MONITORING BITUMEN QUALITY FROM REFINERY TO PAVEMENT

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ABSTRACT

The properties of bitumen change as it ages in bulk storage, transport, and storage on site. The change in bitumen properties from refinery, shipping, storage and road transport is monitored for some bitumen in Australia. The changes are generally related to ageing, and ageing indices are derived for different parts of the process. Using data from two South African and Australian laboratory studies into ageing of bitumen, a lack of correlation between bulk sample ageing and small sample ageing is found. It is found that changes in properties from refinery to pavement also come from micro-contamination from normal work practices. The detection and effects of contamination and how that can be controlled are considered.

Keywords: bitumen storage ageing sampling

1. INTRODUCTION

This paper reports on measured changes in bulk bitumen properties during transport and storage, and discusses those in terms of ageing and contamination found in field and laboratory investigations. These changes are often small, although they can be significant in contractual terms. The paper is focussed on bulk bitumen, and does not cover ageing during asphalt manufacturing/construction nor ageing of bituminous surfacings in service on the road since these have been well dealt with elsewhere in the literature (for example Dickinson, 1984). Data from South Africa and Australia are used since the issue is generic, and the two countries have many similarities in road materials and design systems. The test methods and specifications used throughout this paper are the ones appropriate to that particular bitumen. For example, a reference to viscosity in the context of Class 170 bitumen means Australian specification AS 2008. Similarly a reference to penetration in the context of 60/70 bitumen means SABS 307 specification.

The paper reports on two laboratory studies on ageing. It then reports on how some bitumen properties change from refinery to pavement. The changes are generally related to ageing, and ageing indices are derived for different parts of the process. It is discovered that changes in properties of bulk bitumen from refinery to pavement also come from micro-contamination. The paper then considers the detection and effects of contamination, and how that can be controlled.

2. LABORATORY STUDIES INTO AGEING OF BITUMEN

2.1 Precision of Testing

Two laboratory studies are reported here on the ageing of bitumen in storage. Since some changes in properties with ageing were small, this type of study must be mindful of the limits to precision in testing. Precision limits are specified for many bitumen test methods. For penetration (AS2341.12, 1993), repeatability (duplicate results by the same operator using the same equipment) "should not be considered suspect unless they differ by more than 3% of their mean". Reproducibility (results submitted by each of two laboratories) "should not be considered suspect unless the two results differ by more than 8% of their mean".

There are other issues, specific to bitumen, which affect such ageing studies because its properties can be changed during sampling or testing. The need to heat bitumen to take samples, or to pour into various sample chambers means a potential for oxidisation. The size of hot stored samples and/or the handling of samples at elevated temperatures can affect the rate of oxidisation. The thermal history must be considered in the protocols of testing.

2.2 Bitumen Ageing - South African Study

A South African study into bitumen ageing and the in-tankage changes in the quality of bulk bitumen was done in 2002 (Coe, 2003). Four samples of about four litres each of South African 80/100 bitumen were drawn from large bulk storage at Calref refinery and distributed to participating laboratories. They were instructed to store the samples in their ovens at a temperature of 160°C or as close to this temperature as possible. On a weekly basis, a sample was drawn from the four-litre tin and tested for penetration and softening point. Testing was continued until sample depletion, which took approximately six weeks.

The small size and method of handling the samples had an effect on the results, which was anticipated, and one of the secondary aims of the testing was to determine the variability of testing between different laboratories. The results for penetration are shown in Table 1, and show significant variations with time as expected, but also quite a variation between laboratories. While all showed a trend to decreasing penetration upon ageing, the extent varied. Small variation could be due to the precision of testing, but the variation quickly went outside the precision limits, suggesting that the differences upon ageing were due to factor(s) other than test reproducibility alone.

Table 1. Ageing of 80/100 small samples at 160°C (penetration @ 25°C, 0.1mm).

Days ageing	Laboratory refinery	Laboratory S	Laboratory M	Laboratory C
1	82	80	79	67
3	81	84	74	65
5	75	85	70	60
8	76	83	65	57
10		80	59	51
12		53	54	
15	78	46	48	41
17	77	45	45	40
19	73	45	41	39
22	76	39	36	
24	73	36	34	
26	69	32	31	

The original conclusions of the study were that the variability in results reflected a number of factors, such as reproducibility effects, thermal history, sample preparation (re-heating) techniques and varying oven environments (such as whether the samples were stored with the lids on). It is clear that laboratory ageing of bitumen in small sample sizes brings its own problems of measurement and does not simply replicate bulk storage.

2.3 Bitumen Ageing – Australian Study

To investigate some of the issues found in the South African study above, an Australian study was done (Nov 2003-Jan 2004) on the effect of sample handling on bitumen ageing of small samples. Using Class 170 bitumen (refined from Basra Iraqi crude), a number of 1-litre sample tins were filled and sealed. These tins were aged for varying times at 163°C in an oven before testing. At the same time, a single control tin was aged in the same oven, and was reopened, tested, and resealed each time that an aged sealed tin was tested. Effectively the control tin got emptier and emptier as testing progressed. It was suspected that the oxidation/ageing process might be more aggressive for this tin.

It was clear that opening and reclosing the control tin each time caused the bitumen to age more quickly than the individual samples in the closed tins (Table 2). This was especially evident for the property of Viscosity @60°C, as shown in Figure 1. There was a difference in the change of penetration with ageing between this bitumen and the South African one, which is ascribed to different crude sources and manufacturing route. The changes in the bitumen caused by ageing are dependent on the type of crude from which the bitumen is produced (E&E, 2000). Changes caused by ageing are also dependent upon the mode of manufacture – ie. two bitumens from the same crude but manufactured differently could exhibit different ageing behaviour.

Table 2. Ageing of class 170 small samples at 163°C.

Time	Control sample			Sealed tins		
	Visc60	Visc135	Pen25	Visc60	Visc135	Pen25
0	167	0.510	72	167	0.510	72
5 hours	176	0.370	62	182	0.560	57
24 hours	189	0.510	55	172	0.410	48
3 days	226	0.410	49	189	0.448	45
8 days	343	0.500	23	279	0.442	28
15 days	762	0.633	12	453	0.558	17
19 days	958	0.883	9	563	0.660	13
26 days	1302	1.250	8	1260	0.920	9

Note: Visc60 is viscosity @ 60°C to the rotational viscosity AS 2341.4 (1994) test method. Visc135 is viscosity @ 135°C. Pen25 is penetration @ 25°C

The data were used to develop a model to predict change of viscosity with time for small samples in storage. The data are very limited, and the relationship is for only one bitumen, so the model is tentative. The best fit model was exponential, although it was noted that a linear model was a reasonable fit in the initial aging; say below an ageing index (A_v) of 2. Equation 1 models the change in viscosity with storage time for Class 170 bitumen in small (1 litre) sealed samples at 163°C.

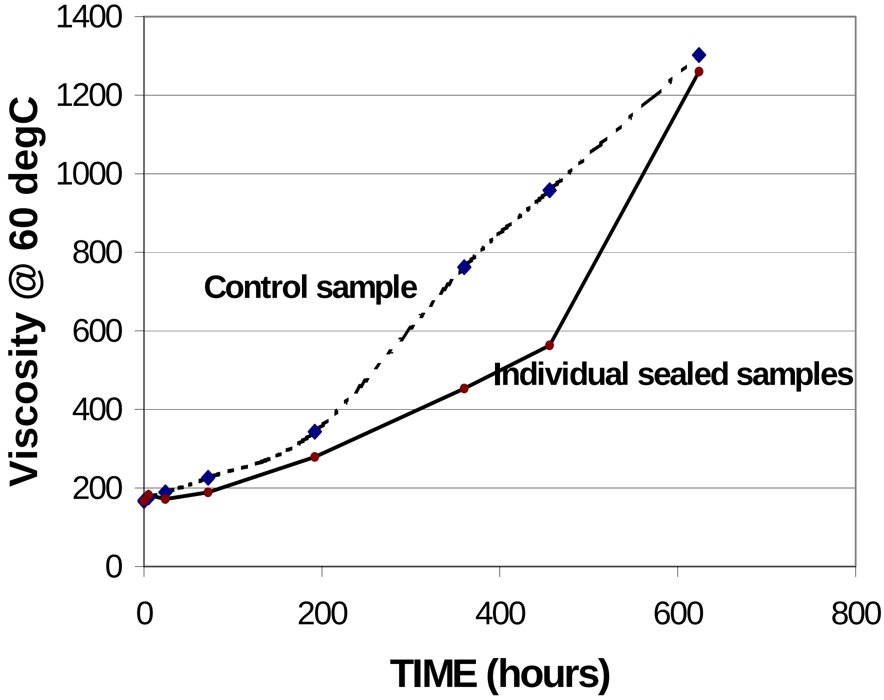

Figure 1. Ageing small samples @ 163°C - viscosity @ 60 degC.

$$V_{60} \text{ aged} = 161 e^{0.0031 t} \quad (R^2 = 0.98) \quad \text{(Eq.1)}$$

where : V_{60} = dynamic viscosity at 60°C, Pa.s

t = time stored at elevated temperature (163°C) (hours)

3. CHANGE IN BITUMEN PROPERTIES FROM REFINERY TO PAVEMENT

The change in bitumen properties from refinery (after production) through to the pavement was monitored by tracking changes in bitumen viscosity during storage, transport, and on site before use in sealing. The changes are small and if taken individually, some of the results could be ascribed to repeatability. However, when taken together, a coherent picture emerges.

3.1 Changes in Properties During Bulk Storage

After bitumen has been produced in a refinery, it is stored initially in bulk storage at the refinery, at a storage depot, or at an on-processing facility. The changes in bitumen properties during bulk storage were measured and are shown in Table 3. The bitumen is Class 170, refined from Basra Iraqi crude, wax content 1.2%, and is the same bitumen used for the Australian laboratory study above and the shipping study below. The storage tanks were properly equipped for bitumen storage, hot oil jacketed, and maintained at a reasonably constant temperature (which is shown in Table 3 for each tank).

It was found by Horvathne and Levey (2000) that an ageing index (A_v) could be established based on the ratio of "viscosity aged" to "viscosity unaged". This did not change significantly by grade of road bitumen. Five paraffin-based penetration graded road bitumens were tested by them: 20/30, 30/50, 40/70, 70/100 and 160/220. Their ageing was done in the laboratory using the RTFO test. The ageing index concept can be used here to normalise ageing patterns across the differing grades of bitumen tested.

Table 3. Change in a Class 170 bitumen properties in bulk storage.

Property	Time in Storage, *days*					
	0	1	4	26	39	59
Tank 1: 2650 tonne capacity, 120 °C						
Viscosity @60°C Pa.s	150	149	150	168	171	173
Viscosity @135°C Pa.s		0.36		0.45		
Penetration @25°C, 0.1mm	73	66	62	64	54	52
Tank 2: 1400 tonne capacity, 165 °C						
Viscosity @60°C Pa.s	141	156		170	170	175
Viscosity @135°C Pa.s		0.34		0.36		
Penetration @25°C, 0.1mm	75	71		57	54	52
Tank 3: 1650 tonne capacity, 140 °C						
Viscosity @60°C Pa.s	145	153		170	175	
Viscosity @135°C Pa.s		0.35		0.39		
Penetration @25°C, 0.1mm	75	68		60	54	

There is little difference found here in the change of bitumen properties upon ageing between the tanks despite the differing storage temperatures, although the maximum difference is considered statistically significant being typically greater than the repeatability limits. The data were combined to model change of viscosity @60°C in storage. Since the data are very limited, and themselves imprecise, the model is approximate, but the use of ageing index approach enables the findings to be applied across other grades of road bitumen from similar crudes. A linear model was a reasonable fit (R^2 = 0.82). This was expected from the finding above that a linear model was a reasonable fit to small sample ageing below an ageing index (A_v) of 2.

The ageing index (ratio of "viscosity aged" to "viscosity unaged") of bitumen in bulk storage at 120-165°C was found to increase by 0.0034 per day. A nominal size of >250 tonnes has been ascribed to bulk storage for this ageing index.

As an example, the change in ageing index for bitumen in bulk storage over 25 days would be 1 + 25 * 0.0034 = 1.0850. For the bitumen in Tank 2, it had a viscosity of 156 Pa.s at Day 1 (Table 3). At Day 26, the model would predict a viscosity of 156 * 1.0850 = 169 Pa.s, which compares with the measured viscosity of 170 Pa.s.

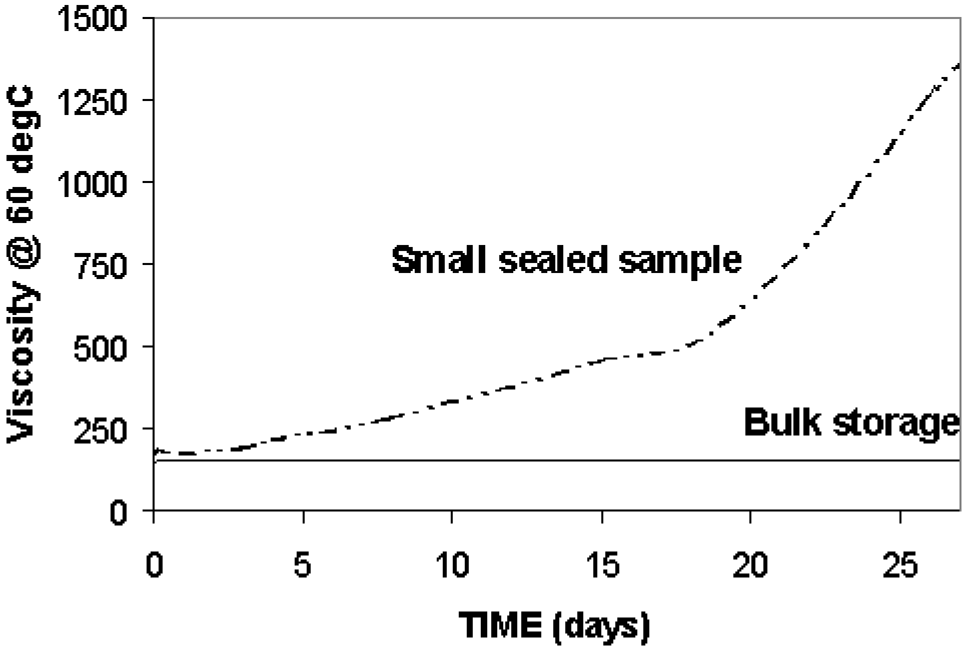

Figure 2. Bitumen ageing in hot storage (small tins 163°C, bulk 120-165°C).

The change in viscosity when ageing in bulk storage was found to be considerably different to that of small samples, even if properly sealed and the same bitumen source. Figure 2 shows the change in viscosity of bulk storage (mean of the three tanks in Table 3) and small samples (sealed tins of Table 2). It shows how any study on bitumen ageing can be confounded by the ageing effect of small samples, as previously suspected. It also indicates that the safe storage period for bitumen at elevated temperatures is longer than that codified. However periodic retesting of bitumen stored at elevated temperatures is warranted in view of the changes in bitumen properties over time.

3.2 Changes in Properties During Sea Transport

A similar change in bitumen properties was found during sea transport. There are specialised ships for bitumen transport with bulk 250-1500 tonne compartments, and bitumen is also transported in specialised bitumen containers with 13-26 tonne capacities.

The change in the properties of a bitumen during bulk sea transport between refinery storage tanks and distant bulk storage is shown in Table 4. The bitumen was Class 170, refined from Basra Iraqi crude. The specialised bitumen carrying ship had 1250 tonne capacity compartments with hot oil heating and jackets. The bitumen was kept hot at 140-155 °C during the voyage, and no heating was used at discharge. The shipping distance was 5400 km (which is typically an 11 day voyage).

Table 4. Change in a Class 170 bitumen during sea transport.

	Before shipping			After shipping		Averages	
Description	**Tank 1**	**Tank 2**	**Tank 3**	**Sample A**	**Sample B**	**Before**	**After**
Viscosity @60°C Pa.s	139	140	138	153	155	139	154
Viscosity @135°C Pa.s	0.33	0.33	0.32	0.341	0.334	0.327	0.338
Penetration @25°C pu	85	83	87	66	77	85	71.5

The results show an increase in viscosity @60°C after shipping, with the ageing index A_v of this bitumen during bulk sea transport at 140-155°C found to increase by 0.0098 per day. No data were available on the effect of shipping bitumen by container.

3.3 Change in Properties During Road Transport and on Site

The change in bitumen properties during road transport and during storage on site was found on a project in Broome, Australia. Class 320 bitumen for spray sealing was hauled 2400kms from the refinery at Perth (North Fremantle) to a site in Broome by road in 11,000 – 28,000 litre tanks. A variety of haulers/trailers and sprayers were used, and usually in a road train configuration. The climate is hot-arid, and haulage was done in warm to hot conditions. The haul time was 3 days, and the bitumen was stored on site for up to 6 additional days. The bitumen was typically loaded at 180°C, and maintained between 140 and 180°C during transport by gas heating every 6 hours. On site, the temperature in storage typically varied between 110 and 200°C.

A bitumen laboratory was established on the project in Broome to test viscosity and loss of mass on heating for quality control purposes. Sampling was done in North Fremantle and Broome using a in line sample cocks (WA700.1, 1998), or from the spray bar during circulation. No bottle sampling was used.

The bitumen as supplied, and after haulage, and after site storage, met specification and there was no gross contamination of the bitumen. But within that broad statement, the detailed results showed confounding by other factors. The detailed data are shown in the Appendix. There was no obvious pattern in the results, apart from a general trend of increasing viscosity with time. The trend is generally statistically significant with many differences being greater than the repeatability limits. However the smaller changes in individual results could be due to micro-contamination or test precision or both, and the scope of testing was insufficient to explore this further.

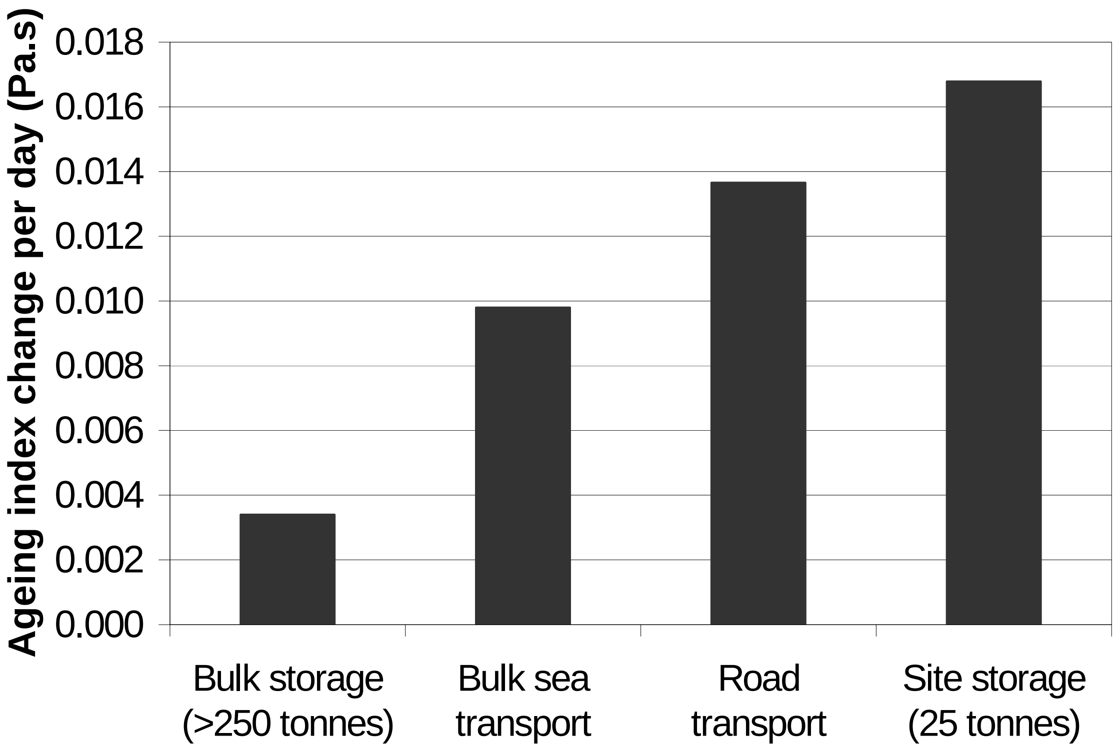

Figure 3. Ageing index change per day for road grade bitumens stored hot (various temperatures).

The data were analysed to quantify the rate of viscosity change, although they were limited, and the experimental design incomplete, making the conclusions tentative. Some of the samples taken at the refinery were taken at the loading point before loading, and some were taken from the sprayer or trailer immediately after loading (as a result of local work practices). This allowed micro-contamination of the 'after loading' samples to occur, which is discussed further below, and the viscosity of micro-contaminated samples was corrected by a standard correction of 20 Pa.s (averaged from the data of 15/11/01 and 18/9/02) before further analysis.

The analysed data showed an increase in viscosity @60 ^{o}C with time, with the ageing index A_v of this Class 320 bitumen during road transport at 140-180^{o}C increasing by 0.0137 per day. The ageing index A_v of this Class 320 bitumen during site storage at 110-200^{o}C increased by 0.0168 per day. Because Horvathne and Levey (2000) had shown that the ageing index did not change significantly by grade of road bitumen, the ageing indices calculated for the different grades of road bitumen (Class 170 and Class 320) were combined in Figure 3. This shows the change in ageing index per day for all modes of shipping and storage and, according to Horvathne and Levey (2000), the findings are applicable across a range of road grade bitumens. The data are limited and the model is therefore tentative.

As an example of the use of ageing index, the change in viscosity of a bitumen in 5 days from an initial viscosity of 170 Pa.s was calculated for each mode of storage and transport:

- Bulk storage (>250 tonnes), with an ageing index A_v of 0.0034 per day, would have an ageing index of 0.0170 after 5 days. Bitumen with an initial viscosity 170 Pa.s would rise to 170 * (1 + 0.0170) = 172.9 Pa.s

- Bulk sea transport, with an ageing index A_v of 0.0098 per day, would have an ageing index of 0.0490 after 5 days. Bitumen with an initial viscosity 170 Pa.s would rise to 170 * (1 + 0.0490) = 178.3 Pa.s

- Road transport, with an ageing index A_v of 0.0137 per day would rise to 181.6 Pa.s.
- Site storage (25 tonnes) with an ageing index A_v of 0.0168 per day would rise to 184.3 Pa.s.

3.4 Micro-Contamination

One finding was micro-contamination as a result of normal practices. The term micro-contamination is introduced deliberately because the effect is small and the contaminated bitumen is still within the specification. It should be noted that part of the viscosity change that is attributed to micro-contamination could be simply due to repeatability differences in testing, although the largest changes were greater than the repeatability limits and were thus considered statistically significant. Also micro-contamination might well be found through other bitumen tests; this discussion is based on viscosity simply because that was the specification and data available. In South Africa, penetration would be another appropriate test to use.

There was no evidence that the micro-contamination has led to problems with the surfacing in service (up to 30 months later). However an understanding of the process serves to shed some light on difficulties with sampling and testing bitumen in the field. Upon investigation into normal practices, it was found that micro-contamination occurs at the loading point of the refinery or bulk storage, but downstream of the loading arm. It occurs in the road vehicle due to current design and operating practices.

The two common road vehicles used to haul bitumen are sprayers and haulers/trailers. For sprayers, it is normal practice for the sprayer bar to be cleaned after use to prevent blocked jets. This is done by circulating a cleaning fluid (solvent) through the spray bar and pump. Medium curing cutter (kerosene or Jet A1) or slow curing cutter (diesel) is used. It was found that up to 20 litres could remain in the system after emptying due to imperfect drainage. The effect of this on viscosity was calculated using the method of Miadonye (equations 8-10 in Miadonye et al., 2000). The effect of 20 litres of kerosene in an 11,500 litre sprayer of bitumen was calculated to reduce viscosity @60^{o}C from 320 to 302 Pa.s, or from 170 to 161 Pa.s. The presence of the cutter might also be seen in the loss of mass on heating, although at these low percentages there is some masking by normal volatiles in the bitumen. The viscosity and loss of mass on heating data in the Appendix suggest that the micro-contamination evaporates over a few days of storage at elevated temperature.

For haulers/trailers loaded from bulk storage, similar micro-contamination could occur since they usually have a pump and lines for transfer and these are similarly cleaned after use. The trailer/hauler tank could also have some residual cutter if it had been previously been cleaned with MC30 (60% bitumen/40% kerosene) or other product containing cutter/diluent. Such cleaning fluids rarely are fully emptied. The effect of 100 litres of MC30 in a 20,000 litre tank of bitumen was calculated to reduce viscosity @ 60^{o}C from 320 to 300 Pa.s, or from 170 to 160 Pa.s.

Sampling procedures in the field are subject to micro-contamination due to the practical difficulties of sampling some of the tanks in service. Cleaning fluid from bitumen transfer lines could contaminate the sample if taken from there. It was calculated that 10 ml of diesel (in a 1 litre tin of bitumen) would reduce viscosity @ 60^{o}C from 381 to 278 Pa.s, and such contamination has been experienced in practice. It is suggested that a code of practice needs to be developed around sampling and the provision of sampling cocks.

4. CONTAMINATION OF BITUMEN

4.1 Detection of Contamination

Contamination in bitumen can be detected by several means. The simplest is the change in properties relative to reference samples (typically viscosity @ 60^{o}C, penetration and/or softening point). Measuring the loss of contaminates during heating (RTFOT test or loss of mass on heating) is very useful. At an advanced level, there is detection by gas chromatography (GC) (Ruud, 1989) and gel permeation chromatography (GPC) with the use of HPLC equipment (Branthaver et al., 1993).

4.2 Effect of Contamination

The effect of contamination on bitumen properties was quantified in the laboratory by adding paraffin and diesel as deliberate contaminants to pure unmodified South African bitumen, and investigating the effect on properties such as penetration and softening point (Table 5, Table 6, Table 7). These tables will serve as a reference for future studies of contamination.

Table 5. Effect of contamination with petrol on bitumen properties.

Test	Base Bitumen 60/70	Base + 0,5% Petrol	Base + 2,0% Petrol
Penetration @25°C (0.1mm)	60	76	153
Softening Point (°C)	52,9	50,9	46,9
GC (area counts)	0	21 700	27 000
RTFOT: mass change (%m/m)		- 0,40%	-1,6%
GC (area counts)	0	0	0
Softening Point (°C)	55,7	55,8	56,3

Note: the base 60/70 used in these 3 tables had been approximately 18 months in cool storage.

Table 6. Effect of contamination with illuminating paraffin on bitumen properties.

Test	Base Bitumen 60/70	Base + 0,5% IP	Base + 2,0% IP
Penetration @25°C (0.1mm)	60	79	130
Softening Point (°C)	52,9	50,4	46,9
GC (area counts)	0	17 400	31 000
RTFOT: mass change (%m/m)		n.a.	n.a.
GC (area counts)	0	0	300
Softening Point (°C)	55,7	55,4	56,2

Note: IP: illuminating paraffin is not dissimilar to Jet A1.

Table 7. Effect of contamination with diesel on bitumen properties.

Test	Base Bitumen 60/70	Base + 0,5% Diesel	Base + 2,0% Diesel
Penetration @25°C (0.1mm)	60	74	117
Softening Point (°C)	52,9	51,0	47,5
GC (area counts)	0	24 800	30 800
RTFOT: mass change (%m/m)		- 0,29%	-1,55 %
GC (area counts)	0	0	n.a.
Softening Point (°C)	55,7	55,9	55,3

The effect of contamination on viscosity was quantified in the laboratory by adding Jet A1 (similar to illuminating paraffin) as deliberate contaminants to pure unmodified Australian bitumen (Table 8).

Table 8. Effect of contamination with Jet A1 (kerosene) on bitumen viscosity.

Test	Base Bitumen Class 320	Base + 2% Jet A1	Base + 4% Jet A1
Viscosity @60 °C (Pa.s)	381	133	78

4.3 Methods to Control Contamination

There is little published to codify the control of contamination in bitumen even though the sources of contamination of bitumen are well known. The bitumen handling codes tend to be focussed on safety (SABITA, 1993), and this is especially true for those derived from the various Mines Acts or Dangerous Goods Acts. There is only limited guidance published on the control of contamination (AIP_CP20, 1993) (AN_10, 1995).

The sampling and testing of bitumen is controlled by test methods for sampling such as those issued by some authorities (WA700.1, 1998), but these are not in widespread use. No protocols exist for long-term bitumen ageing laboratory studies, which makes the laboratory investigation of ageing problematic.

In the light of the findings of this paper, the methods for the control of contamination, and the normal practices of flushing, testing, and sampling of bitumen need revisiting. Specifically a code of practice for the prevention of contamination during haulage is required, as is a test method and protocol for sampling and for the provision of sampling cocks. This should be done to complement other initiatives such as the 2003/4 Sabita investigation into South African bitumen quality haulier involvement in maintaining quality of products (Myburgh, 2003).

It is also considered reasonable to introduce a requirement for periodic retesting of bitumen stored at elevated temperatures in view of the changes in bitumen properties over time that were found in this study. The period for retesting depends on the size of storage and storage temperature, since this affects the rate of deterioration. Because heating costs provide a commercial upper bound to storage temperatures, it is expected that they will be consistent with the temperatures reported in this paper. The proposed retesting requirements of Table 9 were derived from the ageing indices of this paper.

Table 9. Proposed bitumen retesting requirements for bitumen stored at 120-165°C.

Tank size	Maximum storage period between bitumen retesting
Greater than 250 tonnes	45 days
100 tonnes	30 days
25 tonnes	15 days

5. CONCLUSIONS

The studies developed an understanding of bitumen ageing in storage and transport, and found a number of issues with present handling, sampling and testing methods.

- Change in viscosity when ageing in bulk storage was found to occur but be different to that found in ageing small samples in the laboratory.
- The ageing index (ratio of "viscosity aged" to "viscosity unaged") of bitumen stored at elevated temperatures was found to increase by 0.0034 per day in bulk storage, 0.0098 per day for bulk sea transport, 0.0137 per day for road transport, and 0.0168 per day in site storage.
- Micro-contamination occurs as a result of normal practices at the loading point of the refinery or bulk storage, but downstream of the loading arm. It occurs in the road vehicle due to current design and operating practices.
- The effect of contamination on bitumen properties was quantified in the laboratory by adding deliberate contaminants to pure unmodified bitumen and this will serve as a reference for future studies of contamination.

6. REFERENCES

AIP_CP20 (1993) **CP 20 – Safe Handling of Bitumen Products**, Australian Institute Petroleum, Australia.

AN_10 (1995) **Loading Hot Bitumen Products**, Advisory Note 10. AAPA, Australia.

AS2341.4 (1994) **Determination of dynamic viscosity by rotational viscometer**, Australian Standards, Australia.

AS2341.12 (1993) **Determination of penetration**, Australian Standards, Australia.

Branthaver, J. F., Petersen, J., Robertson, R., Duvall, R., Kim, S., Harnsberger, P., Mill, T., Ensley, E., Barbour, F. and Schabron, J. (1993) **Binder characterization and evaluation. Volume 2: Chemistry** SHRP A-368, Strategic Highway Research Program, National Research Council, Washington, DC.

Coe, S. (2003) **Personal Communication**, Bitumen Chemist, Caltex, Cape Town.

Dickinson, E. (1984) **Bituminous Roads in Australia**, Australian Road Research Board, Melbourne.

E&E (2000) **Report of Session 1**, 2nd Eurasphalt & Eurobitume Congress, Barcelona.

Horvathne, E. and Levey, J. (2000) **Structure analysis of road building bitumens – the effect of ageing for the structure; road building case study with modified bitumen** Vol 5.138, 2nd Eurasphalt & Eurobitume Congress, Barcelona.

Miadonye, A., Latour, N. and Puttagunta, V. (2000) **Correlation for viscosity and solvent mass fraction of bitumen-diluent mixtures**, Petroleum Science and Technology, **18,** 1-14.

Myburgh, P. (2003) **Personal communication**, SABITA, Cape Town.

Ruud, O. (1989) **Characterization of bituminous binders by gas chromatography** Vol 5.138, Eurobitume Congress, Madrid.

SABITA (1993) **Bitumen Safety Handbook** Manual 8, Cape Town.

WA 700.1 (1998) **Sampling procedures for bitumens and oils.** Test method. Main Roads Western Australia.

APPENDIX: BITUMEN PROPERTIES DURING ROAD TRANSPORT AND SITE STORAGE

In Australia, a number of bulk loads of Class 320 bitumen were hauled 2400kms from the refinery at Perth (North Fremantle) to a site in Broome by road in 11,000 – 28,000 litre tanks at various times over a year. Testing was done on the bitumen as supplied at the refinery, and after haulage upon arrival at site in Broome, and after site storage on site in Broome. Details of temperatures and haulage are in the text. Each row represents the test results on a single 1 litre retained sample, without duplication. For the viscosity tests, 10% were duplicated for audit purposes. The viscometer was calibrated against reference silicone oil.

Sample	**Time (days)**	**Viscosity @60°C (Pa.s)**	**Loss of mass on heating, %, 163°C, 5 hrs**	**Comment**
Refinery 13/11/01	0	345	0.17	Sampled before loading. Refinery certificate was 340 Pa.s
Site	5	348	0.10	
Site	7	360	0.04	

Sample	**Time (days)**	**Viscosity @60°C (Pa.s)**	**Loss of mass on heating, %, 163°C, 5 hrs**	**Comment**
Refinery 15/11/01	0	317	0.18	Sampled after loading, from a sprayer. Refinery certificate was 340 Pa.s. Suspected partial micro-contamination due bar cleaning fluid residue.
Site	3	299	0.44	Sampled from sprayer. Suspected increased micro-contamination due to additional bar cleaning fluid residue being incorporated during transport. Refinery certificate was 340 Pa.s.

Sample	**Time (days)**	**Viscosity @60°C (Pa.s)**	**Loss of mass on heating, %, 163 °C, 5 hrs**	**Comment**
Refinery 18/9/02	0	330	0.11	Sampled after loading, from a hauler. Refinery certificate of 347 Pa.s. Suspected micro-contamination due bar cleaning fluid residue.
Site – trailer	3	381	0.05	

Sample	Time (days)	Viscosity @60°C (Pa.s)	Loss of mass on heating, %, 163 °C, 5 hrs	Comment
Refinery 29/10/02	0	336	0.11	Sampled before loading. Refinery batch certificate was 342 Pa.s.
Site hauler – trailer 1	3	334	n.a.	Suspected micro-contamination.
Site hauler – trailer 2	3	378	n.a.	
Site – trailer 1	7	363	n.a.	

Sample	Time (days)	Viscosity @60°C (Pa.s)	Loss of mass on heating, %, 163°C, 5 hrs	Comment
Refinery 4/11/02	0	348	0.03	Sampled before loading into sprayer. Refinery certificate was 342 Pa.s.
Site	3	328	n.a.	Sampled from sprayer. Suspected micro-contamination of sprayer by cleaning fluid.

Sample	Time (days)	Viscosity @60°C (Pa.s)	Loss of mass on heating, %, 163°C, 5 hrs	Comment
Refinery – batch 1 14/11/02	0	334	0.05	Sampled before loading. Batch 1 refinery certificate was 320 Pa.s
Site	5	343	0.09	Batch 1
Refinery – batch 2 14/11/02	0	349	0.09	Batch 2. Sampled before loading. Refinery certificate was 342 Pa.s
Site	3	356	0.09	Batch 2
Site	5	364	0.17	Batch 2