NZ TRANSPORT AGENCY
WAKA KOTAHI

TECHNICAL MEMORANDUM

To NZTA Network Management Consultants and Contractors

Cc NZTA Regional Operations Managers and Area Managers
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Subject Procedure for Assessing Bitumens for Acceptance by the Penetration Test

Highways and Network Operations Technical Memorandum No. TM6004

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1.0 Introduction

The penetration test (ASTM D5) carried out at 25°C defines New Zealand bitumen grades (NZTA specification M1: 2010), and the test can be used to make decisions as to whether to accept or reject a bitumen on the basis of the upper and lower limits for the grade. However, the inherent variability of the penetration test method[1] means that it is possible to reject a bitumen for which the true value[2] of penetration meets the specification requirements, and also to accept as satisfactory a bitumen for which the true value of penetration is outside the specification.

As an example, consider a 180/200 bitumen for which the true value of penetration is 190 penetration units[3]. Figure 1 shows the single test result probability distribution for a pen 190 bitumen, together with the upper and lower limits of the New Zealand standard, NZTA M1: 2010.

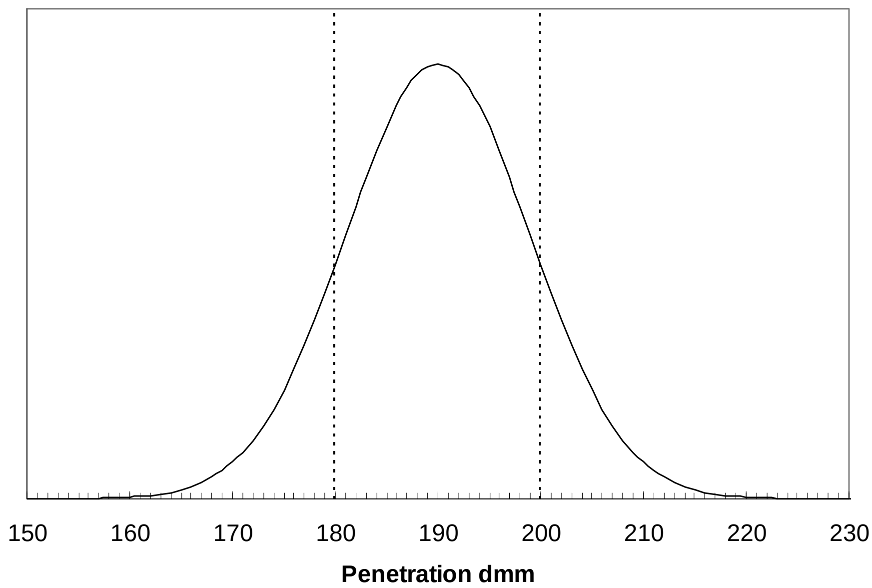

Figure 1: Probability distribution of a single penetration result for a bitumen with a “true” penetration of 190. Lower and upper specification limits are indicated by broken lines.

The areas under the tails of the distribution outside the specification limits indicates that there is a 26.7% chance of a single penetration result falling outside the specification limits.

The variability of the penetration test and the range of the specified penetration limits vary from grade to grade. Table 1 indicates the probability, for the different bitumen grades, of a penetration result falling outside the specification range if the true penetration is in the middle of the range.

[1] For variability data see ASTM specification D5: 2006 and Table 2 below, taken from this specification.
[2] The penetration of a bitumen can only be measured by the standard penetration test, for which the results will vary from one measurement to another. Hence, for practical purposes we define the “true value” as “the value towards which the average of single results obtained by *n* laboratories tends, as *n* tends towards infinity” (International Standard EN ISO 4259:2006). The term “true penetration” refers the true value of the bitumen penetration.
[3] Penetration units are tenths of millimetres, i.e. decimillimetres (dmm).

Bitumen Grade	Percent Probability of Midgrade Penetration Result Outside Specification Limits
40/50	4.6
60/70	6.9
80/100	1.2
130/150	12.2
180/200	26.7

Table 1: Effect of test variability and specification limits on acceptance/rejection of binder

2.0 Development of Acceptance/Rejection Limits

We look to set acceptance limits outside the specification limits by amounts that depend on the variability of the test method. In doing this we have to reckon with two undesirable possibilities:

1. Rejecting a bitumen whose true penetration is actually within the specification limits. Because the penetration test is inherently variable there is always this possibility. Setting the acceptance limits well above and well below the specification maximum and minimum reduces this possibility (Statistically speaking this is referred to as a Type I error).

2. Accepting a bitumen that is actually outside the specification limits (Type 2 error). Setting the acceptance limits well above and well below the specification maximum and minimum increases the chance of this type of error.

The setting of rejection and acceptance limits is therefore a compromise between these two risks.

3.0 Variability Values of Penetration Test Results

The bitumen acceptance procedures proposed below are based on the latest single operator and multilaboratory precisions for the 25°C penetration test published in the American Society for Testing and Materials Specification ASTM D5-06e1. The precisions, in terms of standard deviations of results, are listed in Table 2.

	Single Operator Precision, $S_r(P)$	Multilaboratory Precision, $S_R(P)$
$P \leq 60$	0.8	2.5
$P > 60$	0.8 + 0.03(P-60)	2.5 + 0.05(P-60)

Table 2: Precision levels for ASTM penetration test ASTM D5-06e1. Penetration values (P) and precisions (standard deviations) are in standard units, tenths of millimetres (decimillimetres, dmm).

If a test procedure is repeated k times in the one laboratory, the multilaboratory precision of the test, $S_{R,k}(P)$ becomes

$$S_{R;k}(P) = \sqrt{S_R^2(P) - \left(1 - \frac{1}{k}\right) S_r^2(P)} \qquad (2.1)$$

4.0 Acceptance Procedure

The following procedure is proposed to deal with cases where an *in* specification bitumen has been retested after delivery of a cargo or production batch from the manufacturer and found to be outside the NZTA M1: 2010 specification limits (bitumens supplied with a penetration, *at the point of manufacture*, outside the NZTA M1: 2010 specification limits will not be accepted as meeting the specification under any circumstances).

The average of the penetration at the point of manufacture and one or (if necessary) two retest values (from different laboratories) are averaged and the result compared to acceptance limits as set out below, (Note: the penetration result from a given laboratory may itself be the mean of several replicates).

4.1 Penetration verification tests

Tests shall be carried out by laboratories which are IANZ (International Accreditation New Zealand) accredited to perform the ASTM D5 penetration test or from overseas laboratories accredited to an equivalent international standard level.

4.2 Acceptance limits

Penetration tests performed to verify that a bitumen complies with NZTA M1: 2010 shall be assessed according to whether or not the result falls within the acceptance/rejection limits listed in Table 3.

Bitumen Grade	Lower Acceptance Limit	Upper Acceptance Limit
40/50	37	53
60/70	57	74
80/100	75	105
130/150	121	158
180/200	170	210

Table 3: Acceptance limits for average penetration results for different penetration grades.

These limits have been established so that acceptance/rejection probabilities for the different grades are, as far as possible, the same (allowing for the need to report penetrations in integer units*).* The upper and lower acceptance limits have been chosen so that a bitumen with a true penetration equal to the upper specification limit has only a 6.8% chance of being rejected on the basis of the average of two penetration test results from independent laboratories. Similarly a bitumen of true penetration equal to the lower specification limit has 4.8 % chance of rejection. A bitumen that has a true penetration equal to the midpoint of a given specification range has a less than 0.2% chance of being rejected on the basis of the average of two test results.

Probabilities of rejection of an in specification bitumen on the basis of the average of three test results from independent laboratories are lower (see, for example, Table 5).

Inasmuch as the acceptance limits lie outside the specification limits they represent the *maximum* probability of the material not meeting specification that the recipient of the material is prepared to accept. A bitumen will not be accepted on the basis of a penetration testing result outside the acceptance limits, no matter how close the penetration value is to falling within the acceptance range.

4.3 Acceptance based on Two Test Results from Independent Laboratories

If the two penetration results are P_1 (penetration at the point of manufacture) and P_2, (retest penetration) the procedure is as follows:

4.3.1 Calculate the average of the two penetrations:

$$\bar{P} = \frac{P_1 + P_2}{2} \qquad (4.1)$$

4.3.2 Calculate the multilaboratory precision, $S_R(\bar{P})$, and single operator precision, $S_r(\bar{P})$ for penetration $\bar{P}$.

$$\begin{aligned} S_R(\bar{P}) &= 2.5 & \bar{P} &\leq 60 \\ &= 2.5 + 0.05(\bar{P} - 60) & \bar{P} &> 60 \\ S_r(\bar{P}) &= 0.8 & \bar{P} &\leq 60 \\ &= 0.8 + 0.03(\bar{P} - 60) & \bar{P} &> 60 \end{aligned} \qquad (4.2)$$

4.3.3 Calculate the precision, $S_{R;k1,k2}(\bar{P})$ for the difference $|P_1 - P_2|$, where k1 and k2 are the number of repetition measurements used in obtaining the values P_1 and P_2 respectively.

$$S_{R;k1,k2}(\bar{P}) = \sqrt{S^2_{R;k1}(\bar{P}) + S^2_{R;k2}(\bar{P})} \qquad (4.3)$$

where $S_{R;k1}(\bar{P})$ and $S_{R;k2}(\bar{P})$ are defined as in equation (2.1).

4.3.4 If $|P_1 - P_2| > 1.96\ S_{R;k1,k2}(\bar{P})$, (4.4)

the difference between the two penetration results is too large (at the 95% probability level) for an assessment of the bitumen to be made. With the size of the difference between the penetration values being improbably large, at least one of the values is likely to be in error. It is, however, impossible to tell which of the two is the more accurate unless a third, independent, test is carried out (see Section 4.5 below).

Note: If k1 = k2 = 1, expression (4.4) reduces to

$$|P_1 - P_2| > 2.772\ S_R(\bar{P}) \qquad (4.4a)$$

4.3.5 If, $|P_1 - P_2| \leq 1.96\ S_{R;k1,k2}(\bar{P})$ (4.5) accept the bitumen if $A_2 \le \bar{P} \le A_1$ (where A_1 and A_2 are the upper and lower acceptance limit values from Table 3). Otherwise, reject the bitumen as not meeting specification requirements.

For k1=k2=1, condition (4.5) becomes

$$\left| P_1 - P_2 \right| \le 2.772 \; S_R\left(\bar{P}\right) \qquad (4.5a)$$

4.4 Acceptance based on Three Test Results from Independent Laboratories

In the event of failure of the mean from two independent accredited laboratories to fall within the Table 3 limits, the recipient and supplier may agree to obtain a further test result from a further independent laboratory. Samples tested by the third laboratory shall be taken from the initial material supplied for assessment, with a minimum of further heating.

The penetration results, P_1, P_2 and P_3, *must* be from three independent laboratories.

4.4.1 Calculate the average of the three penetrations:

$$\bar{P} = \frac{P_1 + P_2 + P_3}{3} \qquad (4.6)$$

4.4.2 Calculate the multilaboratory precision, $S_R(\bar{P})$, for the new value of $\bar{P}$, as in 4.3.2.

4.4.3 Assess for the presence of an outlier result as follows:

(i) Let $\left| P_a - \frac{P_b + P_c}{2} \right|$ be the maximum value of

$$\left| P_1 - \frac{P_2 + P_3}{2} \right|, \left| P_2 - \frac{P_3 + P_1}{2} \right| \text{ and } \left| P_3 - \frac{P_1 + P_2}{2} \right|.$$

(ii) Calculate the precision $S_{R;ka;kb,kc}(\bar{P})$ for the quantity $\left| P_a - \frac{P_b + P_c}{2} \right|$, where ka, kb and kc are the number of repetition measurements used in obtaining the values P_a, P_b and P_c respectively.

$$S_{R;ka;kb,kc}(\bar{P}) = \sqrt{S^2_{R;ka}(\bar{P}) + \frac{S^2_{R;kb}(\bar{P}) + S^2_{R;kc}(\bar{P})}{4}} \qquad (4.7)$$

(iii) If $\left| P_a - \frac{P_b + P_c}{2} \right| > 1.96\, S_{R;ka;kb,kc}(\bar{P})$, (4.8)

P_a is an outlier result. Calculate a new average penetration, $\bar{P} = \frac{P_b + P_c}{2}$, and retest as for 4.3.3 and subsequent instructions.

Note: If ka = kb = kc =1, expression (4.7) reduces to

$$\left| P_a - \frac{P_b + P_c}{2} \right| > 2.4005\ S_R(\bar{P}) \quad (4.8a)$$

4.4.4 If $\left| P_a - \frac{P_b + P_c}{2} \right| \leq 1.96\ S_{R;ka;kb,kc}(\bar{P})$, (4.9)

or, if ka = kb = kc = 1, $\left| P_a - \frac{P_b + P_c}{2} \right| < 2.4005\ S_R(\bar{P})$ (4.9a)

accept the bitumen if $A_2 \leq \bar{P} \leq A_1$. (4.10)

Otherwise reject the bitumen as not meeting specification.

In all instances the mean of *all* penetration results for the bitumen shall be assessed as falling within the acceptance/rejection limits for the penetration grade. A penetration result shall only be excluded from contributing to the mean if it fails to meet the outlier test (expression 4.8 or 4.8a)

5.0 Discussion of the Statistical Basis of the Test Procedures

5.1 Assigning Acceptance/Rejection Penetration Limits

The statistical assessments of the acceptance/rejection procedures described in Sections 4.0 and 5.0 are a special case of general rules for assessing petroleum products (International Standard EN ISO 4259:2006). The methodology has had to be adjusted slightly to allow for the fact that the variability of the penetration test depends on the size of the penetration value.

If P_U and P_L are the upper and lower specification limits for a particular penetration grade of bitumen, and A_1 and A_2 are the upper and lower acceptance/rejection limits, we write for the purposes of discussion:

$$A_1 = P_U + K_U S_R(P_U) \quad (5.1)$$

$$A_2 = P_L - K_L S_R(P_L) \quad (5.2)$$

$S_R(P_U)$ and $S_R(P_L)$ are the multilaboratory precision standard deviations for a single penetration test (S_R) at penetration values P_U and P_L, calculated according to the formulae in Table 2. K_U and K_L are therefore constants indicating the number of multilaboratory standard deviations between the respective specification penetrations and acceptance/rejection penetration levels.

In the present case A_1 and A_2 have been chosen on the basis of the most extreme test values that a client is likely to be prepared to accept for a 180/200 bitumen (A_1 = 210 and A_2 = 170).

We therefore have $$K_U = \frac{A_1 - P_U}{S_R(P_U)} = \frac{210 - 200}{S_R(200)} = \frac{10}{9.55} = 1.0471 \qquad (5.3)$$

Similarly $$K_L = \frac{10}{8.5} = 1.1765 \qquad (5.4)$$

Referring to tables of the normal probability distribution this means that A_1 and A_2 have been chosen so that a bitumen with a true penetration of 200 dmm has an 85.2% chance of being accepted on the basis of a single penetration test result, i.e. there is approximately a 14.8 % chance of a penetration result above 210. Similarly a bitumen of true penetration 180 dmm has an 87.5 % chance of testing at 170 dmm or above (12.5% chance of rejection).

Values of A_1 and A_2 for other grades of bitumen (Table 3) were obtained by applying equations 5.1 and 5.2, calculating the appropriate values of $S_R(P_U)$ and $S_R(P_L)$, and using the same values of K_U and K_L (equations 5.3 and 5.4). Values of A_1 were rounded up to the nearest integer, and A_2 down to the nearest integer, since penetration results are reported to the nearest integer.

According to International Standard EN ISO 4259:2006, this provides a slightly more rigorous requirement than is typical for petroleum products. The default procedure, in the absence of a different agreement between supplier and recipient, is to set upper and lower acceptance limits so that a material with a true value of the specification property has a 95% chance of acceptance. This amounts to writing

$$K_L = K_U = 1.645 \qquad (5.5)$$

For the NZTA M1: 2010 specification, this would produce the following acceptance/rejection limits (Table 4).

Bitumen Grade	Lower Acceptance Limit	Upper Acceptance Limit
40/50	35	55
60/70	55	75
80/100	74	108
130/150	120	162
180/200	166	216

Table 4: Acceptance limits for different penetration grades based on a single penetration test result with a 5% chance of rejection of a bitumen with a true penetration at the upper or lower limit of the specification range. These are *not* the proposed limits.

Accepting these wider limits would reduce the chance of rejecting a bitumen that is within specification limits, but it would also increase the chance of accepting a bitumen with a true penetration outside the limits (see 2.0 above). The chance of rejecting a within-specification bitumen is reduced by testing at two or three laboratories.

5.2 Results from Independent Laboratories

The mean value of the standard deviation for several laboratories has a lower standard deviation than that of one laboratory; hence it will typically be closer to the true penetration.

For two penetrations P_1 and P_2 the average, $\bar{P} = \frac{P_1 + P_2}{2}$, has a standard deviation

$$S_{R;k1,k2}(\bar{P}) = \frac{\sqrt{S^2_{R,k1}(P_1) + S^2_{R,k2}(P_2)}}{2} \qquad (5.6)$$

where result P_1 was obtained as the mean of k1 repetitions, and P_2 of k2 repetitions. Since our operating hypothesis is that $P_1 = P_2$, we will use the multilaboratory precision calculated at $\bar{P}$ as the test standard deviation:

$$S_{R;k1,k2}(\bar{P}) = \frac{\sqrt{S^2_{R;k1}(\bar{P}) + S^2_{R;k2}(\bar{P})}}{2} = \frac{S_R(\bar{P})}{\sqrt{2}}\sqrt{1 - \left[1 - \frac{1}{2k1} - \frac{1}{2k2}\right]\frac{S^2_r(\bar{P})}{S^2_R(\bar{P})}}$$

$$\leq 0.7071 \; S_R(\bar{P}) \qquad (5.7)$$

The distribution of the mean of two independent measurements therefore has a reduced standard deviation, and, because it is therefore narrower, a greater proportion of the distribution for a within specification bitumen lies inside the acceptance/rejection limits, i.e. the chance of rejecting a within-specification bitumen is reduced if the mean of two independent test results is used. A mean of three independent penetration tests reduces the chances further. See Table 5 for details with a 180/200 bitumen.

True Penetration	No. of Independent Results	Probability Mean Penetration >210	Probability Mean Penetration <170
180	1	0.02%	12.0%
	2	Negligible	4.8%
	3	Negligible	2.1%
190	1	1.3%	1.3%
	2	0.08%	0.08%
	3	Negligible	Negligible
200	1	14.6%	0.08%
	2	6.8%	Negligible
	3	3.4%	Negligible

Table 5: Probability of Mean Penetration Results Outside Acceptance Limits for a 180/200 Bitumen. Effect of Multiple Penetration Testing (single results from each laboratory (i.e. k1 = k2 = k3 = 1).

5.3 Repeated Within-Laboratory Testing

This practice can only tighten acceptance requirements by a small amount. If a test procedure is repeated k times, the multilaboratory precision of the test, $S_{R,k}$ becomes

$$S_{R;k}(P) = \sqrt{S_R^2(P) - S_r^2(P)\left(1 - \frac{1}{k}\right)} \quad (5.8)$$

S_R (P) and S_r (P) are as defined in Table 2. Calculating a sample of values of $S_{R;k}$ for k=2 and 3 we obtain the results shown in Table 4.

Penetration	S_R	$S_{R;2}$	$S_{R;2}/S_R$	$S_{R;3}$	$S_{R;3}/S_R$
45	2.50	2.435	0.974	2.413	0.965
65	2.75	2.667	0.970	2.638	0.959
90	3.95	3.769	0.954	3.707	0.939
140	6.50	6.093	0.937	5.952	0.916
190	9.00	8.364	0.929	8.141	0.905

Table 6: Multilaboratory precisions for multiple within-laboratory testing

The proportional changes in $S_{R;k}$ as k goes from 1 to 3 are small (this is a consequence of S_r being significantly smaller than S_R), and the effect on acceptance/rejection limits is also small. Contrast this for a number of single results from independent laboratories, where the standard deviations of the mean are $0.7071S_R$ and $0.5774\ S_R$ respectively.

For this reason, testing to check a supplied penetration result should always comprise single results from independent laboratories (repeat results contributing to the supplier's reported penetration value).

5.4 Detection of Outlying Results

5.4.1 Two Independent Penetration Results

For two penetrations P_1 and P_2, the difference (P_1 - P_2) has a standard deviation of $S_{R;k1,k2}(\overline{P})$, with an expected value of zero. Following International Standard EN ISO 4259:2006 we require for acceptance that the absolute value of (P_1-P_2) lies within the range for 95% probability of acceptance that; i.e.

$$|P_1 - P_2| \le 1.96\ S_{R;k1,k2}(\overline{P}) \quad (5.9)/(4.5)$$

If this relationship is not obeyed the difference of the penetration values is large, and at least one of the values is likely to be in error. It is, however, impossible to tell which of the two results is the more accurate unless a third, independent, test is carried out.

5.4.2 Three Independent Penetration Tests

We take $\left| P_a - \frac{P_b + P_c}{2} \right|$ as the maximum value of $\left| P_1 - \frac{P_2 + P_3}{2} \right|$, $\left| P_2 - \frac{P_3 + P_1}{2} \right|$ and $\left| P_3 - \frac{P_1 + P_2}{2} \right|$.

The standard deviation of $\left(P_a - \frac{P_b + P_c}{2} \right)$ is

$$S_{R;ka;kb,kc}\left(P_a - \frac{P_b + P_c}{2} \right) = \sqrt{S^2_{R;ka}(P_a) + \frac{S^2_{R;kb}(P_b) + S^2_{R;kc}(P_c)}{4}} \quad (5.10)$$

We have evaluated this quantity by substituting the average penetration $\bar{P}$ into equation (5.10), on the grounds that the acting hypothesis is that the true value of penetration is $\bar{P}$. Operating at the 95% acceptance level, the test for an outlier becomes

$$\left| P_a - \frac{P_b + P_c}{2} \right| > 1.96\, S_{R;ka;kb,kc}(\bar{P}) \quad (5.11)/(4.8)$$

$$\text{or} \left| P_a - \frac{P_b + P_c}{2} \right| > 2.4005\, S_R(\bar{P}) \quad (5.11a)/(4.8a)$$

in the event that k1 = k2 = k3 = 1.

6.0 Registered Testing Laboratories in New Zealand

Use the following web address to search for laboratories with IANZ (International Accreditation New Zealand) registration for the penetration test, ASTM D5. Search by keyword for D5:06 or D5-06

http://cabis.ianz.govt.nz/ianzwebportal/

7.0 References

American Society for Testing and Materials specification ASTM D5-06e1 (2006) Standard test method for penetration of bitumous materials

International Standard EN ISO 4259:2006 (2006) Petroleum projects – Determination and application of precision data in relation to methods of test

NZ Transport Agency Specification NZTA M1: 2010 Specification for Reading Bitumens