CETANZ

Civil Engineering Testing Association of New Zealand

TECHNICAL REPORT ON

PENETRATION OF BITUMINOUS MATERIALS

CETANZ Technical Report	**TR 5**
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Report Date – First Issue	**August 2011**
Report Revision Date	**-**
Revision Number	**-**
Associated Test Method(s)	**ASTM D5**

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Inter-Laboratory Study of ASTM D5 “Standard Test Method for the Penetration of Bituminous Materials”

August 2011

1 ACKNOWLEDGEMENTS

The support of Fulton Hogan Limited, Nelson region, who prepared and distributed the test specimens, is gratefully acknowledged.

The funding provided by Z Energy Limited for the analysis of the test results and preparation of the report is gratefully acknowledged.

2 INTRODUCTION

A bitumen proficiency test round was arranged following concerns over the bitumen Penetration test (ASTM D5) returning differing results between laboratories with ostensibly the same material. Sixteen laboratories offering the Penetration test were identified using the IANZ web site www.ianz.govt.nz and they were invited to participate. All invited laboratories responded positively to the invitation.

In addition to the Penetration test, laboratories were invited to carry out the Softening Point test (ASTM D36) and to determine the viscosity at 60° C using their preferred method.

Two bitumen samples were distributed to the participating laboratories, nominally 180/20 and 40/50 grades. Laboratories were requested to prepare two test specimens for both materials and test in duplicate.

3 PARTICIPANTS

The following laboratories were invited to, and participated in the proficiency test round:

Bitumen and Pavement	Auckland
Downer	Tauranga
Downer	Christchurch
Downer	Auckland
Fulton Hogan	Christchurch
Fulton Hogan	Hamilton
Fulton Hogan	Dunedin
Fulton Hogan	Auckland
Fulton Hogan	Nelson
Higgins Laboratory	Napier
Independent Petroleum Laboratory	Ruakaka
Intertek Testing Services	Ruakaka
Isaac Construction Company	Christchurch
Opus Auckland Laboratory	Auckland
Opus Central Laboratories	Wellington
Opus Hamilton Laboratory	Hamilton

Laboratories were randomly assigned an identifying number to preserve anonymity.

4 SAMPLE PREPARATION

A proficiency test exercise such as this is only useful if identical samples are distributed to each of the participants. Consequently great care was taken to ensure, as much as possible, that the set of individual test samples for each grade of bitumen were identical.

Samples were prepared at Fulton Hogan Port Nelson bitumen plant. The following technique was used:

1. Bitumen was transferred between a bulk storage tank and a day tank for each grade of bitumen. For the 180/200 the bulk storage tank held a single recent ship discharge, hence represented a single "batch" of material.

2. Part way through the bitumen transfer an approximately 20-L sample was drawn from the transfer line. This samples was rendered homogeneous by several rapid transfers backwards and forwards (i.e. "boxing") into another 20-L sample container. Between 30 and 40 0.5-L test samples were then poured from the 20-L bulk samples.

Samples were covered, allowed to cool and sealed. The 180/200 bitumen was labelled as "A", and the 40/50 as "B". Samples were distributed to participating laboratories.

5 TESTING

All laboratories were requested to carry out the Penetration test to ASTM D5 for both samples, A and B. In addition, laboratories were requested to prepare two test specimens for both bitumens, to provide duplicate test results.

It is standard practice to carry out three determinations, using three Penetration needles for each test. The Penetration value for each sample is then the average of the three results obtained. Participants were requested to provide the three individual results for each needle to allow the analysis of unrounded results for each sample.

6 RESULTS

Results were provided promptly from each laboratory.

Table 1: Penetration Test Results (all laboratories)

Laboratory	Operator	Sample A, Test 1				Sample A, Test 2				Sample B, Test 1				Sample B, Test 2			
		1	2	3	Mean	1	2	3	Mean	1	2	3	Mean	1	2	3	Mean
1	1	172	171	169	170.7	171	170	170	170.3	45.0	44.0	44.0	44.3	45.0	44.0	44.0	44.3
2	1	180	184	182	181.8	177	177	176	176.5	44.7	45.1	44.6	44.8	45.2	45.0	45.3	45.2
3	1	186	183	182	183.7	187	183	183	184.3	46.0	45.0	44.0	45.0	45.0	44.0	44.0	44.3
4	1	180	179	180	179.2	181	185	180	181.5	46.0	46.0	47.0	46.3	47.0	46.0	47.0	46.7
4	2	181	188	185	184.3	180	188	183	183.5	45.0	47.0	45.0	45.7	44.0	46.0	44.0	44.7
5	1	164	168	150	160.7	150	150	140	146.7	15.0	18.0	10.0	14.3	22.0	26.0	28.0	25.3
6	1	181	182	182	181.7	182	182	181	181.7	45.0	44.0	44.0	44.3	45.0	45.0	45.0	45.0
7	1	173	172	173	172.7	170	171	170	170.3	43.0	43.0	43.0	43.0	38.0	40.0	40.0	39.3
8	1	179	170	182	177.0	185	178	174	179.0	45.0	46.0	45.0	45.3	46.0	44.0	45.0	45.0
9	1	181	177	175	177.7	175	170	170	171.7	43.0	43.0	43.0	43.0	44.0	43.0	42.0	43.0
10	1	179	180	179	179.3	182	184	180	182.0	44.0	44.0	42.0	43.3	44.0	46.0	44.0	44.7
11	1	175	175	175	175.0	175	176	176	175.7	45.0	46.0	46.0	45.7	45.0	45.0	47.0	45.7
11	2	176	175	173	174.7	172	171	170	171.0	43.0	43.0	44.0	43.3	44.0	44.0	44.0	44.0
12	1	178	176	173	175.7	176	170	171	172.3	43.0	43.0	45.0	43.7	45.0	44.0	45.0	44.7
13	1	180	177	180	179.0	179	180	182	180.3	45.0	45.0	43.0	44.3	42.0	42.0	45.0	43.0
14	1	173	172	172	172.3	173	174	172	173.0	44.0	44.0	44.5	44.2	45.0	43.5	44.0	44.2
15	1	171	169	172	170.3	167	170	174	170.3	43.0	43.0	42.5	42.8	43.5	42.0	43.0	42.8
16	1	177	177	175	176.3	170	176	176	174.0	43.0	45.0	44.0	44.0	44.0	44.0	44.0	44.0
Grand average					176.2				174.7				42.6				43.1
Standard Deviation (P)					5.53				8.33				6.94				4.56
ASTM D5-06 SD (section 11.1)					8.31				8.23				2.50				2.50
ASTM D5-06 acceptable ranges (section 11.2)					24				23				7				7
Minimum Penetration					164				163				39				40
Maximum Penetration					188				186				46				47

7 ANALYSIS

7.1 REVIEW OF DATA

Table 1 above provides the results as returned by the laboratories for the Penetration test. Individual test results are provided to three significant figures for clarity, irrespective of how they were reported. Mean Penetration values are based on unrounded individual test results.

The Standard Deviation for each test was calculated and compared with the maximum value allowed by ASTM D5. The allowable limits of ASTM D5 were applied to the sample means, allowing an acceptable results range for each sample to be calculated. Laboratories 5 (both grades) and 7 (40/50 test B2) returned Penetration values that fell outside of the acceptable range of ASTM D5. However this analysis is based on the assumption that the set of results are statistically identical. Further analysis of the data is required to identify and eliminate test results that are statistical outliers.

ASTM E691 was used to analyse the test results. The data set was analysed to determine if any results should be excluded as outliers.

E691 determines consistency using “h” and “k” statistics. The h statistic measures the between-laboratory consistency for each result. The k statistic measures the within-laboratory for the data submitted by each laboratory.

Table 5 of E691 lists critical values for the h and k statistics at the 0.5% significance level. Calculation of the h and k statistics for the Penetration results can be compared with the critical values to identify outliers that should be excluded from the analysis. These comparisons were done and plots prepared comparing the h and k statistics against the E691 critical values.

Review of the h criterion charts (figures 1 - 4) shows that the data from laboratory 5 can be considered an outlier and excluded from the data sets.

Review of the k criterion charts shows that the individual results (i.e. for each Penetration needle) are within acceptable limits although the data laboratory 5 appears to show more variability than other laboratories.

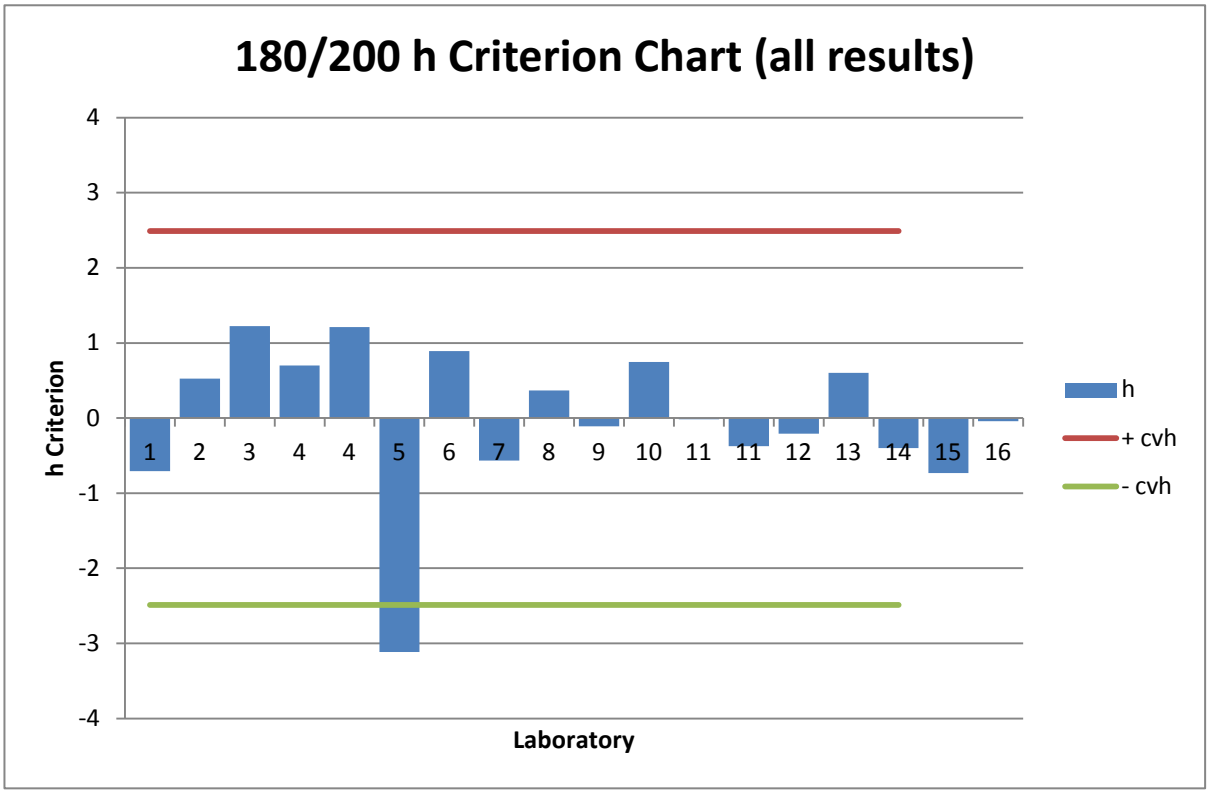

Figure 1: **180/200 h Criterion Chart (between laboratories)**

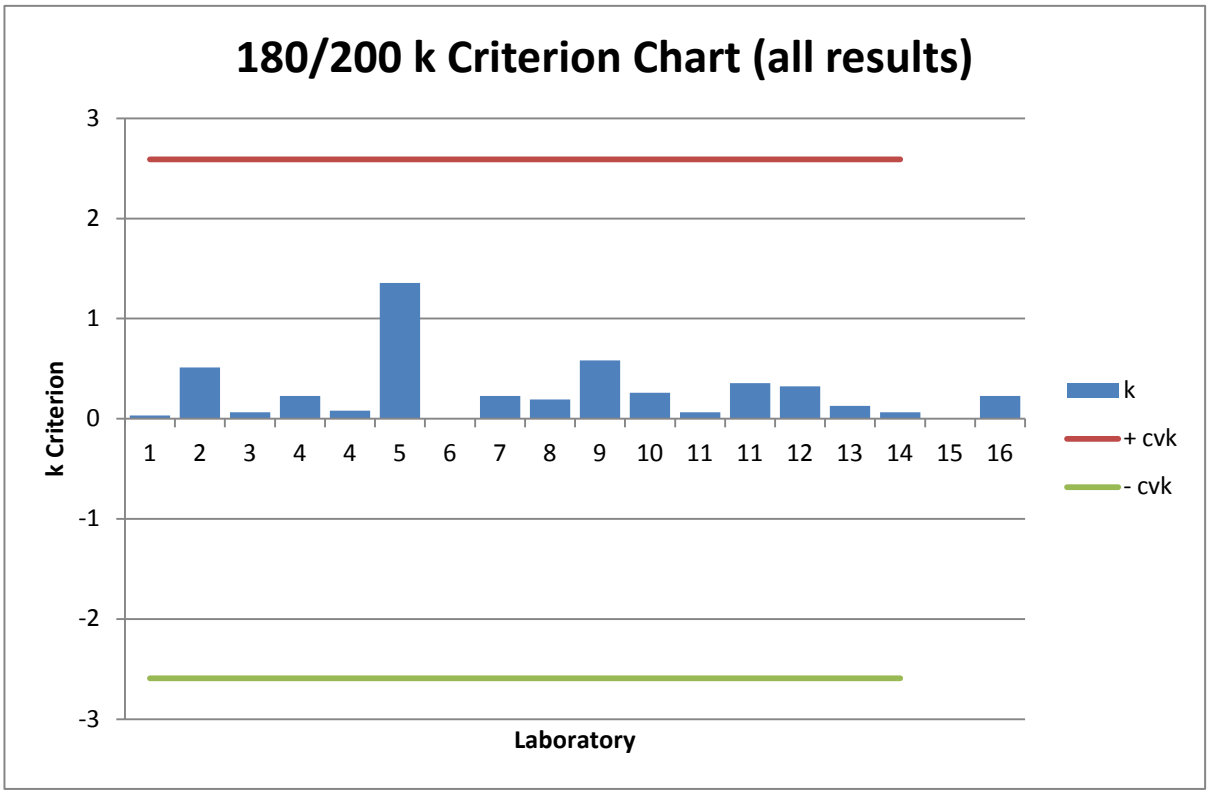

Figure 2: **180/200 k Criterion Chart (within laboratory)**

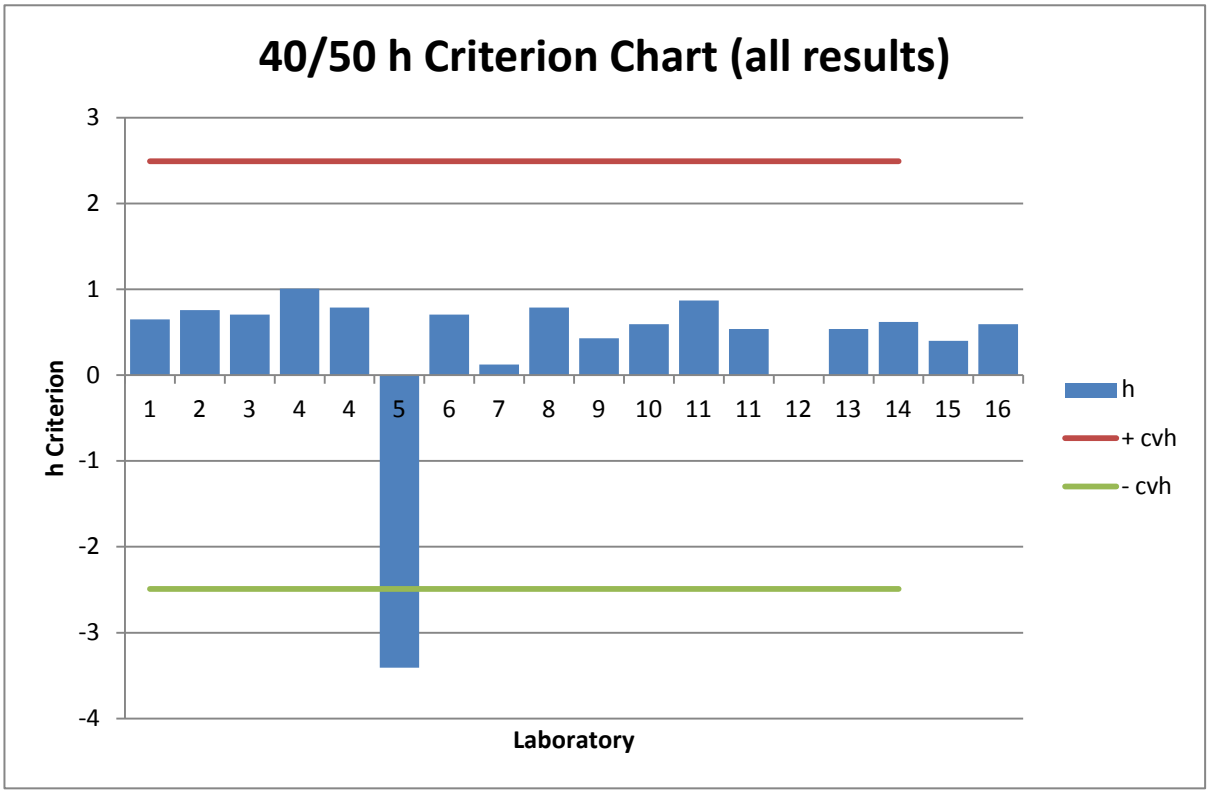

Figure 3 **40/50 h Criterion Chart (between laboratories)**

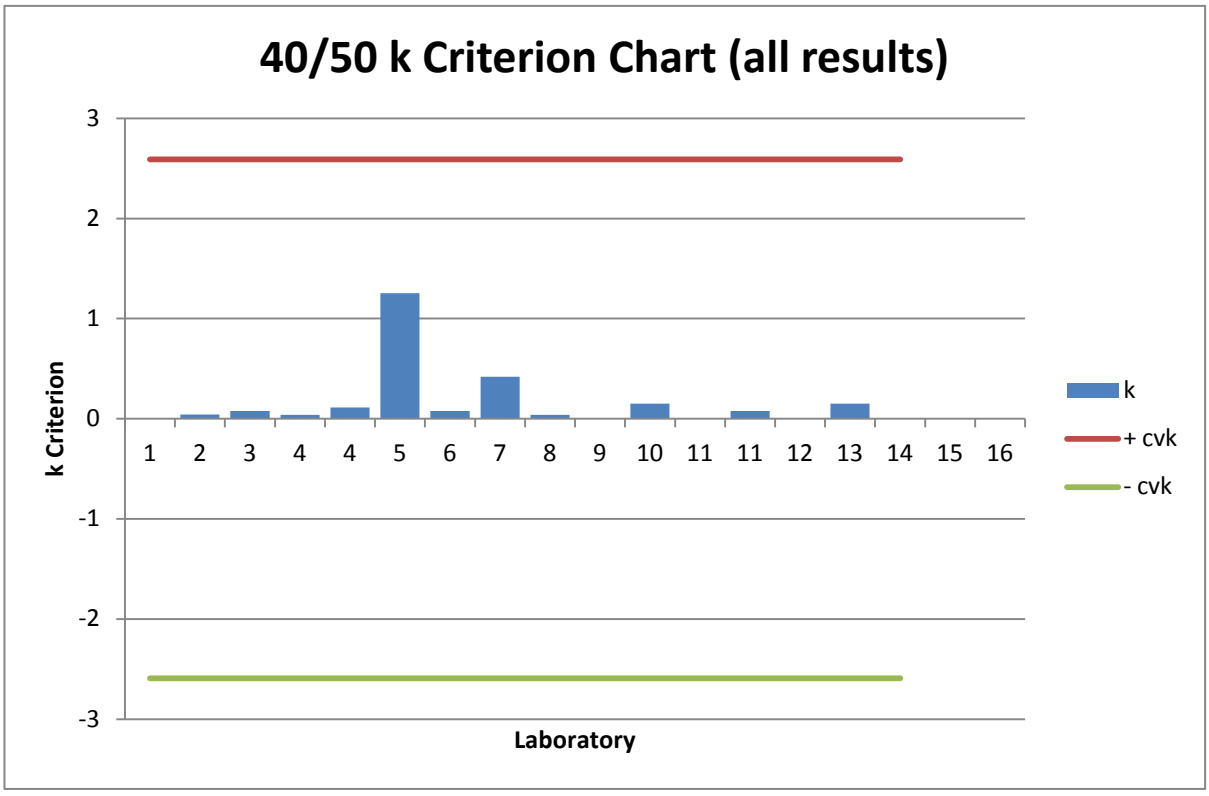

Figure 4 **40/50 k Criterion Chart (within laboratory)**

7.2 ASSESSMENT

The results for laboratory 5 were excluded from the data sets and analysis was repeated. Table 2 (over) contains the data and adjusted calculations. Review of the results shows that the mean Penetration result for 40/50 test B2 reported by laboratory 7 continues to fall outside the ASTM acceptable ranges.

Amended h and k statistic plots were prepared. Refer to figures 5 – 8.

The Penetration result for the 40/50-grade bitumen (sample B) reported by laboratory 7 marginally fails the h criterion limits. Consequently this result could be considered to be an outlier, but given the compliance with the other criteria it was decided to leave the result in the analysis. However it is recommended that laboratory 7 review its procedures in respect of the Penetration test.

Repeatability and reproducibility values can be calculated from the test results and compared with the values in ASTM D5. Refer to Table 2 below.

Table 2: Repeatability and Reproducibility Results

Material	Repeatability		Reproducibility	
	Interlab	ASTM D5	Interlab	ASTM D5
180/200 Bitumen (sample A)	5.3	12	13.2	24
40/50 Bitumen (sample B)	2.2	2	3.7	7

The repeatability result obtained during the interlab exceeds the ASTM D5 value. Deleting laboratory 7 results for the 40/50 bitumen (Sample B) lowers the interlab repeatability result to 1.3, and the reproducibility result to 2.8. Both results are substantially less that the ASTM D5 values.

Table 3: Penetration Test Results (laboratory 5 excluded)

Laboratory	Operator	Sample A, Test 1				Sample A, Test 2				Sample B, Test 1				Sample B, Test 2			
		1	2	3	Mean	1	2	3	Mean	1	2	3	Mean	1	2	3	Mean
1	1	172	171	169	170.7	171	170	170	170.3	45	44	44	44.3	45	44	44	44.3
2	1	180	184	182	181.8	177	177	176	176.5	44.7	45.1	44.6	44.8	45.2	45	45.3	45.2
3	1	186	183	182	183.7	187	183	183	184.3	46	45	44	45.0	45	44	44	44.3
4	1	180	179	180	179.2	181	185	180	181.5	46	46	47	46.3	47	46	47	46.7
4	2	181	188	185	184.3	180	188	183	183.5	45	47	45	45.7	44	46	44	44.7
5	1																
6	1	181	182	182	181.7	182	182	181	181.7	45	44	44	44.3	45	45	45	45.0
7	1	173	172	173	172.7	170	171	170	170.3	43	43	43	43.0	38	40	40	39.3
8	1	179	170	182	177.0	185	178	174	179.0	45	46	45	45.3	46	44	45	45.0
9	1	181	177	175	177.7	175	170	170	171.7	43	43	43	43.0	44	43	42	43.0
10	1	179	180	179	179.3	182	184	180	182.0	44	44	42	43.3	44	46	44	44.7
11	1	175	175	175	175.0	175	176	176	175.7	45	46	46	45.7	45	45	47	45.7
11	2	176	175	173	174.7	172	171	170	171.0	43	43	44	43.3	44	44	44	44.0
12	1	178	176	173	175.7	176	170	171	172.3	43	43	45	43.7	45	44	45	44.7
13	1	180	177	180	179.0	179	180	182	180.3	45	45	43	44.3	42	42	45	43.0
14	1	173	172	172	172.3	173	174	172	173.0	44	44	44.5	44.2	45	43.5	44	44.2
15	1	171	169	172	170.3	167	170	174	170.3	43	43	42.5	42.8	43.5	42	43	42.8
16	1	177	177	175	176.3	170	176	176	174.0	43	45	44	44.0	44	44	44	44.0
Grand average					177.1				176.3				44.3				44.1
Standard Deviation (P)					4.16				4.95				1.02				1.53
ASTM D5-06 SD (section 11.1)					8.36				8.32				2.5				2.5
ASTM D5-06 acceptable ranges (section 11.2)					24				24				7				7
Minimum Penetration					165				164				41				41
Maximum Penetration					189				188				48				48

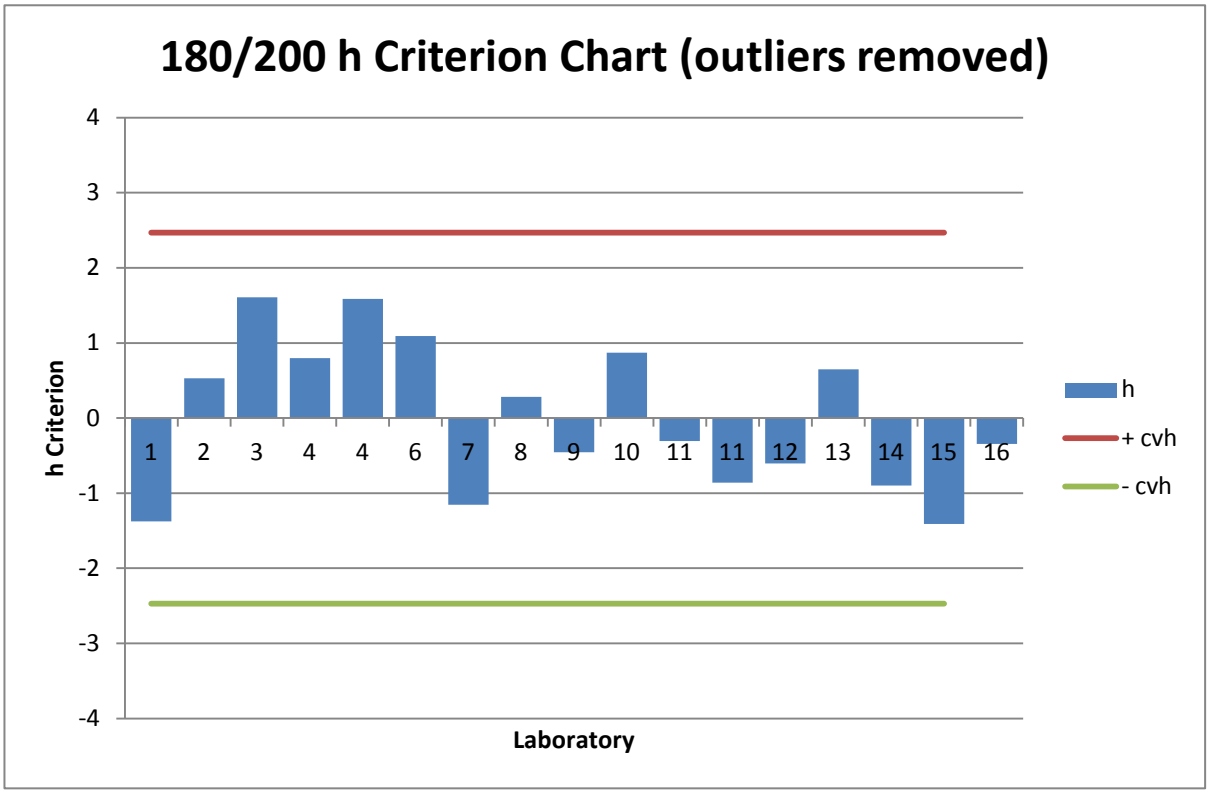

Figure 5: **180/200 h Criterion Chart (between laboratories, outliers removed)**

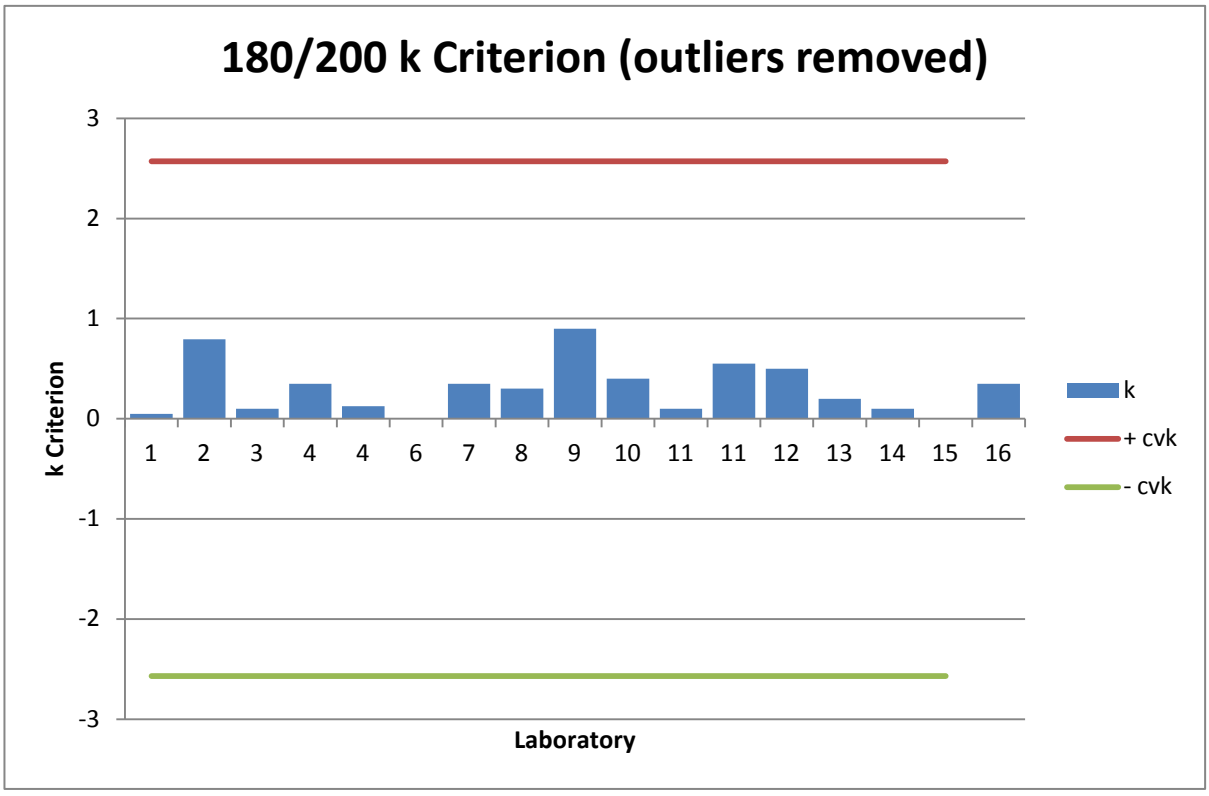

Figure 6: **180/200 k Criterion Chart (within laboratory, outliers removed)**

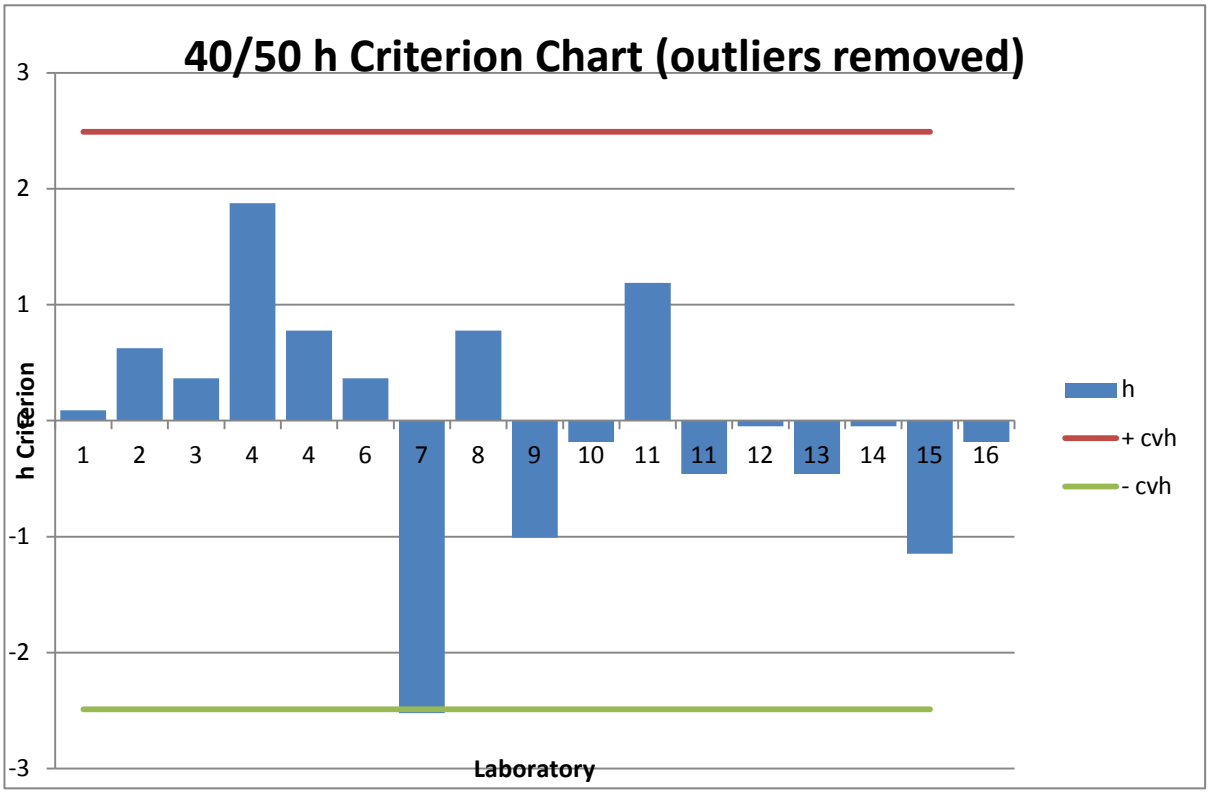

Figure 7 **40/50 h Criterion Chart (between laboratories, outliers removed)**

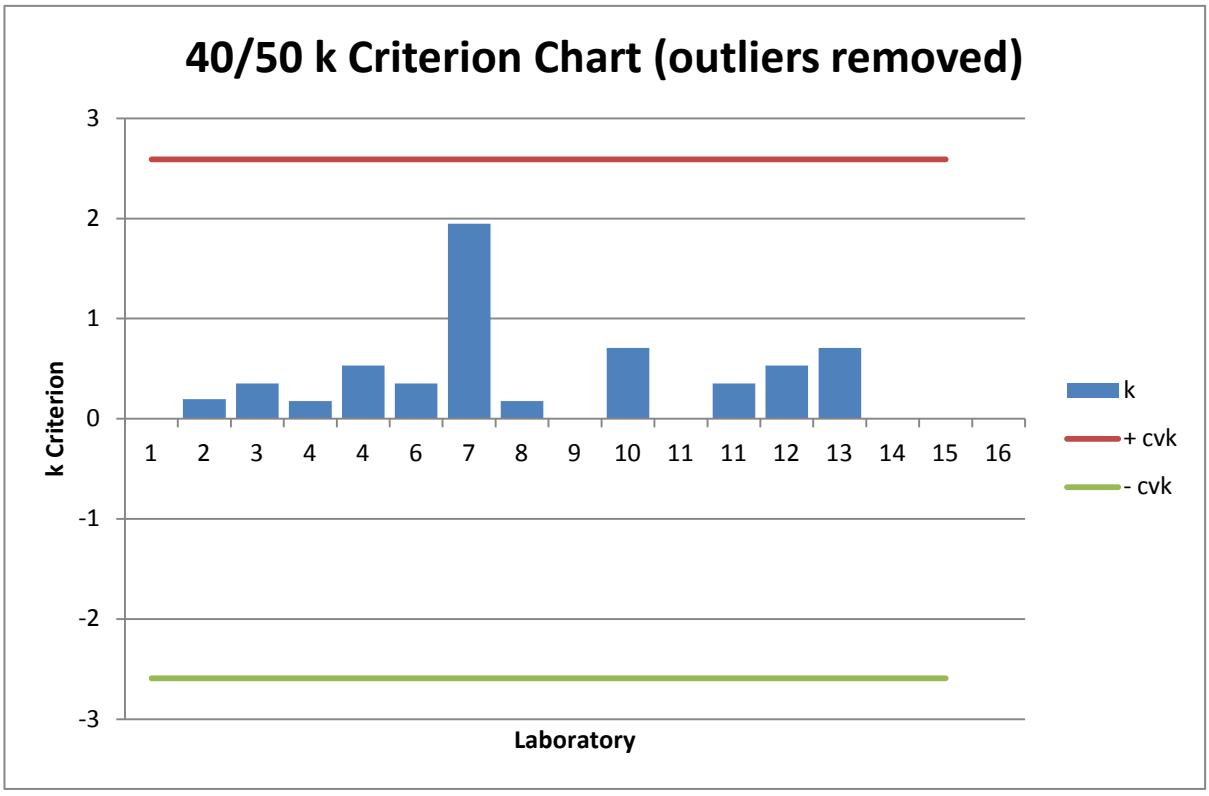

Figure 8 **40/50 k Criterion Chart (within laboratory, outliers removed)**

8 OTHER TESTS

Laboratories were encouraged to carry out further testing on the bitumen samples to maximise the value of the interlaboratory round. These results are tabulated below.

Due to the limited number of results reported for the other tests no analysis was carried out.

Table 4: Other Test Results

Laboratory	Sample A						Sample B					
	Softening Point	Viscosity					Softening Point	Viscosity				
	ASTM D36	ASTM D2170	ASTM D2171	AS 2341.2	AS 2341.3	ASTM D4402	ASTM D36	ASTM D2170	ASTM D2171	AS 2341.2	AS 2341.3	ASTM D4402
	(°C)	(mm^2/s)	Pa.s	Pa.s	(mm^2/s)	Pa.s	(°C)	(mm^2/s)	Pa.s	Pa.s	(mm^2/s)	Pa.s
1	42.1			60.1		62.25	54.1			522.9		555
2	41.4						53.8					
3												
4	40.2					53.5	52.8					509
5												
6												
7												
8	40.6						53.8					
9												
10	41.0				58300		53.4				562000	
11		60300										
12												
13	40.4						53.8					
14												
15	40.7		61.5				52.0			530		
16	42.2		58.7				55.4		521			

9 CONCLUSIONS

The results obtained during this interlaboratory series for ASTM D5 Penetration shows that New Zealand bitumen testing laboratories are generally testing within the repeatability and reproducibility limits of ASTM D5. Exceptions to this are laboratories 5 and 7.

Laboratory 5 appeared to have a systematic testing error and an investigation is recommended.

Laboratory 7 results for the 40/50 (sample B) bitumen marginally failed the between-laboratory “h” criterion.

10 RECOMMENDATIONS

On the basis that the Penetration test used for bitumen acceptance testing it is recommended that this interlaboratory exercise is repeated on an annual basis.

GM Bosma

September 2011