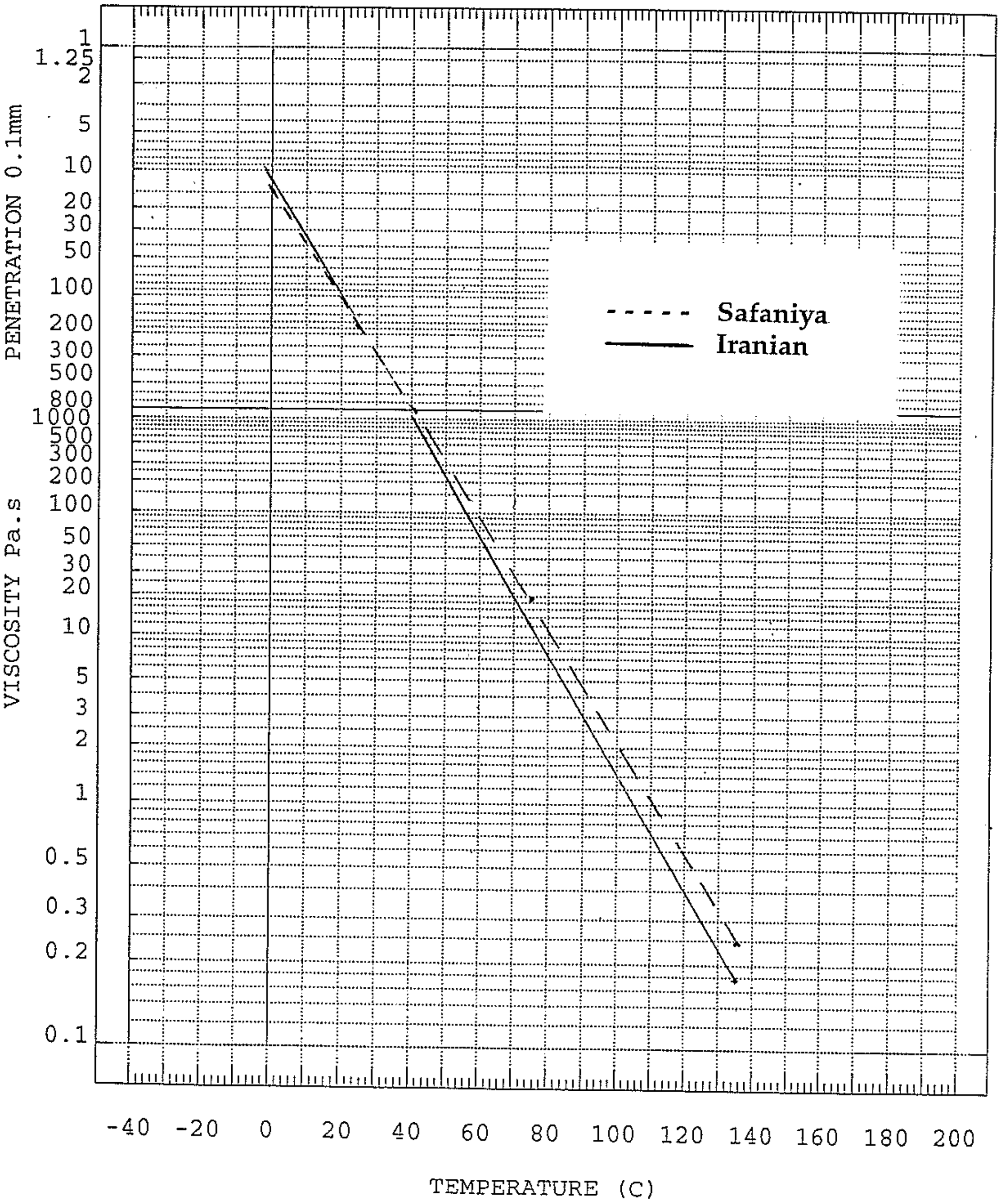
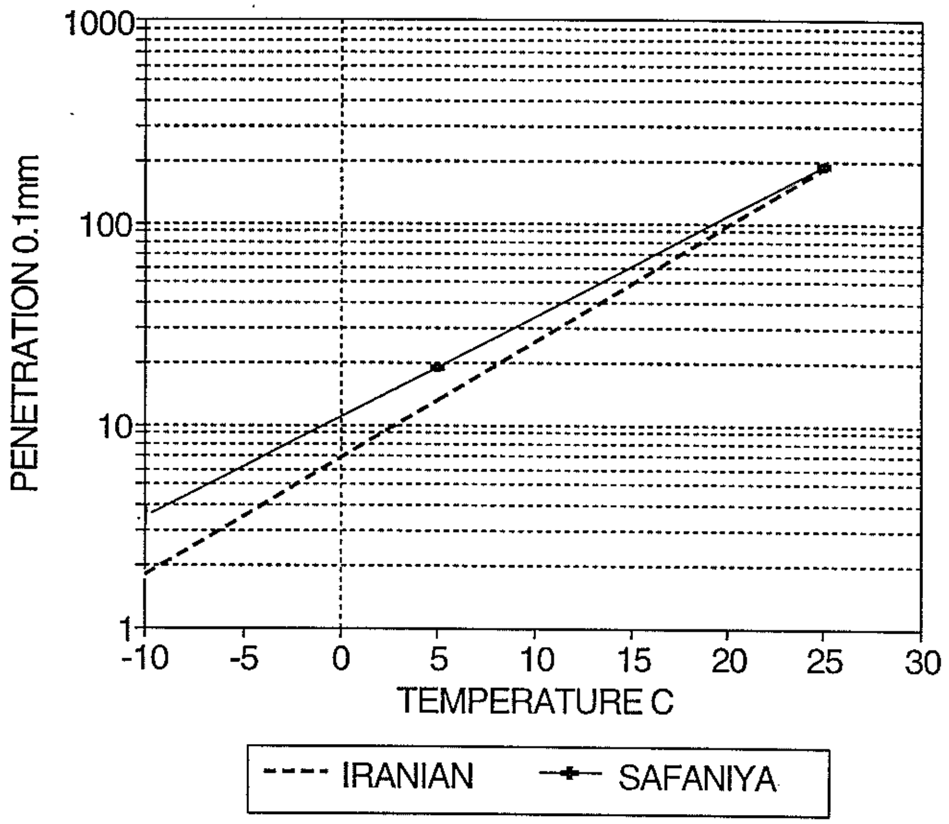


Figure 3.2 Penetration temperature relationship between the two bitumens.



4. LABORATORY SIMULATION

To compare the performance of the two bitumens in a chipseal under laboratory conditions, chipseals using two sizes of chip were compacted on steel plates and then subjected to an impact force at a range of temperatures.

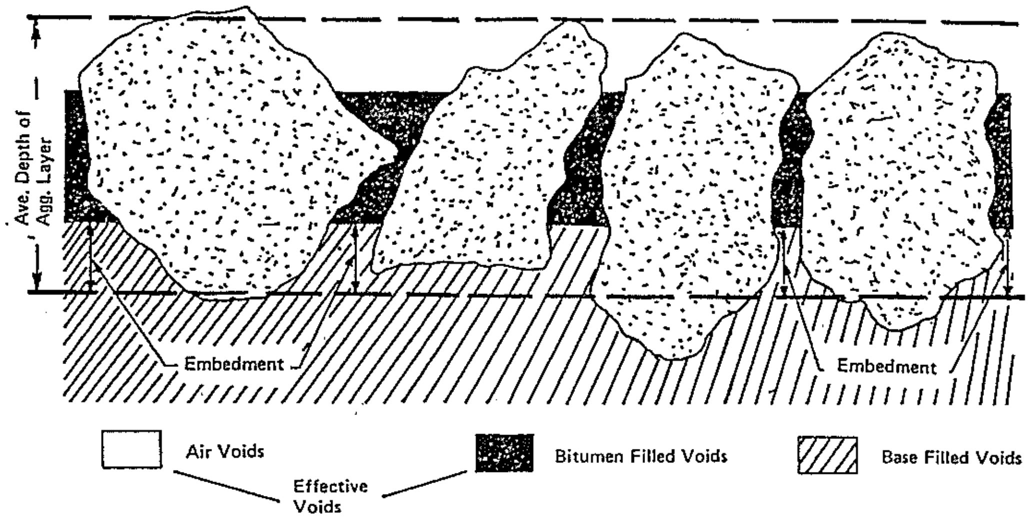
The rationale behind the testing programme was that the strength of a chipseal depends on both the binder properties and the degree of chip interlock. Houghton & Hallett (1987) reported that chip loss at the beginning of winter was liable to occur when less than 35% of the voids were filled with binder in a single coat chipseal. The void analysis used by Houghton & Hallett was based on that of Potter & Church (1976) and is illustrated in Figure 4.1.

When a seal is constructed, the total voids are high and under traffic chip re-orientation takes place and they decrease. As the total voids decrease, the voids filled with bitumen increase. The increased interlock of chip as compaction occurs results in an increase in strength.

4. Laboratory Simulation

The laboratory simulations were designed to assess the relative importance of bitumen properties and chip interlock on the resulting seal strength, both at low pavement temperatures ≈0 °C and at high pavement temperatures ≈60 °C.

Figure 4.1 Illustration of the void concept of a single coat chipseal
(from Potter & Church 1976).



4.1 Test Equipment

The equipment used (Figures 2.1-2.4) was developed for Stage 1 of this project (by Ball et al.), where it is described in detail in Section 2 of this report. However in Stage 2 the impact tester was used at both high and low temperatures.

4.1.1 Compaction Equipment

A treaded pneumatic rubber-tyre rolling apparatus was used to construct the chipseals using steel plates (400 mm x 280 mm) as the substrate.

The equipment (Stage 1, Figure 2.4) consists of a rolling wheel that is moved back and forward over the seal at a rate of 0.4 m/sec. At the same time the steel plate is moved slowly transversely (0.5 mm/sec) so that the total width of the seal is compacted.

4.1.2 Impact Tester

This tester is based on a modified Avery hardness testing machine, as shown in Figures 2.1, 2.2, 2.3, and described in Section 2.1.3, Stage 1 of this report.

4.2 Materials and Test Method

4.2.1 Preparation of Bitumens

Comparative tests were performed using Iranian and Safaniya bitumens and sealing chip of grades 3 and 5 (approximately 16 mm and 10 mm diameter) complying to TNZ M/6:1993, *Specification for sealing chip* (TNZ 1993).

The bitumen and chip application rates used were based on those given in the Transit New Zealand *Bituminous Sealing Manual* (1993) for a traffic volume of 1000 vehicles/lane/day. This resulted in application rates of 0.75 l/m^2 for the grade 5 chip which had an average least dimension (ALD) of 5.04 mm, and 1.30 l/m^2 for the grade 3 chip (ALD 8.66 mm).

The bitumen was weighed onto warmed steel plates so that the bitumen self-levelled and the chip was spread loosely by hand. The plates were then placed in the compaction apparatus and rolled for different times to obtain a range of voids filled.

The void relationship was determined using the sand circle test to determine the volume of air (Figure 4.1) and the bitumen application rate. As the substrate is a steel plate, no embedment could occur. The texture depth (in mm) obtained from the sand circle test is numerically equivalent to volume in l/m^2.

The compacted seals were then brought to the appropriate temperature in a temperature-controlled cabinet. They were then quickly placed in the impact apparatus and impacted. If no distress was obtained, the plates were conditioned at a higher or lower temperature (depending on whether high or low pavement temperatures were being simulated) and then re-tested. Temperature steps of approximately 5 °C were used and the number of chips lost at each temperature recorded. At high temperatures failure was found to be catastrophic, but at low temperatures progressive chip loss occurred as the temperature was decreased. Failure at low temperatures was therefore defined as the temperature at which 15 chips were lost, derived from a plot of chip loss versus temperature.

4.2.2 High Temperature Tests

The test results are given in Table 4.1.

The failure temperatures were converted to viscosity, and the relationship between voids filled and viscosity is illustrated in Figure 4.2 which shows that there is no discernible difference between the bitumens. There is a definite trend of a lower failure viscosity with increasing percentage of voids filled.

The temperature range of the tests was 40-60 °C and, as shown in Figure 3.1, in this range there is approximately 5 °C difference in the viscosity of the two bitumens.

Figure 4.2 Comparison of high temperature stability.

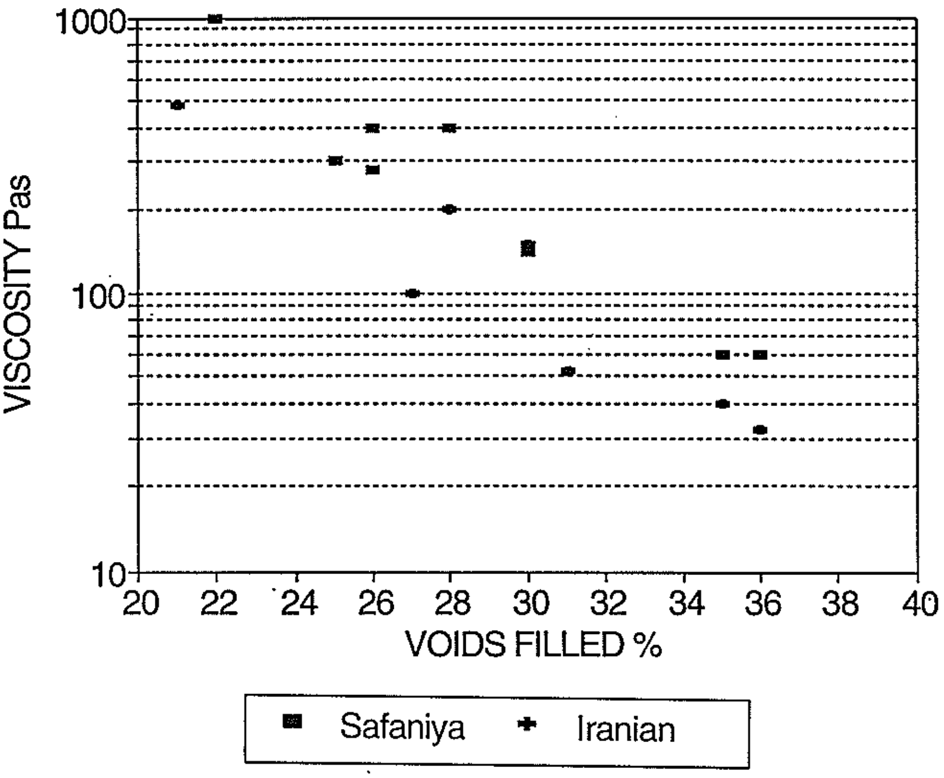


Table 4.1 High temperature strengths of test bitumens.

Voids filled (%)	Temperature (°C) at failure			
	Grade 3 chip		Grade 5 chip	
	Safaniya	Iranian	Safaniya	Iranian
21				40.0
22				
25	46.7		40.0	
26	47.5			
27		50.0	45.0	
28				
30	52.5	47.5	45.0	45.0
31		55.0		
35				
36	>60	>60	60.0	57.5

4.2.3 Low Temperature Tests

Low temperature tests were performed over the range of 10°C to −15°C. The penetration value of the bitumen at the failure temperature was calculated from extrapolation of the results given in Table 3.1.

Figure 4.3 Comparison of low temperature strengths of the bitumens.

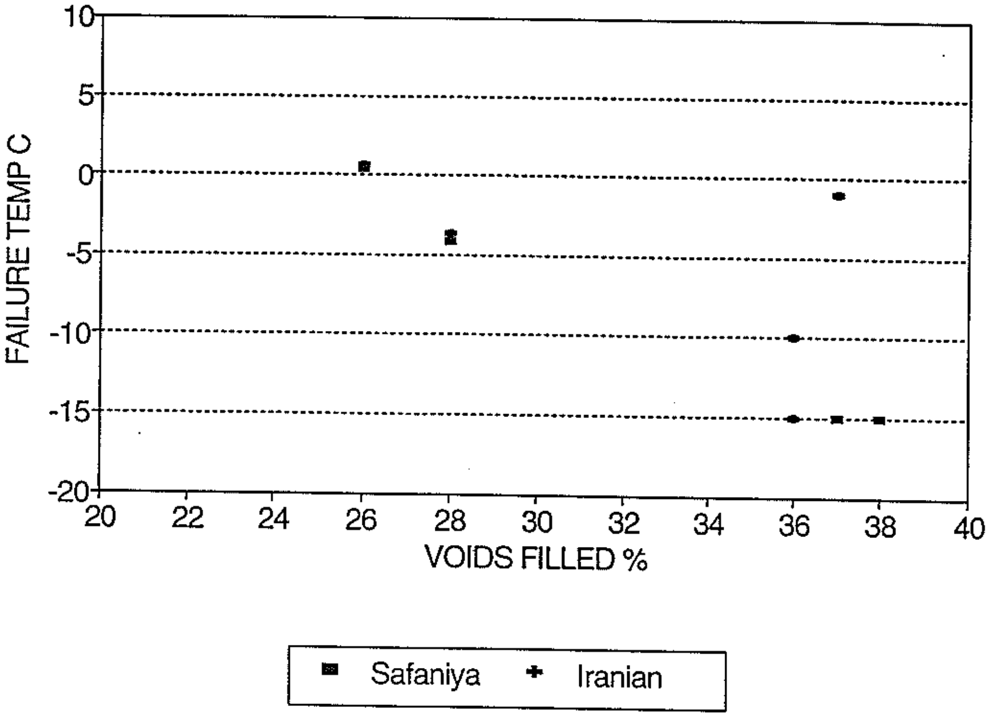
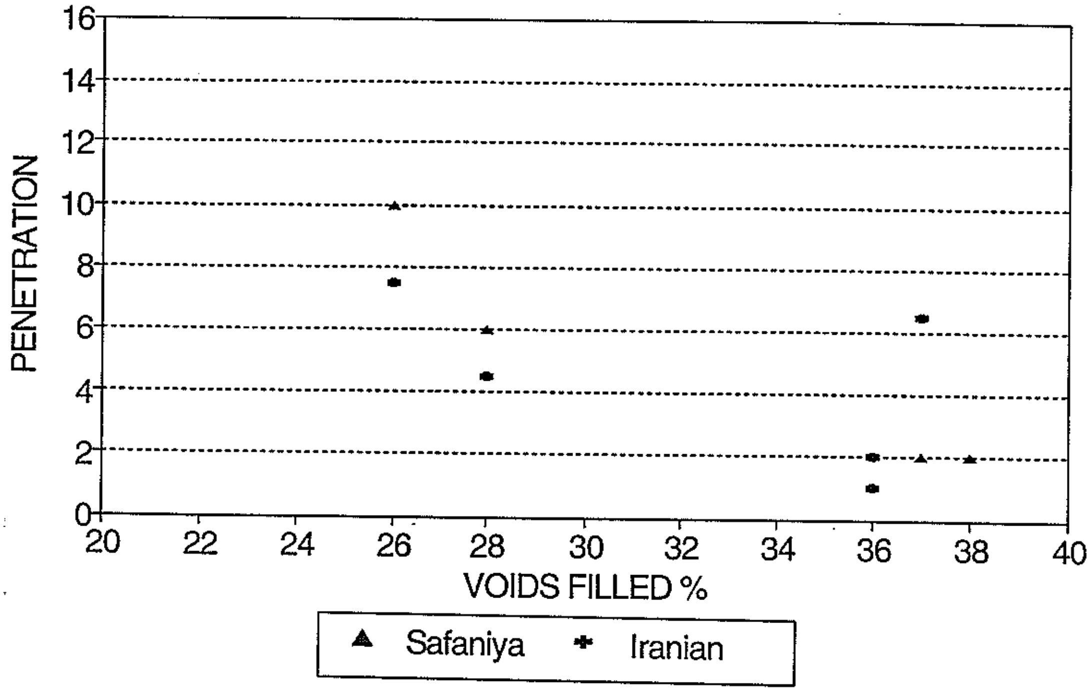


Figure 4.4 Low temperature strength as a function of bitumen penetration.



Failure temperatures obtained are given in Table 4.2 and plotted in terms of temperature and penetration value in Figures 4.3 and 4.4.

Table 4.2 Low temperature strengths of the two bitumens.

Voids filled (%)	Temperature (°C) at failure			
	Grade 3 chip		Grade 5 chip	
	Safaniya	Iranian	Safaniya	Iranian
26	0, 7.5			
28		1.2, –2.5	0, –4.2	–1.5
29		–15.0		–6.0
36				–10.0
37	–15.0			–1.0
38			–15.0	

4.3 Discussion

The laboratory simulation tests used are not very precise in that failure temperatures varied by up to 7.5°C. When the results are plotted, as in Figures 4.2, 4.3, and 4.4, it is clear that bitumen properties and the degree of compaction, as defined by percentage of voids filled, have a significant effect on seal strength.

The test conditions, in terms of the impact force imposed on the test plates, are arbitrary in that, although the force imposed is of similar magnitude to that imposed by traffic, the conditions were selected to obtain failure in the temperature range at which field failure occurs.

The results therefore cannot be applied directly to the field, but it is believed that the trends and magnitude of the changes will reflect field behaviour.

At low temperatures the Iranian bitumen is harder than Safaniya and, in terms of temperature, this equates to a difference of approximately 5°C. For an equivalent degree of trafficking on a chipseal this would mean that a seal constructed with this bitumen would lose chip at a pavement temperature approximately 5°C higher than currently being experienced.

The same trend is evident from the high temperature data in that, at high pavement temperatures, a seal constructed with Iranian bitumen would be expected to exhibit chip rollover problems at a temperature 5°C lower than that currently experienced with Safaniya bitumen.

5. FIELD TRIALS

5.1 Introduction

Comparative seal trials using the Iranian and Safaniya bitumens were performed to determine if the results of the laboratory simulation were reflected in the field situation.

Trials were carried out in Canterbury on a straight section of highway (called Mouse Point) and on a number of curves. The Mouse Point trial used a range of bitumen application rates and kerosene contents.

5.2 Mouse Point Trial

This trial section is on a straight section of State Highway 7, route position 44/1.86 to 44/3.06, with a traffic volume of 800 vehicles/day.

The trial was laid on 25 January 1995 under fine conditions, air temperatures 17°C at 8.30 am, 25°C at noon, and 24°C at 2.30 pm.

The sealing chip was a grade 3 with an ALD of 8.8 mm. As per the normal practice in this area, 1 pph of automotive gas oil (AGO) was used as a flux in the bitumen, with 0.5 pph adhesion agent.

Three binders using 180/200 grade bitumen were used in the trial:

1. Safaniya, with 1 pph AGO plus 3 pph kerosene plus 0.5 pph adhesion agent;
2. Iranian, with 1 pph AGO plus 3 pph kerosene plus 0.5 pph adhesion agent; and
3. Iranian, with 1 pph AGO plus 1 pph kerosene plus 0.5 pph adhesion agent.

The two concentrations of kerosene were used with the Iranian bitumen so that diluent contents equivalent to that used in the Safaniya bitumen (recipe 2) and viscosities equivalent to those occurring at 60°C (recipe 3) could be obtained. This was based on the current sealing practice of determining cutback properties based on the viscosity at 60°C.

The design residual binder application rate of 2.0 l/m^2 was obtained using the methodology in the Transit New Zealand (1993) *Bituminous Sealing Manual.*

The applications used with the various binders are given in Table 5.1.

Table 5.1 Mouse Point trial section.

Section no.	Route position	Bitumen source	Kerosene content (pph)	Target application rate (l/m^2)	Application rate achieved (l/m^2)
1	1.86 - 2.06	S	3	D	1.95
2	2.06 - 2.16	S	3	D - 10%	1.75
3	2.16 - 2.26	S	3	D	1.98
4	2.26 - 2.41	S	3	D - 20%	1.56
5	2.41 - 2.56	I	3	D	2.10
6	2.56 - 2.66	I	3	D - 10%	1.90
7	2.66 - 2.76	I	3	D - 20%	1.70
8	2.76 - 2.86	I	1	D	2.00
9	2.86 - 2.96	I	1	D - 10%	1.80
10	2.96 - 3.06	I	1	D - 20%	1.60

Note: D = design, S = Safaniya, I = Iranian

The above variations from the design application were used to attempt to determine if the Iranian bitumen was more sensitive to variations in binder application rate than Safaniya bitumen.

5.3 Results

Formal inspections were performed on 30 January 1995, 11 February 1995, 27 April 1995 and 25 March 1996. Sand circle tests were performed at selected sites, and a visual inspection of every section was made.

For the first two inspections (5 and 17 days after construction), all the sections were in excellent condition. By April, signs of chip loss were occurring and at the end of May, when an informal inspection was performed, extensive chip loss on section 10 had occurred.

The results of the April 1995 and March 1996 visual inspections are given in Table 5.2, and texture depth measurements are given in Table 5.3.

Table 5.2 Visual assessments of trial section in April 1995 and March 1996.

Section no.	Bitumen source	Kerosene content (pph)	Application rate (l/m^2)	Inspection on 27 April 1995	Inspection on 25 March 1996
1	S	3	D	Excellent condition. Small amount of chip loss on centreline.	Very good condition. Minor chip loss on centreline and between wheeltracks.
2	S	3	D - 10%	Excellent condition. As for site 1.	Similar condition to site 1.
3	S	3	D	Very good condition. Only slight chip loss on centreline.	Similar condition to sites 1 and 2, but with more chip loss on the centreline and minor chip loss between wheelpaths.
4	S	3	D - 20%	As for sites 1 and 2.	As for sites 1 and 2.
5	I	3	D	As for site 1, including small amount of chip loss on centreline.	Similar condition to site 1.
6	I	3	D - 10%	As for site 1, including small amount of chip loss on centreline.	Similar condition to site 1.
7	I	3	D - 20%	Similar to site 3.	Very good condition, but with minor chip loss on centreline and between wheeltracks.
8	I	1	D	Excellent condition.	Chip loss on the centreline and between wheeltracks.
9	I	1	D - 10%	Excellent condition.	More noticeable chip loss on centreline and between wheelpaths.
10	I	1	D - 20%	Chip loss occurring on the centreline.	Extensive chip loss throughout the section.

Note: D = design, S = Safaniya, I = Iranian

Table 5.3 Texture depth and % voids filled, in test sections of Mouse Point trial.

Section no.	Bitumen source	Kerosene content (pph)	Application rate (l/m^2)		Texture depth (mm)							
					30 January 1995		11 February 1995		27 April 1995		25 March 1996	
			Target	Achieved	Wheeltrack	Centreline	Wheeltrack	Centreline	Wheeltrack	Centreline	Wheeltrack	Centreline
1	S	3	D	1.95	2.55	3.39	2.84	3.29	2.23	2.92	2.23	5.19
2	S	3	D - 10%	1.75							2.54	4.73
3	S	3	D	1.98							3.94	2.55
4	S	3	D - 20%	1.56							3.14	4.73
5	I	3	D	2.10	2.29	3.39	2.10	2.72	2.05	3.14	2.55	4.00
6	I	3	D - 10%	1.90							3.14	4.73
7	I	3	D - 20%	1.70							3.14	3.97
8	I	1	D	2.00	2.48	3.14	2.92	3.66	1.98	3.39	2.92	4.73
9	I	1	D - 10%	1.80							2.55	–
10	I	1	D - 20%	1.60	2.54	3.55					2.55	–

Note: D = design; S = Safaniya; I = Iranian

5.4 Braking Tests

During the first three inspections an attempt was made to assess the seal strength by conducting locked wheel braking tests over a range of speeds from 10 to 30 km/h. The tests were to determine if a difference in performance could be determined from the braking speed at which seal distress occurred. Tests were performed on the sections with the design application rate, i.e. road sections 1, 3, 5 and 8.

The pavement temperature for the February test was 28 °C, and for the April test it was 23 °C.

For all tests no discernible difference in the performance of the seals was observed. Below 20 km/h, no chip rollover was observed; at 25 km/h some minor chip movement occurred. There appeared to be less movement in the April tests compared with those performed in February, but no conclusive results were obtained.

5.5 Discussion

This trial, on a straight flat section of highway, has shown that chipseals using Iranian bitumen can perform well, at least over the first year.

The failure of the sections with Iranian chipseals using one part kerosene and low application rates, while the control section with Safaniya had only minor chip loss, does raise a concern over the performance of the bitumen.

As the properties of the Iranian bitumen in the temperature range of 10-40 °C are very similar to those of Safaniya, any difference in performance would be expected to occur outside this temperature range.

On this field trial, when low temperatures occurred at the beginning of winter, chip loss occurred. The ability of a bitumen to hold chip during low temperatures is a function of the binder rise around the chip and its hardness.

The combination of a lower binder application rate and reduced kerosene appear to have resulted in the Iranian bitumen reaching a condition at which failure occurred at lower temperatures.

6. CONCLUSIONS

The three aspects of this research, namely standard physical tests, laboratory simulation, and field trials, have all confirmed that the Iranian bitumen is more temperature-sensitive than the Safaniya-based material that is currently used on New Zealand roads.

The following conclusions have been drawn.

1. At low pavement temperatures (approximately 0°C) the Iranian bitumen is harder than Safaniya by a factor equivalent to a change in temperature of approximately 4°C.

2. At high pavement temperatures (approximately 60°C) the Iranian bitumen is softer than Safaniya by a factor equivalent to a change in temperature of approximately 5°C.

3. The results of laboratory simulations of chipseal strength confirm the results of the physical tests in that failure of chipseals constructed using Iranian bitumen occur at different temperatures from Safaniya chipseals. This is consistent with their differences in physical properties.

4. The results of the field trial indicate that seals constructed with Iranian bitumen may be more sensitive to chip loss at the onset of winter if the cutback content is based on obtaining a 60°C viscosity, which is equivalent to that traditionally used in New Zealand.

APPENDIX 1
AUGUST 1993 TRIAL BITUMEN

A1.1 PRODUCTION

The New Zealand Refining Company (NZRC) produced a bitumen from their new high vacuum distillation unit (HVU2). The aim was to produce a bitumen using a different method, yet was still similar to the 180/200 Safaniya bitumen normally used for chipsealing on New Zealand roads. In the event, it was similar to other bitumens produced by NZRC, and it was not used for further testing in this assessment of temperature-sensitive bitumens.

The short residue from this unit had an approximately 130 dmm penetration, and so was mixed with heavy waxy distillate from the new HVU2 unit and re-distilled through the old HVU1 unit to bring it to about 180/200 penetration grade.

Composition of the long residue feed and short residue product is reported by the NZRC as follows:

Source	**Long residue HVU2 feed (%)**	**Short residue HVU2 product (130 pen) (%)**
Low sulphur crude	11.26	3.21
Arabian Light	59.46	59.37
Arabian Heavy (Safaniya)	16.48	25.65
Various Middle East	12.79	11.77

A1.2 LABORATORY TESTING PROGRAMME

The testing programme for the NZRC bitumen was as follows:

- full test to TNZ M/1;
- comparative adhesion tests (Vialit test equipment);
- effect of kerosene and diesel on 60°C viscosity;
- chemical characterisation.

A1.3 FULL TESTING TO TNZ M/1 : 1989

Results are listed in Table A1.1.
In most cases duplicate results are provided, one from NZRC and the other obtained by Opus Central Laboratories (OCL). Data additional to TNZ M/1 requirements, namely specific gravity and 5°C penetration figures, are also given. For comparative purposes, typical data for Safaniya 180/200 bitumens are listed also.

The trial bitumen is slightly less acid and a little harder at low temperatures (5°C) than Safaniya bitumen, but apart from this there is little difference between results.

Table A1.1 Tests to TNZ M/1 : 1989 for a range of bitumens.

Test	NZRC	OCL	Typical Safaniya	TNZ M/1 Spec/n
Penetration, 25 °C (dmm)	174	177	183	180-200
Softening point (°C)	39.3	39.6	39.8	37-43
Ductility, 25 °C (cm)	>100	>100	>100	100 min
Flash point (°C)	325	324	312	218 min
Viscosity, 70 °C (cSt)	18470	18000	19000	14000 min
Viscosity, 135 °C (cSt)	221	230	248	140-350
Solubility (%)	99.8	99.99	99.89	99.5 min
Density, 25 °C (kg/l)	1.019	-	1.023	-
RTFO				
% retained penetration	-	56.5	56.8	50 min
ductility, 25 °C (cm)	-	>100	>100	60 min
Penetration, 5 °C	-	15	20	-
Acid number (mgKOH/g sample)	-	0.12	0.25	0.35 max
Durability oven test (days)	-	13.8	13.5	12 min

min - minimum; max - maximum
cSt - centistokes; RTFO - rolling thin film oven test

A1.4 COMPARATIVE ADHESION TESTS

Adhesion tests were performed according to the OCL binder adhesion test (B301-89T), as specified in the TNZ Specification for Adhesion Agents (TNZ M/13:1989).

Comparative tests with the trial bitumen and Safaniya bitumen were carried out simultaneously to minimise the effect of the variability of the test method.

A standard dosage of 0.7 pph (by weight) of Redicote E-9 paste adhesion agent was used in all tests.

Comparisons were performed with grade 3 Rangitikei chip and with grade 3 chip from Winstones' Lower Hutt quarry. Results are listed in Table A1.2.

Table A1.2 Adhesion test (TNZ M/13:1989) results for trial bitumen and Safaniya bitumen.

Sealing chip	Trial bitumen (%)	Safaniya bitumen (%)
Rangitikei	94	98
Winstone's (Lower Hutt)	85	90

Specification TNZ M/13:1989 requires that a sealing chip–bitumen combination must have an adhesion of at least 80% to be acceptable for sealing. The trial bitumen gives lower adhesion levels than the Safaniya bitumen, but the differences are not large and there should be no problems obtaining sufficient adhesion in sealing work.

A1.5 EFFECT OF KEROSENE AND DIESEL ON 60°C VISCOSITY

Kerosene and automotive gas oil (AGO, diesel) are commonly added to bitumen for hot sealing work to bring the binder viscosity to a desired level on the road. The quantities of diluents added are often verified by a 60°C viscosity measurement on the binder. To perform this check, a relationship between binder viscosity and diluent quantities is needed. Results of viscosity measurements carried out on a range of binders are listed in Table A1.3.

Table A1.3 Viscosity (60°C) values for different kerosene and AGO quantities.

Kerosene pph (volume)	AGO pph (volume)	Viscosity (60°C) mm^2/s	
		Measured	Curve fit
0	0	51220	56320
0	4.99	19300	17210
5.00	5.00	5845	6505
7.50	0	11360	10280
14.99	0	2880	2914

These results were fitted by regression procedures to the following equation:

$$\log \log V = \frac{1}{(100 + K + D)} (67.6756 - 0.3751K - 0.3718D)$$

where:

V is the viscosity (mm^2/s) at 60°C of the binder
K is the quantity of kerosene (pph by volume at 15°C)
D is the quantity of AGO (pph by volume at 15°C)
The logarithms are to base 10.

Fitted viscosities for the diluent quantities used in the determination are listed in Table A1.3, alongside the experimental values. The equivalent equation for a typical 180/200 Safaniya bitumen is:

$$\log \log V = \frac{1}{(100 + K + D)} (67.44 - 0.4250K - 0.1971D)$$

Comparing the constants of the two equations indicates that, while kerosene has approximately the same effectiveness in reducing viscosity for both bitumens, AGO (diesel) is far more effective for the trial bitumen than for the Safaniya bitumen. The difference is so pronounced (only just over 50% of diesel used in Safaniya bitumen is required to get the same result in the trial bitumen) that repeat testing to check the result and verify that contamination has not occurred may be advisable.

A1.6 CHEMICAL CHARACTERISATION

A1.6.1 Introduction

The NZRC trial bitumen was compared to Safaniya bitumen using thermogravimetry, gel permeation chromatography, and on the basis of asphaltene (n-heptane) content. Experimental conditions are given in Appendix 3.

A1.6.2 Thermogravimetry

This technique involves heating a small sample of bitumen at a carefully controlled rate until fully combusted. A thermogram of weight versus temperature is obtained. The first differential of this curve (the DTG curve) is calculated to make qualitative evaluation easier. To quantitatively compare the bitumens, the weight loss curves were divided into temperature intervals and the percentage mass lost in each region was calculated. Data for the trial bitumen and typical values (Ball & Herrington 1993) for Safaniya 180/200 bitumen are presented in Table A1.4. DTG curves for the trial bitumen and a typical Safaniya bitumen are given in Figure A1.1.

Table A1.4 Thermogravimetric analysis of the trial bitumens.

Bitumen	Percentage weight lost*			
	200-300°C	300-400°C	400-500°C	500-600°C
NZRC trial bitumen	3.8	19.2	52.8	24.0
Safaniya 180/200**	8.0 ±0.6	20.4 ±1.8	47.6 ±1.8	23.7 ±2.3

* Mean of triplicate measurements.

** Mean ±95% confidence limits for 34 samples taken over the course of a year.

Table A1.4 and Figure A1.1 show the trial bitumen to lose less weight than the equivalent penetration Safaniya product in the 200°–300°C interval, indicative of lower levels of volatile species. This is desirable because bitumens with high levels of volatiles are generally less durable. However, it cannot be inferred from this that the trial bitumen will be more durable than the Safaniya product because it is less volatile as other factors are also involved.

All experiments run under air show numerous non-reproducible sharp spikes and peaks that can occur at any point in the profile, although they usually appear between ~340°– 460°C. This noise is not an instrumental artefact and its origin has been discussed elsewhere (Herrington et al. 1992).

Apart from this region, both curves are qualitatively very similar. Although the trial bitumen is less volatile in the 200°–300°C interval, the onset of weight loss is about the same (250°C) as for the Safaniya.

A1.6.3 Gel Permeation Chromatography

This technique involves separating the bitumen according to the molecular size of its individual components by passing a solution of bitumen through a column of a porous polymer. Large molecules pass through the column more rapidly than smaller ones. A chromatogram is obtained in which the relative amount of a particular species (in terms of a detector response) is plotted against time.

Chromatograms for the trial bitumen and a typical Safaniya product are presented in Figure A1.2. This shows the trial bitumen to have less large molecular size material than the Safaniya. Quantitative measurements are made by measuring the area of 30-second slices taken across the chromatogram, and these results are given in Table A1.5.

Table A1.5 GPC chromatograms for the trial bitumens.

Bitumen	Slice area as a percentage of total Slice no.						
	1	2	3	4	5	6	7
NZRC trial bitumen	0.5	14.8	21.5	22.4	25.3	12.9	2.6
Safaniya 180/200*	2.3 ±0.6	21.6 ±0.6	24.0 ±0.6	22.0 ±0.2	19.8 ±0.4	8.7 ±0.2	1.7 ±0.2

* Mean ±95% confidence limits for five samples taken over the course of a year (Ball & Herrington 1993)

These results confirm the qualitative observations made above, and appear to contradict the thermogravimetric data which intuitively suggests that the less volatile NZRC trial bitumen should contain relatively more large molecular size material than the Safaniya and not less.

Figure A1.1 Thermogravimetric first derivative of weight loss curves for (a) trial bitumen, and (b) Safaniya 180/200 bitumen.

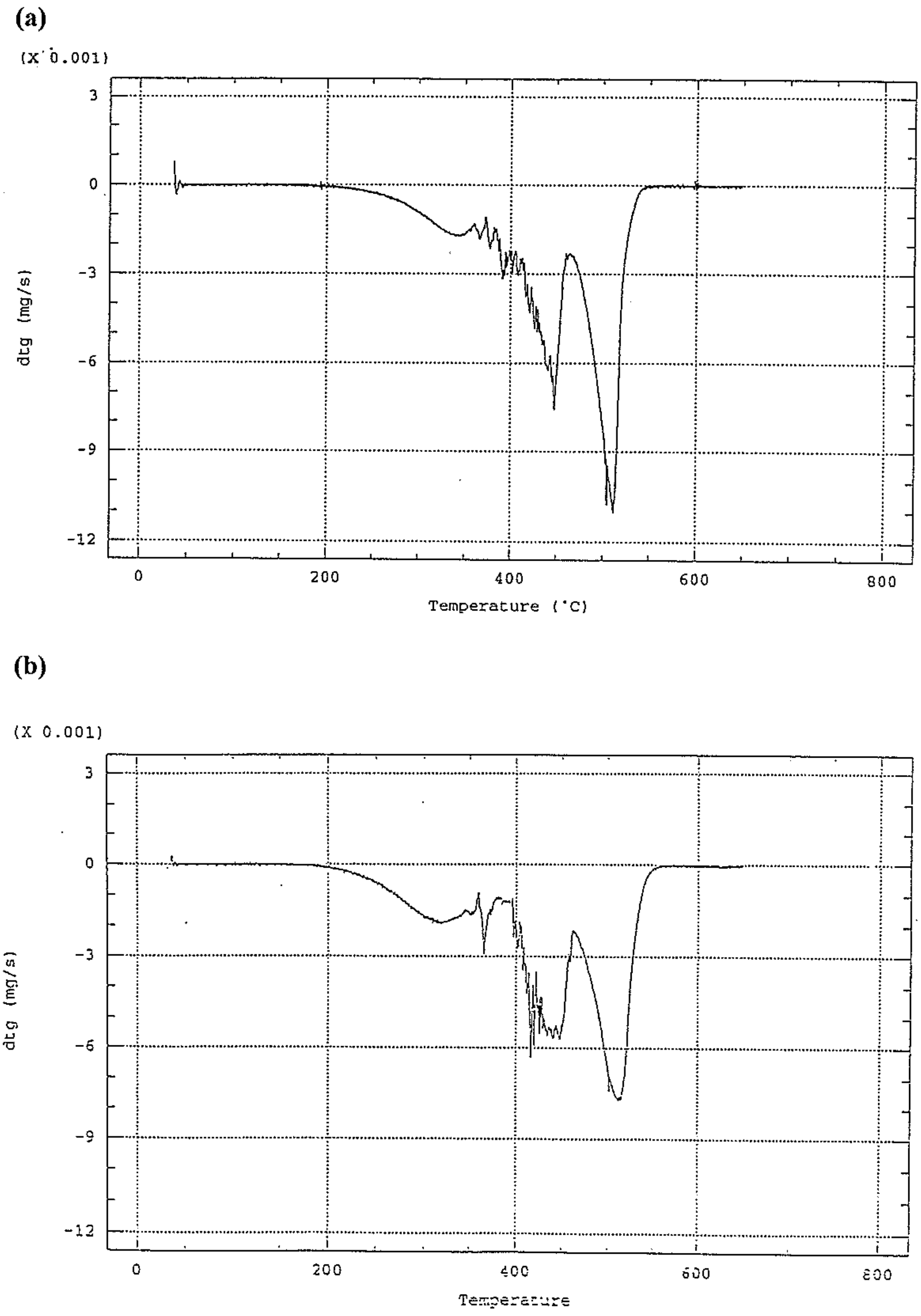
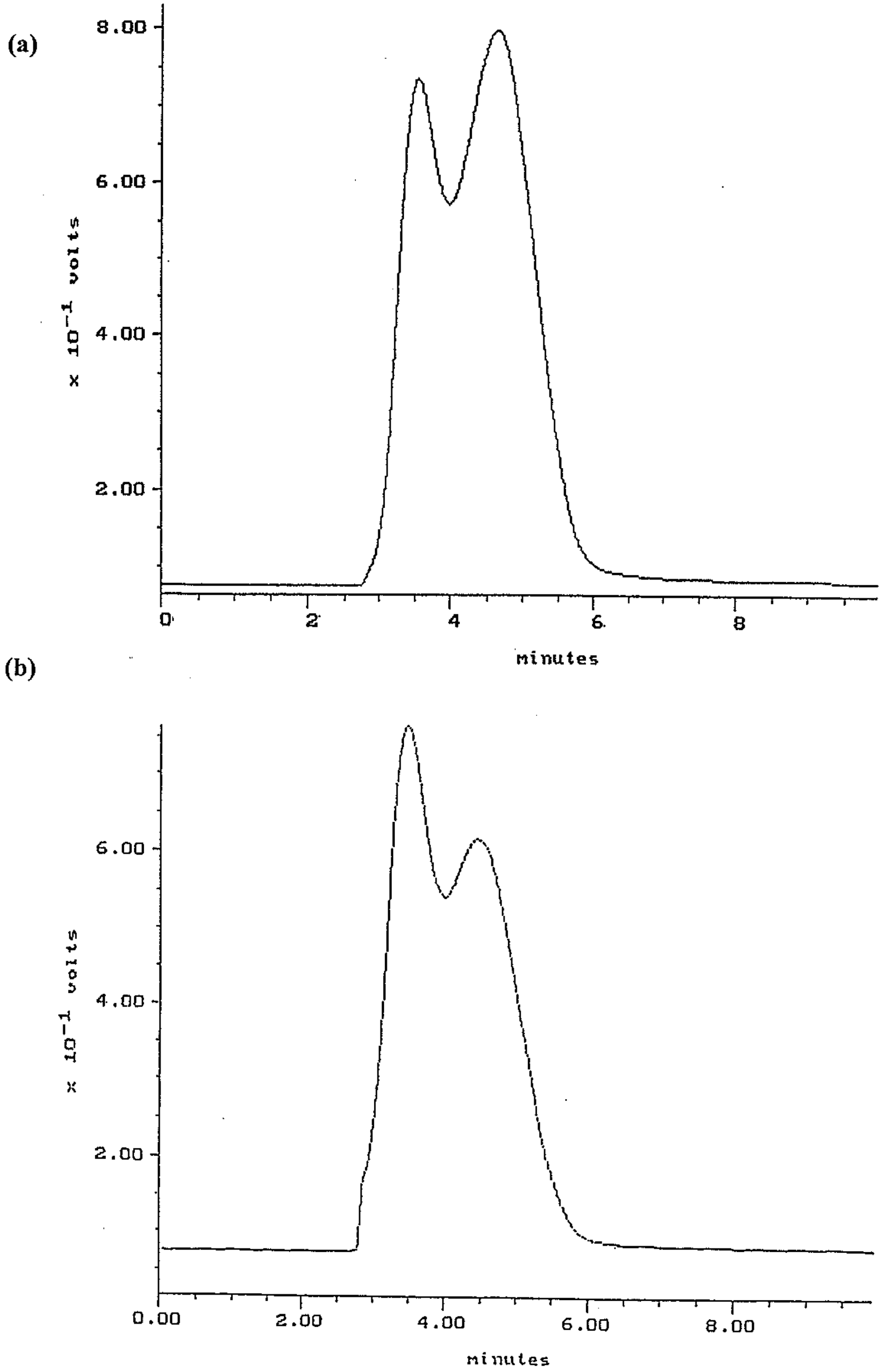


Figure A1.2 GPC chromatograms for (a) trial bitumen, and (b) Safaniya 180/200 bitumen.



However, it is also known that species eluting in the large molecular size region of the chromatogram can actually consist of agglomerations of associations of polar molecules, i.e. species in this region are not necessarily individual molecules. Thus the results indicate a greater propensity for inter-molecular association in the Safaniya bitumen than the trial bitumen.

A1.6.4 Asphaltene Content

Results of asphaltene measurements are given in Table A1.6.
The trial bitumen has a significantly lower asphaltene content than the Safaniya bitumen. This is consistent with the observations made above in the GPC section on the smaller degree of inter-molecular association apparent in the trial bitumen.

Table A1.6 Asphaltene contents in the trial bitumens.

Bitumen	Asphaltene content % (w/w)
NZRC trial	10.5
Safaniya 180/200*	13.2 ±0.9

* Mean ±95% confidence limits for nine samples taken over the course of a year (Ball & Herrington 1994)

APPENDIX 2
HEAVY IRANIAN 180/200 TRIAL BITUMEN

A2.1 PRODUCTION

The New Zealand Refining Company (NZRC) also produced another trial bitumen from Heavy Iranian crude oil through the old vacuum distillation unit (HVU1), using the same procedures as currently used for Safaniya bitumen. The material tested was sampled as the product was run into the storage tank, and is therefore not mixed with any residual Safaniya material. It was sufficiently different to be used for Stage 2 of this project for assessment of temperature-sensitivity of bitumens for chipsealing on New Zealand roads.

A2.2 LABORATORY TESTING PROGRAMME

The testing programme was as follows:

- full test to Specification TNZ M/1;
- comparative adhesion tests (Vialit test equipment);
- rheological tests on 50:50 blend of Heavy Iranian 180/200 and Safaniya 180/200 bitumens;
- rheological tests on 80/100 bitumen produced from Heavy Iranian 180/200 and Safaniya 45/55 bitumens;
- effect of kerosene and diesel on 60°C viscosity of Heavy Iranian 180/200 bitumen;
- chemical characterisation.

A2.3 FULL TESTING TO TNZ M/1 : 1989

Results for this testing are listed in Table A2.1.

Data additional to TNZ M/1 requirements, namely specific gravity and 5°C penetration figures, are also given. For comparative purposes, rheological test results from the Safaniya 180/200 bitumen used to produce the 50:50 blend are also included.

Table A2.1 Tests to Specification TNZ M/1 : 1989 for the test bitumens.

Test	Heavy Iranian 180/200	Safaniya 180/200	Typical Safaniya	TNZ M/1 Spec/n
Penetration, 25 °C (dmm)	186	180	-	180-200
Softening point (°C)	41.2	41.0	-	37-43
Ductility, 25 °C (cm)	>100	-	>100	100 min
Flash point (°C)	312	-	312	218 min
Viscosity, 70 °C (cSt)	12200	21470	-	14000min
Viscosity, 135 °C (cSt)	177	260	-	140-350
Solubility (%)	99.98	-	99.89	99.5 min
Density, 25 °C (kg/l)	1.012	-	1.023	-
RTFO				
% retained penetration	58	-	57	50 min
ductility, 25 °C (cm)	>100	-	>100	60 min
Penetration, 5 °C	15	18	-	-
Acid number (mgKOH/g sample)	0.13	-	0.25	0.35 max
Durability oven test (days)	11.5	-	13.5	12 min

min - minimum; max - maximum
cSt - centistokes; RTFO - rolling thin film oven test

Values of the viscosities, penetrations and softening point of the Heavy Iranian 180/200 are plotted on a Shell bitumen test data chart (BTDC) (Heukelom 1973), along with values for the Safaniya bitumen used for blending (dotted curve) (Figure A2.1). In this chart the penetration scale is logarithmic (log 10), while the viscosity scale is designed to give a straight line plot against temperature. The penetration and logarithmic scales intersect at a penetration value of 800 dmm (taken to approximate the penetration at the bitumen ring and ball softening point) and a viscosity of 1300 Pa.s.

The two bitumens are similar at low temperatures, the Heavy Iranian being slightly harder at very low temperatures. However, in the viscosity-temperature plot region the Heavy Iranian 180/200 material has a significantly lower viscosity (in fact, below the prescribed TNZ M/1 values). The larger discontinuity between the viscosity curve and the penetration curve for the Heavy Iranian bitumen would normally be ascribed to a greater proportion of waxy materials liquefying rapidly in the vicinity of the softening point, and resulting in the lower viscosities. Without this waxy component the two bitumens would be very similar as regards temperature sensitivity.

A2.4 COMPARATIVE ADHESION TESTS

The procedure adopted here was precisely as for the August 1993 trial bitumen (Section A1.4). The results are listed in Table A2.2.

Figure A2.1 Bitumen test data chart for the three 180/200 bitumens.

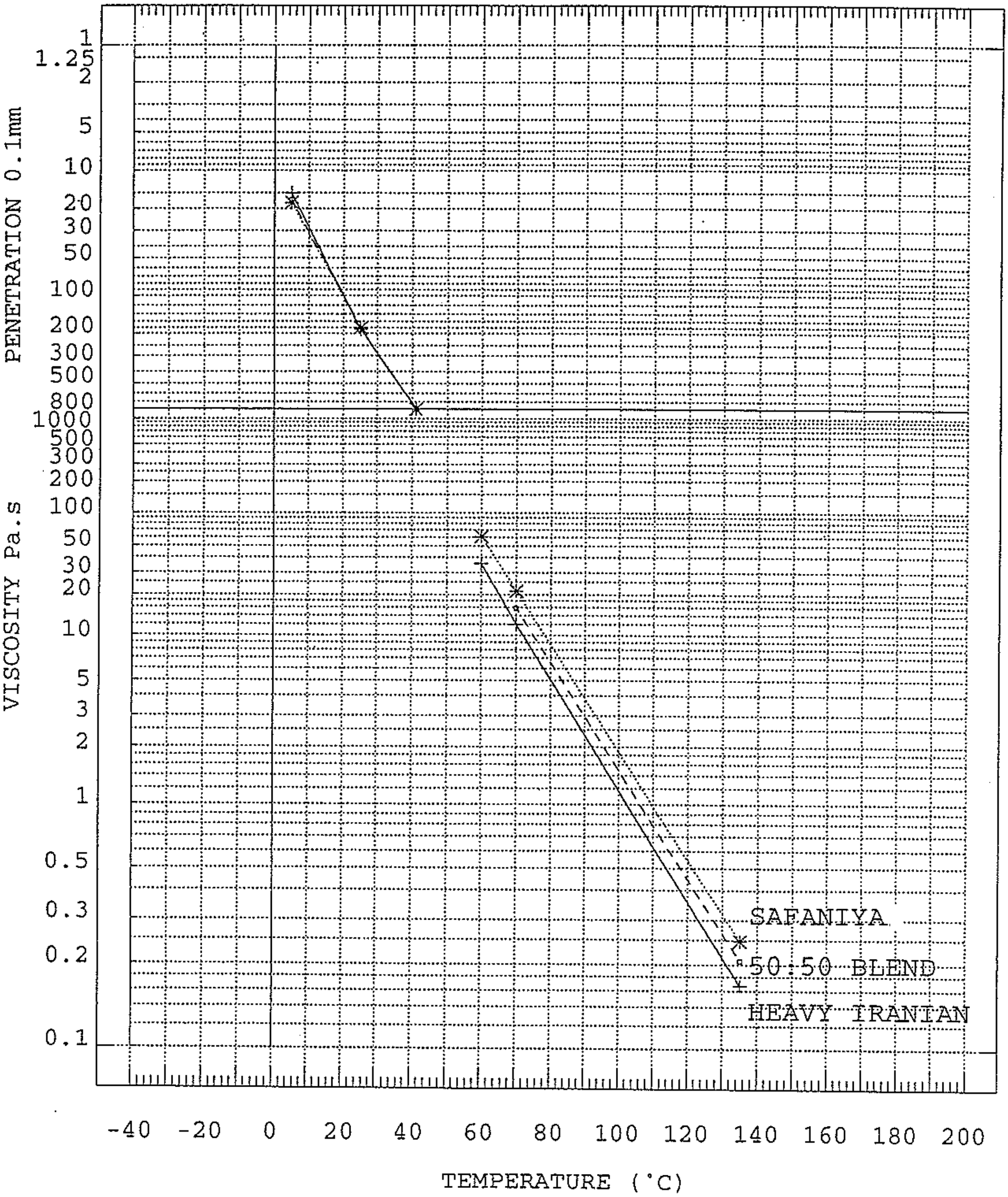


Table A2.2 Adhesion test results for Heavy Iranian and Safaniya 180/200 bitumens.

Sealing chip	Heavy Iranian bitumen (%)	Safaniya bitumen (%)
Rangitikei	96	98
Winstone's (Lower Hutt)	92	91

Given the repeatability of the test, the Heavy Iranian and Safaniya 180/200 bitumens are indistinguishable in performance. As in the case of the August 1993 trial bitumen (Table A1.2), the Rangitikei chip gives slightly greater adhesions than chip from Winstone's.

A2.5 BLENDS OF HEAVY IRANIAN AND SAFANIYA BITUMENS

A2.5.1 Purpose of Blending

If Heavy Iranian 180/200 bitumen is not considered to adequately conform to the rheological requirements of TNZ M/1 at higher temperatures, it will be necessary to blend it with Safaniya bitumens. For blending bitumens the penetrations (P) and viscosities (V) at defined temperatures can usually be predicted accurately by linear relationships of the form:

$$\log P\ (\text{mixture}) = m \log P\ (\text{bitumen 1}) + (1-m) \log P\ (\text{bitumen 2}) \qquad (\text{A2-1})$$

and

$$\log \log V\ (\text{mixture}) = m \log \log V\ (\text{bitumen 1}) + (1-m) \log \log P\ (\text{bitumen 2}) \qquad (\text{A2-2})$$

where:

m is the proportion of bitumen 1 in the mixture
$(1-m)$ is the proportion of bitumen 2 in the mixture

These relationships are approached with various degrees of accuracy depending on the bitumens, but in certain cases, usually with incompatible bitumens, they may be quite inaccurate, especially when roughly equal quantities of the two bitumen types are present. It is necessary to check whether this is the case. Compatibility can be further checked by ductility measurements on the blend, both before and after rolling thin film oven (RTFO) treatment.

Two blends were produced:

1. A 50:50 blend (by mass) of Heavy Iranian 180/200 bitumen with a 180/200 Safaniya bitumen; and

2. A blend of Heavy Iranian bitumen with a 45/55 Safaniya bitumen (penetration 50 dmm). The blend was targeted (using the formula for penetration of blends) to have a penetration of 90 dmm.

A2.5.2 Properties of Blends

A2.5.2.1 180/200 Blend

Properties of this blend, along with the equivalent properties of the component parts, and the values for rheological (flow) properties of the blend calculated from equations (A2-1) and (A2-2), are listed in Table A2.3.

Table A2.3 Properties of the test 180/200 bitumens.

Property	Heavy Iranian 180/200	Safaniya 180/200	50:50 blend	Predicted properties of blend
Penetration, 5 °C (dmm)	15	18	17	16.4
Penetration, 25 °C (dmm)	186	180	186	183
Softening point (°C)	412	41.0	41.2	-
Viscosity, 60 °C (cSt)	35580	-	60970	46420
Viscosity, 70 °C (cSt)	12200	21470	15890	16120
Viscosity, 135 °C (cSt)	177	260	219	214
Ductility, 25 °C (m)	1.0+	1.0+	1.0+	-
Density, 25 °C (kg/l)	1.012	1.020	1.016*	1.016
RTFO residue				
retained penetration (%)	5.8	-	-	-
ductility, 25 °C (m)	1.0+	-	-	-

* Calculated; min - minimum; max - maximum
cSt - centistokes; RTFO - rolling thin film oven test

The rheological (flow) properties of the blend have been plotted on the bitumen test data chart in Figure A2.1. It is apparent, from this chart and from the predicted properties column of Table A2.3, that equations A2-1 and A2-2 predict blend properties accurately, and can therefore be used for calculating the properties for blends with other proportions of the two component bitumens. The two bitumens are compatible.

A2.5.2.2 80/100 Blend

This blend consisted of 45.1% of Heavy Iranian 180/200 bitumen and 54.9% of Safaniya 45/55 bitumen (penetration 50 dmm). Properties of the blend are listed in Table A2.4, along with those of an 80/100 Safaniya bitumen (sampled November 1991) and the relevant TNZ M/1 specification. The rheological properties of the blend and the Safaniya 80/100 bitumen are also plotted together on a bitumen test data chart (Figure A2.2).

Figure A2.2 Bitumen test data chart comparison of 80/100 bitumens.

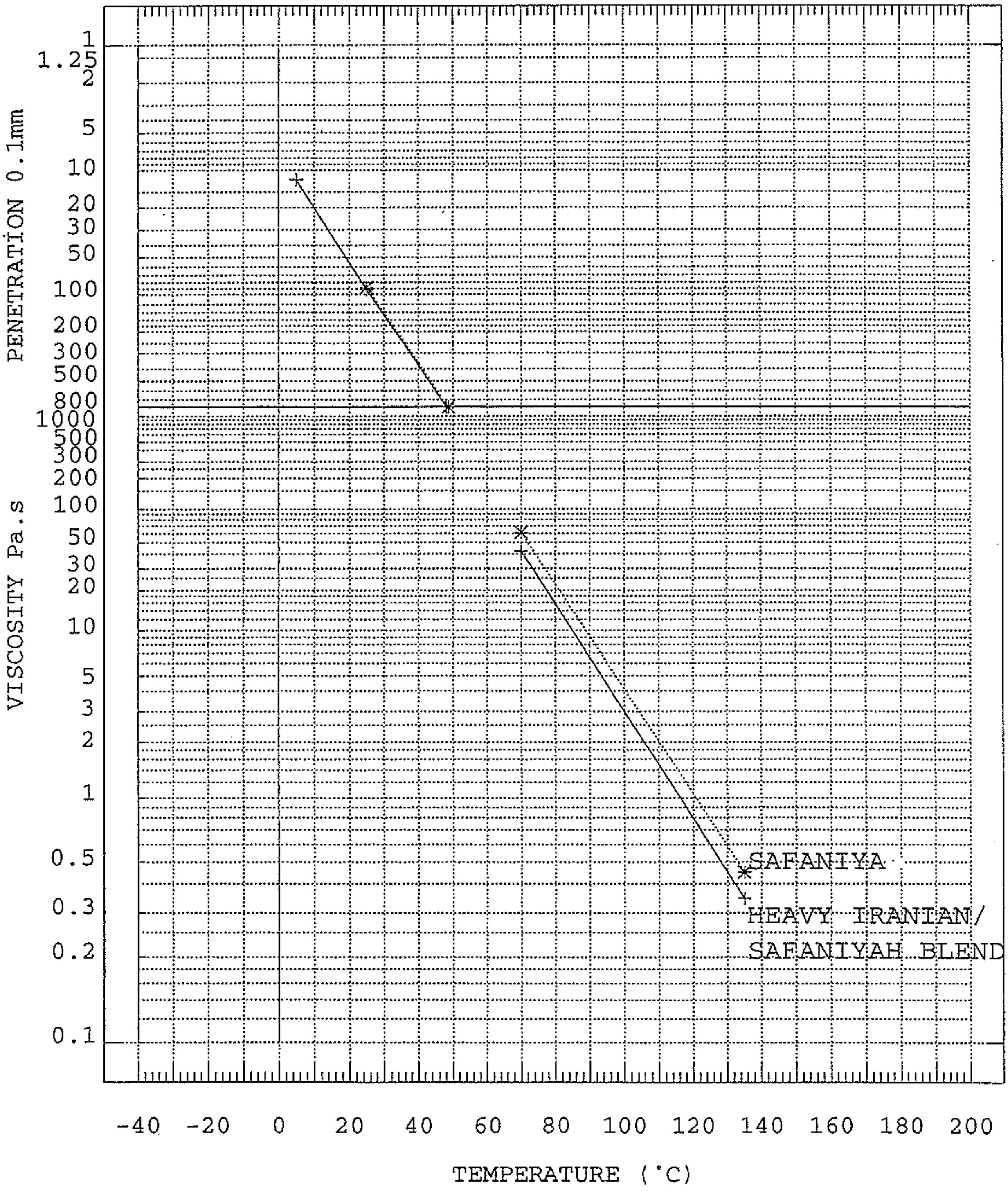


Table A2.4 Properties of 80/100 blend bitumens.

Property	Heavy Iranian/ Safaniya blend	Safaniya	TNZ M/1 speci
Penetration, 5 °C (dmm)	12	-	-
Penetration, 25 °C (dmm)	92	88	80-100
Softening point (°C)	48.1	48.7	45-52
Viscosity, 70 °C (cSt)	42380	61200	40000 min
Viscosity, 135 °C (cSt)	363	472	300-650
Ductility, 25 °C (m)	1.0+	-	1.0 min
Density, 25 °C (kg/l)	1.020*	1.027	-
RTFO residue			
retained penetration (%)	58	66	50 min
ductility, 25 °C (m)	1.0+	1.0+	0.60 min

* Calculated; min - minimum; max - maximum
cSt - centistokes; RTFO - rolling thin film oven test

The low viscosity levels for the 180/200 Heavy Iranian bitumen are reflected in those of the blended 80/100 product. However, the blended product meets the requirements of the TNZ M/1 specification for 80/100 bitumens, even if the viscosity levels are considerably below those of typical Safaniya 80/100 bitumens.

The RTFO residues easily meet the TNZ M/1 requirements.

A2.5.3 Conclusion

Heavy Iranian 180/200 bitumen can be blended with Safaniya bitumens to produce within-grade products.

A2.6 EFFECT OF KEROSENE AND DIESEL ON 60°C VISCOSITY

A check of the effects of various quantities of these diluents was carried out as in Section A1.5 of this report for the August 1993 trial bitumen.

Results of viscosity measurements are listed in Table A2.5.

Table A2.5 Viscosity (60°C) values for different kerosene and AGO quantities.

Kerosene pph (volume)	AGO pph (volume)	Viscosity (60°C) mm²/s	
		Measured	Curve fit
0	0	35580	36511
0	5.01	12880	12987
5.00	0	11840	11278
5.00	5.01	4939	4905
10.02	0	4244	4331
15.01	0	1975	1979

These results were fitted by regression procedures to the following equation:

$$\log\log V = \frac{1}{(100+K+D)}(65.9196-0.4224K-0.2837D) \qquad \text{(A2-3)}$$

where:

V is the viscosity (mm²/s) at 60°C of the binder
K is the quantity of kerosene (pph by volume at 15°C)
D is the quantity of AGO (pph by volume at 15°C)
The logarithms are to base 10.

For a typical 180/200 Safaniya bitumen we have the equivalent equation:

$$\log\log V = \frac{1}{(100+K+D)}(67.44-0.4250K-0.1971D) \qquad \text{(A2-4)}$$

The large constants (65.9196 and 67.44) in equations A2-3 and A2-4 reflect the viscosities of the undiluted bitumens, which are significantly different (36511 mm²/s as against 53086 mm²/s). The sensitivities of the two bitumens to kerosene content are practically identical (coefficients of K are 0.4224 and 0.4250 respectively). The Heavy Iranian bitumen is slightly more sensitive to diesel than Safaniya, but at the dosages of diesel normally used the difference is of no practical significance. (For example, at around 5 pph diesel content, viscosity levels predicted from using the two diesel constants – 0.2837 and 0.1971 – are equivalent to a change of approximately 0.5 pph of diesel content.)

For blends of Heavy Iranian and Safaniya bitumen, therefore, a predictive equation of the following form will be satisfactory for practical purposes:

$$\log\log V = \frac{1}{(100+K+D)}(B-0.4250K-0.1971D) \qquad \text{(A2-5)}$$

where:

$$B = 100\,[\,m \log\log(36511) + (1-m)\log\log(53086)\,]$$
$$= 67.44 - 1.5204\,m$$

and

m is the proportion of Heavy Iranian bitumen in the blend by mass

A2.7 CHEMICAL CHARACTERISATION

A2.7.1 Introduction

The chemical analysis procedures were the same as for the NZRC 1983 trial bitumen, as detailed in Section A1.6 of Appendix 1 of this report.

A2.7.2 Thermogravimetry

Data for the Heavy Iranian 180/200 bitumen are compared with Safaniya 180/200 results in Table A2.6; DTG curves for the Heavy Iranian and Safaniya 180/200 bitumens are compared in Figure A2.3.

Table A2.6 Thermogravimetric analysis of test bitumens.

Bitumen	**Percentage weight lost***			
	200-300°C	**300-400°C**	**400-500°C**	**500-600°C**
Heavy Iranian 180/200	7.5	22.5	53.2	16.4
Safaniya 180/200	8.0 ±0.6	20.4 ±1.8	47.6 ±1.8	23.7 ±2.3

* Mean of duplicate measurements

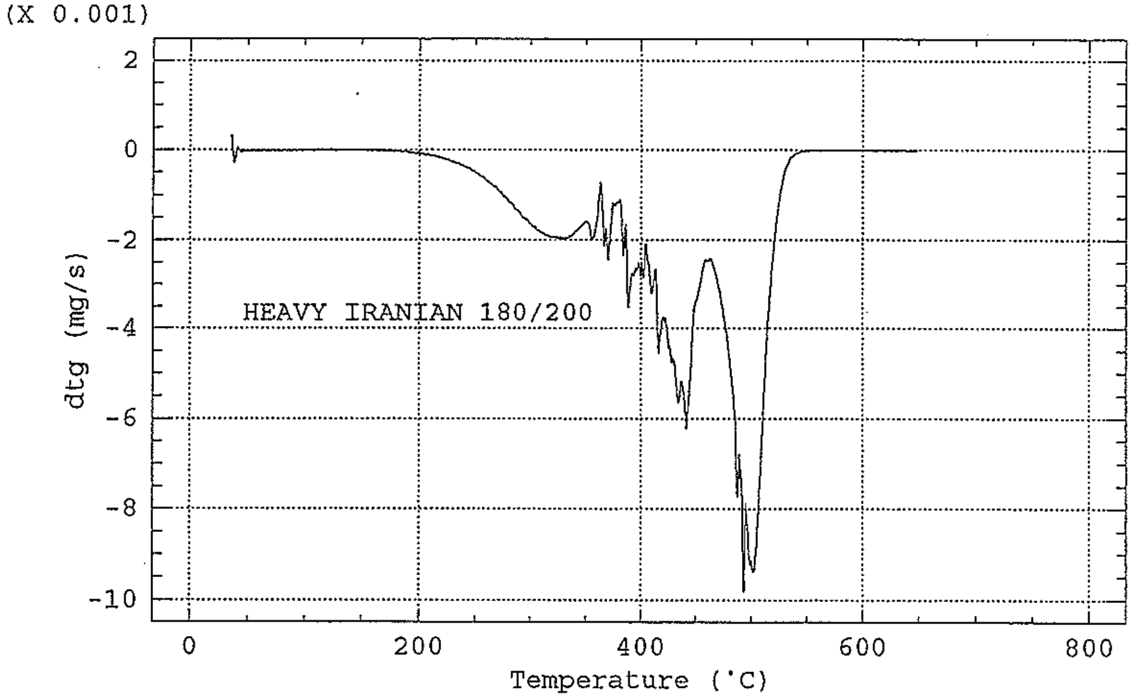
Up to 450 °C the Heavy Iranian and Safaniya bitumens cannot be distinguished, within the repeatability of the experimental method. The high temperature peak of the Heavy Iranian is narrower than that of the Safaniya, so that full combustion/volatilisation is completed at a slightly lower temperature. The results indicate that in the range of temperatures that occur in sealing and paving processes the two bitumens will be similarly affected.

A2.7.3 Gel Permeation Chromatography (GPC)

A chromatogram for the Heavy Iranian bitumen is shown in Figure A2.4, along with a Safaniya 180/200 bitumen for comparison. This shows the Heavy Iranian 180/200 to have less large molecular size material than Safaniya. Measurements of the areas of 30 second slices taken across the chromatogram are given in Table A2.7. A comparison with the data of Table A1.5 (Appendix 1 of this report) indicates that the Heavy Iranian bitumen also has somewhat smaller molecular sized materials than the August 1993 trial bitumen.

Figure A2.3 Thermogravimetric first derivative of weight loss curves for (a) Heavy Iranian and (b) Safaniya 180/200 bitumens.

(a)



(b)

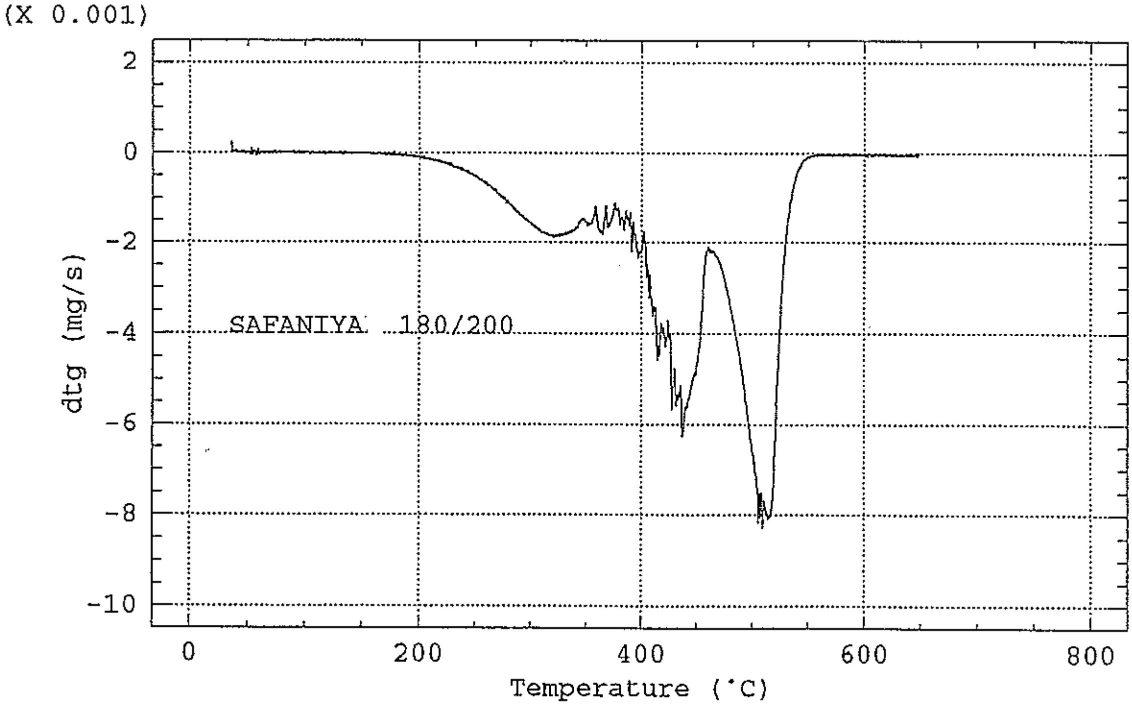
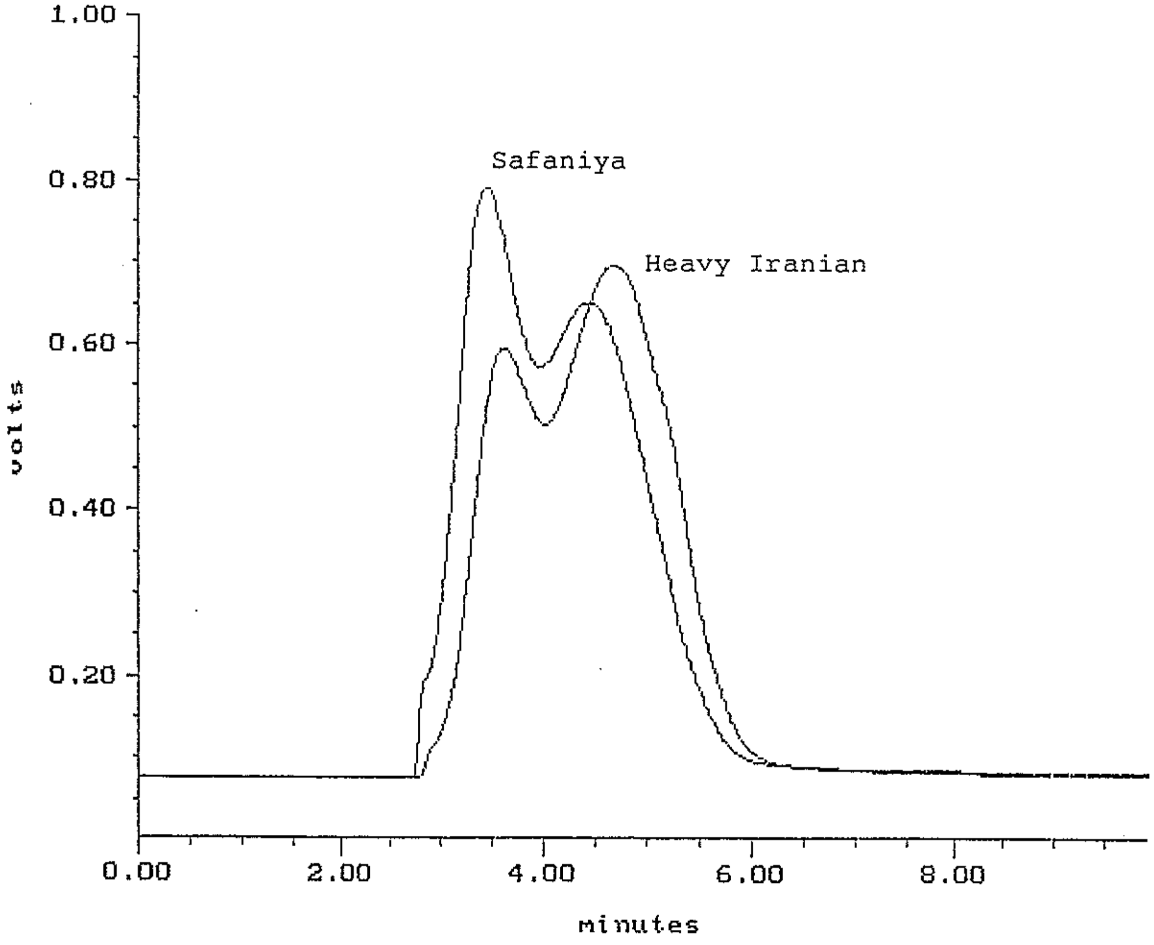


Table A2.7 GPC chromatograms of test bitumens.

Bitumen	Slice area as a percentage of total Slice no.						
	1	2	3	4	5	6	7
Heavy Iranian 180/200	0.6	10.8	21.0	21.6	26.0	16.2	3.8
Safaniya 180/200	2.3 ±0.6	21.6 ±0.6	24.0 ±0.6	22.0 ±0.2	19.8 ±0.4	8.7 ±0.2	1.7 ±0.2

Figure A2.4 GPC chromatograms for Heavy Iranian 180/200 and typical Safaniya 180/200 bitumens.



A2.7.4 Asphaltene Content

Results of asphaltene measurements are given in Table A2.8.

Table A2.8 Asphaltene contents of test bitumens.

Bitumen	Asphaltene content % (w/w)
Heavy Iranian 180/200	9.5
Safaniya 180/200	13.2 ±0.9

The Heavy Iranian bitumen has a significantly lower asphaltene content than Safaniya bitumen, and a slightly lower one than the August 1993 trial bitumen. This is consistent with the GPC results, for which the lower proportion of high molecular size material for the Heavy Iranian bitumen suggests that the Safaniya bitumen has the greater propensity for inter-molecular association.

A2.7.5 Aromatic Character

Proton nmr (nuclear magnetic resonance) spectra were recorded to obtain data related to the degree of aromatic character of the bitumen. This parameter is related to the compatibility of bitumen and polymer binders.

The results in Table A2.9 indicate that Heavy Iranian bitumen is expected to have similar compatibility to polymers as the Safaniya product. Safaniya results are from Ball & Herrington (1994).

Table A2.9 Aromatic character of the test bitumens.

Bitumen	Aromatic hydrogen %
Heavy Iranian 180/200	7.1
Safaniya 180/200	7.0 ±0.6

APPENDIX 3
CHEMICAL METHODS FOR CHARACTERISING BITUMENS

A3.1 THERMOGRAVIMETRY

Measurements were carried out on a Mettler TG50 thermobalance controlled by a Mettler TC10A processor. The raw data were transferred to a computer for analysis. The bitumen sample (10-11 mg) was heated at 5 °C/min in an open platinum crucible (5 mm high, 5 mm i.d.) in an air purge (200 cm^3 min^{-1}). The inorganic ash content of the bitumen was negligible (<0.1% w/w).

A3.2 GEL PERMEATION CHROMATOGRAPHY (GPC)

The GPC system comprised a Waters 600E Powerline pump module connected to a Hewlett Packard 1050 UV detector and a Waters 410 refractive index detector in series, an Eldex column heater and a Rheodyne 7125 injection valve. The following operating conditions were used:

Column:	Waters Ultrastyragel 10^3Å
Column Temperature:	30 °C
Mobile Phase:	Toluene, 2 ml/min (helium de-gassed)
UV Detector:	400 nm
RI Detector:	35 °C

Condition of the column was monitored using injections of 10% ortho-dichlorobenzene in toluene and a refractive index detector operating at 35 °C. No column deterioration (as reflected in plate count) was observed after the bitumen sample was run.

The bitumen sample was prepared as follows: 0.500 g of bitumen in a sealed vial was dissolved by the addition of 10.0 ml of toluene and allowed to stand overnight (18.75 hours) at room temperature (~20 °C) with occasional shaking. The solution was filtered through a 0.2 μ teflon syringe filter and 20 ml injected (sample loop) into the GPC system.

The use of a single column, a high flow rate, high sample concentration and non-polar mobile phase were all purposely chosen to minimise disruption of molecular associations within the sample (Brule et al. 1986, Yapp et al. 1991).

A3.3 ASPHALTENE CONTENTS

Asphaltenes (n-heptane) were determined according to ASTM D3279-97 with the exception that the dispersions were allowed to stand for 17-20 hours after reflux. This modification allows a more complete precipitation of the asphaltenes (Speight et al. 1984). All analyses were carried out in duplicate.

A3.4 PROTON NUCLEAR MAGNETIC RESONANCE SPECTRA

Proton nmr spectra were recorded at Industrial Research Ltd, Lower Hutt. Samples were run at room temperature as 10.0% solutions in $CDCl_3$ on a Bruker 300 MHz instrument using trimethyl-silane (TMS) as internal standard.

Duplicate samples were run. The spectra were divided into regions according to the proton type contributing to the signal in that region. These assignments were as follows:

Chemical Shift Range (ppm from TMS)	*Assignment* (relative to anaromatic ring)
0.5-1.0	H_γ (γ or greater CH_3)
1.0-2.0	H_β (β CH_3, β or greater CH_2, β or greater CH)
2.0-4.5	H_α (α CH_3, α CH_2, α CH, $\alpha\alpha$ CH_2, possibly some CH_2 as in indane)
4.5-6.0	$H_{olefinic}$ (not observed in any of the bitumens studied)
6.2-9.3	H_a (aromatic ring protons, aromatic heterocycles)

Following the approximation usually made in the literature, the contribution from protons directly attached to heteroatoms (which would be very small) was ignored.

The percentage aromatic hydrogen was calculated as follows:

$$\% \text{ aromatic H} = \frac{H_a}{(H_\alpha + H_\beta + H_\gamma)} \times 100$$

where:
H_a, H_α, H_β and H_γ are the areas under the nmr spectrum between the limits given above.

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