DETERMINATION OF UNCERTAINTY OF MEASUREMENT FOR PENETRATION TESTING

J O'CONNELL, **G A J MTURI and J.MAINA**

CSIR Built Environment, Transport Infrastructure Engineering, Pretoria 0001, South Africa
Tel: +27 12 841 2295; E-mail: joconnell@csir.co.za

ABSTRACT

Any measurement has an error associated with it. The parameter that quantifies the boundaries of the error of that measurement is known as the uncertainty of measurement. Without determining the uncertainty of measurement, any measurement lacks merit.

An assessment of uncertainty of measurement is carried out for the standard test method for penetration of bituminous materials as prescribed by ASTM D5. The assessment of uncertainty of measurement is performed using the principles stated in the ISO Guide to the Expression of Uncertainty in Measurement (ISO GUM).

A model was formulated for the purposes of determining the uncertainty of measurement, incorporating the equipment (based in the advanced testing laboratories at the CSIR), environment and test procedure. Sources of measurement variation were identified and isolated within the model. Furthermore, these sources were classified as either random or systematic, as well as whether they were subject to type A or type B analyses.

Experimental processes quantified the various sources of uncertainty and an overall uncertainty of measurement was calculated for the test method.

This paper illustrates how ISO GUM may be applied to determine the accuracy of the penetration test method. The results can be used to refine the penetration test procedure and tighten up on equipment specifications in order to reduce the level of test uncertainty. In addition, this paper may serve as an example of how to extend ISO GUM to other laboratory test methods.

1 INTRODUCTION

Any measurement has an error associated with it. The parameter that quantifies the boundaries of the error of that measurement is known as the uncertainty of measurement. Without determining the uncertainty of measurement, any measurement lacks worth.

An assessment of uncertainty of measurement is carried out for the standard test method ASTM[1] D5 (2006) "Test Method for Penetration of Bituminous Materials" as supplied by ASTM International and as amended by SANS[2] 307 (2005)

The amendment according to SANS 307 (2005), Paragraph 6.2 requires that the penetration needle be pre-treated with oleic acid. This is in reference to ASTM D5 (1986), Note 4, where it is stated that international experience has shown that the use of 1% Oleic

Proceedings of the 30th Southern African Transport Conference (SATC 2011) 11 - 14 July 2011
Proceedings ISBN Number: 978-1-920017-51-4 Pretoria, South Africa
Produced by: Document Transformation Technologies cc Conference organised by: Conference Planners

acid in toluene improves the repeatability of results obtained on bitumen samples derived from some crude oils.

The assessment of uncertainty is limited to the CSIR Built Environment pavement materials laboratory for intra-laboratory single-operator determinations.

The assessment of uncertainty of measurement is performed using the principles stated in the ISO Guide to the Expression of Uncertainty in Measurement (ISO GUM). The steps can be summarized as follows (Drongo's guide, 2006; Cook, 2002):

- Develop a model of the measurement process, including all steps pertaining to sample procurement (sampling) and preparation (heating and agitation)
- Determine all the uncertainty components based on the model
- Calculate the sensitivity coefficients
- Calculate the component uncertainties.
- Calculate the associated degrees of freedom if required.
- Convert all uncertainties into uncertainties of the same unit as the measurand.
- Combine all the uncertainties.

Sources of uncertainty can be classified as either random or systematic:

- Random Errors (wikipedia, physics.umd.edu): Random errors are errors in measurement that lead to measurable values being inconsistent when repeated measures of a constant attribute or quantity are taken. Random error is always present in a measurement and is caused by unknown and unpredictable changes in the measuring instrument and/or in the environmental conditions (eg electronic noise, ambient temperature, etc). Random errors often have a Gaussian normal distribution promoting the use of statistical methods to analyze the data.

- Systematic Errors (Wikipedia): Systematic errors are predictable, and typically constant or proportional to the true value. If the cause of the systematic error can be identified, then it can usually be eliminated or a correction factor can be applied. Systematic errors always affect the results of an experiment in a predictable direction. Examples include imperfect calibration of measurement instruments, imperfect methods of observation or interference of the environment with the measurement process.

Another form of error affecting experimental results is known as blunders. Blunders cannot be easily quantified and are not addressed in this paper. Examples of blunders include:

- Improper deviation from the standard test method
- Incorrect sampling / sample preparation procedures
- Computational errors

2 THE MODEL

The development of a model of the measuring process is a crucial first step in order to accurately identify all the sources of uncertainty. The accuracy of the combined uncertainty derived at the end of the process is dependent on the realism attained in the initial model.

The penetration test, ASTM D5 (2006), is used to determine the consistency of bituminous (asphaltic) materials expressed as the distance in tenths of a millimetre (dmm) that a standard needle vertically penetrates a sample of the material under known conditions of loading, time, and temperature. The most common conditions are 100 g penetrating for 5 seconds at a temperature of 25°C. (Higher values of penetration indicate a softer consistency of the bituminous material.)

The model is summarized in Figure 1.

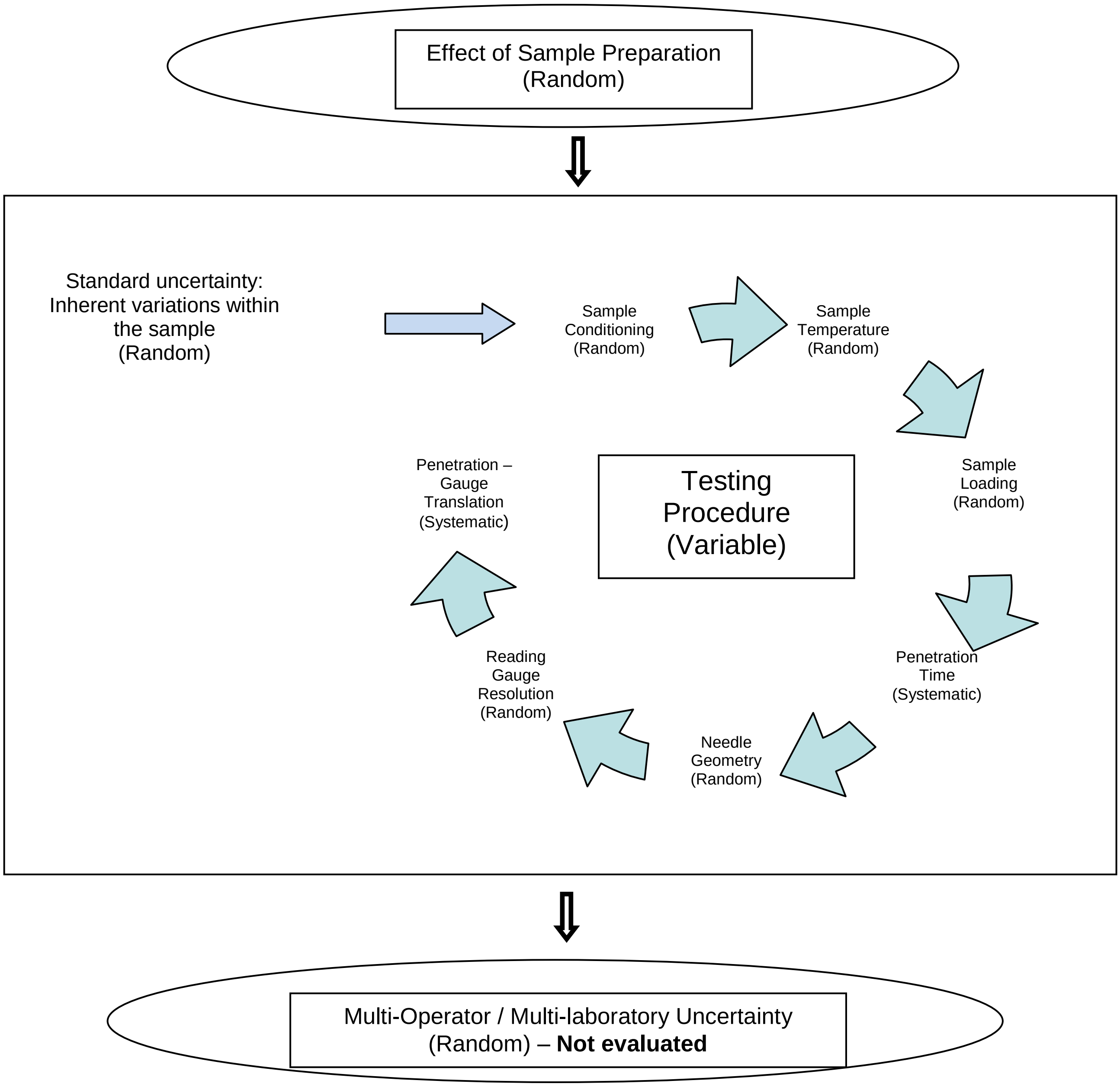

Figure 1: Model for the presentation of penetration of bituminous materials

3 QUANTIFICATION OF UNCERTAINTY COMPONENTS

The evaluation of uncertainty components can be classified as either Type A or Type B (Cook, 2002). Type A evaluation uses statistical calculations for the determination of uncertainties and is most often associated with random errors that can be presented by a normal or Gaussian distribution. Type B evaluation uses means other than standard statistical methods to determine an uncertainty component. Examples include using data provided in a calibration certificate or reference books or an estimate by an experienced expert in the field based on sound principles (stated in the evaluation).

3.1 The uncertainty component due to sample preparation (u_{prep}): Type A

ASTM D5 (2006) states that sample preparation entails heating the sample until it becomes sufficiently fluid to pour. The temperature of the sample may not be raised to more than 90˚C above the expected softening point of the sample. The time required to fluidize a sample would be dependent on the sample size, the oven temperature and the binder stiffness. As a rule of thumb, the procedure followed for a 1l sample container at the CSIR is as follows (Protocol):

- The container is placed in an oven maintained at a temperature of approximately 160˚C and the sample is stirred periodically.
- After one hour, when the sample is sufficiently fluid for thorough mixing, the sample container is removed from the oven and an appropriate volume of sample is taken for testing.
- Upon being taken out of the oven, the approximate sample temperature is ≤110ºC (maximum 60˚C above the expected softening point)

ASTM D5 (2006) does not place a limit on the time for which the sample is heated or specify a maximum oven temperature. Due to possible ageing of the binder during this heating time, a variation in heating time may result in a variation of the penetration measured for the sample. In order to determine the effect of heating time, identical binder samples were tested; one after 45 minutes of heating and another after 90 minutes of heating. Results are presented Table 1.

Table 1: Variation in Penetration Measurements with Preparation Time

Heating Condition	Reading 1 (dmm)	Reading 2 (dmm)	Reading 3 (dmm)	$\bar{x}$	σ
45 minutes	50	50	50	50	0
90 minutes	50	50	50	50	0

The results indicate that the variation arising from 45 minutes of additional heating is negligible under the standard method, ie (u_{prep}) = 0

3.2 Standard uncertainty (u_s): Type A

The standard uncertainty represents the inherent variability within the sample itself; and can be calculated from the Experimental Standard Deviation of the Mean (ESDM), where

$$\mathrm{ESDM} = \frac{\sigma}{\sqrt{n}} \qquad (1)$$

σ is standard deviation, and
n is number of readings

Using the results for the 3 hour conditioning time from Table 2, we have

$$\text{ESDM} = \frac{0.43}{\sqrt{12}}$$

Or $u_s = 0.12$ dmm

3.3 The uncertainty component due to sample conditioning (u_{con}): Type A

ASTM D5 (2006) requires sample conditioning at 25˚C prior to testing. The conditioning period is 1 to 1.5 hours in air and 1 to 1.5 hours in water, allowing for a full one hour's difference between the minimum and maximum allowable times. Bituminous binder is known to re-align its molecular structure after cooling to room temperature, thereby influencing the consistency of the binder. This influence was investigated using the minimum and maximum allowable conditioning periods for identical samples of binder.

Table 2: Variation in Penetration Measurements with Conditioning Time

	Reading 1 (dmm)	Reading 2 (dmm)	Reading 3 (dmm)	Average per specimen
Sample Conditioning 3 hrs	68	67	68	68
	68	68	68	68
	68	68	68	68
	69	68	68	68
			$\bar{x}$ = 3hr	68
			σ = 3hr	0.43
Sample Conditioning 2 hrs	70	70	69	70
	68	69	69	69
	70	70	70	70
	69	69	70	69
			$\bar{x}$ = 2hr	69
			σ = 2hr	0.67
Total Sample Average and Standard deviation			$\bar{x}$	69
			σ	0.90

From equation 2 the component uncertainty (u_i) can be determined.

$$u_i = \frac{\sigma}{\sqrt{n}} \qquad (2)$$

$$u_{con} = \frac{0.90}{\sqrt{24}}$$

or $u_{con} = 0.18$ dmm

3.4 The uncertainty component due to sample temperature variation (u_T): Type B

This Type B evaluation calls for an informed judgement regarding the variation within the sample temperature, considering that extensive records of such temperature variation are not available. A limited measurement of sample temperature variations, using a calibrated thermometer with known uncertainty, indicates that the maximum temperature variation is ± 0.2 ˚C. In order to convert this uncertainty into one that has the same unit as penetration, we need to calculate the sensitivity coefficient. This was done by measuring the average penetration for identical samples over a temperature range (23 – 27˚C). The results are presented in Figure 2.

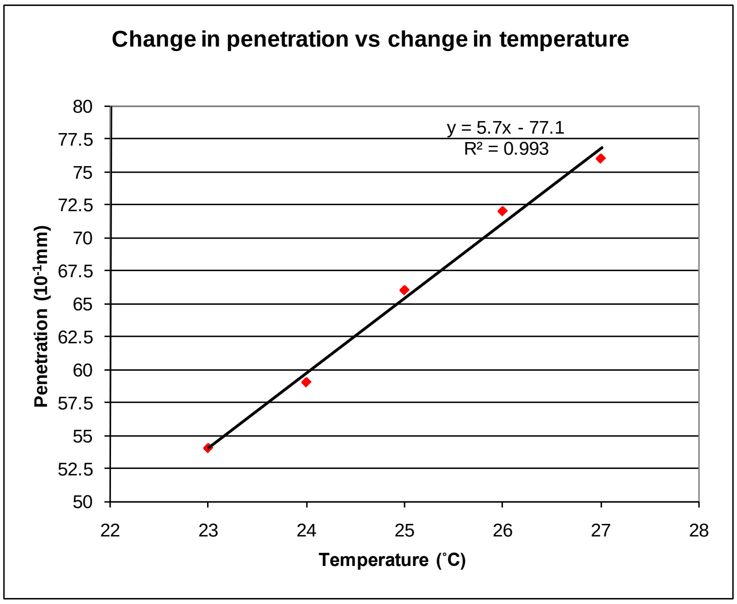

Figure 2: Effect of temperature on penetration measurements

The gradient of the trend line represents the temperature coefficient to relate penetration measurements with temperature. Using the temperature coefficient we then calculate the error arising from the temperature as follows:

5.7 dmm / ºC X ± 0.2ºC = ± 1.14 dmm

Assuming a rectangular distribution, then the semi-range, *a,* is 1.12 dmm. According to the law of uncertainties for rectangular distributions,

$$u_T = \frac{a}{\sqrt{3}} \qquad (3)$$

$$u_T = \frac{1.14}{\sqrt{3}}$$

or $u_T =$ 0.66 dmm

3.5 The uncertainty component due to load variation (u_{load}): Type B

ASTM D5 (2006) allows for a needle load variation of 100 ± 0.10 g. In order to convert this uncertainty into one that has the same unit as penetration, we need to calculate the sensitivity coefficient. This was done by measuring the average penetration for identical samples over a load range of approximate one gram. The results are presented in Figure 3.

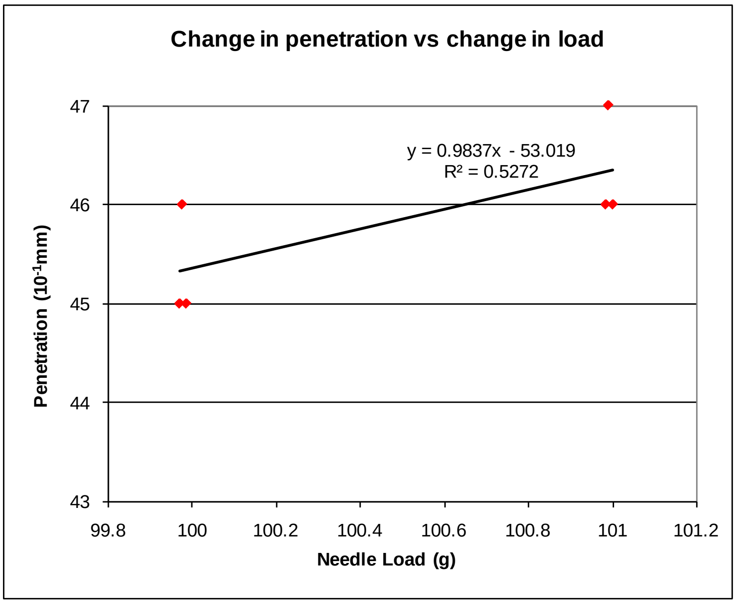

Figure 3: Effect of needle load on penetration measurements

The gradient of the trend line represents the load coefficient to relate penetration measurement with load. Since the maximum permissible load variation is 0.10 g, the load coefficient allows us to calculate the error as follows:

0.98 dmm /g X ± 0.10 g = ± 0.10 dmm

Assuming a rectangular distribution, then the semi-range *a* is 0.10 dmm. Therefore, according to the law of uncertainties (eqn 3) for rectangular distributions,

$$u_{\text{load}} = \frac{0.10}{\sqrt{3}}$$

or $u_{\text{load}} =$ 0.06 dmm

3.6 The uncertainty component due to loading time (u_t): Type B

The loaded needle is applied to the sample for a period of 5.0 s. ASTM D5 (2006) requires timing accuracy of 0.1 s. The automated timer is verified using a calibrated stopwatch and the test method limit appears to be complied with. As the automated timer cannot be adjusted, a different approach is required to estimate a timing coefficient to correlate time, s, with penetration, dmm.

Assuming a proportional relationship between time and penetration over this short time period, 0.1 s would represent 0.1/5.0 (or 2 %) of 65 for a typical 60/70 penetration-grade bitumen. Assuming a value of a = 1.30, then:

$$u_t = \frac{1.30}{\sqrt{3}}$$

or $u_t =$ 0.75 dmm

3.7 The uncertainty component due to needle geometry (u_{ng}): Type B

Variations in penetration due to variations in needle geometry are monitored by verification of all needles against a standard calibrated needle. Verification records indicate that the maximum difference obtained between the standard needle and the average from a set of 3 working needles is 0.67 dmm. Assuming the value of a = 0.67, then:

$$u_{ng} = \frac{0.67}{\sqrt{3}}$$

or $u_{ng} =$ 0.39 dmm

3.8 The uncertainty component due to resolution of the reading gauge (u_{res}): Type B

Due to error of parallax it is assumed that the reading could be in error by approximately half a unit, which is ± 0.5 dmm. Assuming the value of a = 0.50, then:

$$u_{res} = \frac{0.50}{\sqrt{3}}$$

or $u_{res} =$ 0.29 dmm

3.9 The uncertainty component due to resolution of the reading gauge (u_{meas}): Type B

Another possible source of systematic error could be during the translation process whereby the penetration depth is converted to a gauge reading. This translation is verified by measuring the depth of 50.00 dmm depth within a stainless steel block. The block has been calibrated and the calibration certificate states an uncertainty of ± 0.01 dmm. This is small enough to be ignored for the purposes of our determination. The measurements on the block indicate that no correction is necessary for the dial at a reading of 50.00 dmm.

4 Combined Uncertainty (u_c)

The combined uncertainty can be determined using Equation 4. Logically, the standard uncertainty value can be ignored as it is already quantified when determining the uncertainty component for conditioning time.

$$u_c = \sqrt{\sum_{i=1}^{N} u_i^2} \qquad (4)$$

$$u_c = \sqrt{u_{prep}^2 + u_s^2 + u_{con}^2 + u_T^2 + u_{load}^2 + u_t^2 + u_{ng}^2 + u_{res}^2}$$

$$u_c = \sqrt{0 + 0 + 0.18^2 + 0.66^2 + 0.06^2 + 0.75^2 + 0.39^2 + 0.29^2}$$

u_c = 1.13 dmm

The expanded uncertainty is then (using a coverage factor of 2) equal to U = 2.24 dmm. Since the penetration is reported in whole units, this uncertainty will be rounded to the closest whole unit.

The uncertainty of the penetration is ± 2 dmm with a confidence level of approximately 95%. This would be applicable to a 40/50 or 60/70 penetration-grade bitumen

5 CONCLUSIONS AND RECOMMENDATIONS

This paper illustrates how ISO GUM may be applied to determine the accuracy and uncertainty of the penetration test method. The model and uncertainty determination have provided a means with which to analyse the method in detail. This enables the researcher to propose modifications to the test method to improve uncertainty level for the method.

For example, paragraph 7.3 of the test method requires conditioning periods of between 1 and 1½ hours in air and between 1 and 1½ hours in water. This permissible variation is shown to affect the repeatability of the results. At the CSIR Built Environment road materials testing laboratories an amendment to the test method has been implemented, whereby the sample undergoes temperature conditioning for exactly 1½ hours in air and 1½ hours in water. This amendment results in a change in the combined uncertainty from u_c = 1.13 dmm to u_c = 1.11 dmm. The difference is not big enough to have an effect on the expanded uncertainty, but it is a move in the right direction.

A larger difference will be evident if the timer can be calibrated/verified as having an uncertainty of ± 0.03 s. The effect would be to reduce the combined uncertainty from u_c = 1.11 dmm to u_c = 0.85 dmm.

This paper may serve as an example of how to extend ISO GUM to other laboratory test methods.

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