CALIBRATION OF NUCLEAR DENSITY METERS: STANDARD DENSITY BLOCKS

1 SCOPE

This method describes the procedure for the calibration of Nuclear Density Meters using Standard Density Calibration Blocks.

The calibration is applicable for the normal operational range of the meter (nominally 1.4 t/m^3 to 2.65 t/m^3). The calibration can cover an extended density range to approximately 3.05 t/m^3, when needed for higher density granular materials. The equations produced for density are an expression of the relationship between the count ratio and the wet density.

2 SAFETY

This method does not attempt to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate occupational health and safety practices that meet statutory regulations.

3 REFERENCED DOCUMENTS

The Australian Radiation Protection and Nuclear Safety Agency - Code of Practice and Safety Guide – Portable Density/Moisture Gauges Containing Radioactive Sources. This document is available at the ARPANSA – Code of Practice website (www.**arpansa**.gov.au/Publications/**code**s/rps2.cfm).

Nuclear Density Meter Manufacturers Handbook

Calibration Method WA 2040.1 "Standard Density Blocks for Nuclear Moisture/Density Meter Calibration".

AS 1289.5.8.4 Nuclear Surface Moisture-Density Gauges – Calibration using Standard Blocks

ISO/IEC Guide 98 Uncertainty of measurement - Guide to the expression of uncertainty in measurement

4 APPARATUS

(a) **Nuclear Density Meter** and its **nylon reference block** for carrying out standard counts and stability and drift tests.

(b) **Normal operational range** - A minimum of five Standard Density Calibration Blocks calibrated in accordance with Method WA 2040.1, evenly spaced to provide a range of densities from approximately 1.40 t/m^3 to approximately 2.65 t/m^3.

(c) **Extended density range** - A minimum of four Standard Density Calibration Blocks calibrated in accordance with Method WA 2040.1, evenly spaced to provide a range of densities from approximately 2.65 t/m^3 to approximately 3.05 t/m^3.

The highest standard density calibration block used for the normal operational range calibration is also used for the extended density range calibration. This calibration block, common to the normal operational range and the extended density range, shall be within 0.05 t/m^3 of 2.65 t/m^3.

NOTE: The intent is to obtain a range of natural stone blocks that are as evenly spaced in densities as practicable.

NOTE: The extended density range calibration is optional and only necessary when calibrations are required to cover higher density granular materials. materials.

(d) Bannister brush or similar

5 PREPARATION

Clean the cavity of the Nuclear Density Meter and charge and/or change the batteries before commencing calibration.

Perform the manufacturer's operational checks not more than two days prior to the Nuclear Density Meter density calibration.

NOTE: If the manufacturer's function test includes a drift test this may be carried out during the calibration, after the appropriate time lapse, as stated in the manufacturer's handbook.

Clean the base of the Nuclear Density Meter and the surface of the reference block with a banister brush.

6 PROCEDURE

6.1 Density Calibration

(a) Turn the Nuclear Density Meter on not less than thirty minutes prior to initiating the first standard count.

NOTE: Some types of Nuclear Density Meter turn themselves off prior to thirty minutes. If this is the case, keep the meter activated by taking a few counts.

(b) Place the Nuclear Density Meter onto the nylon reference block in accordance with the manufacturer's requirements. When on the reference block ensure the meter is located at least 2 m from any vertical projection more than 50 mm above the base of the meter and at least 5 m from any surface water, or material having a hydrogen content similar to water, and at least 0.5 m above the ground water level. Maintain the same location for all standard counts undertaken as part of the calibration.

NOTE: Unless adequately shielded, ensure that any gamma radiation source not exceeding 370 MBq or neutron emitting source not exceeding 1.85 GBq is at a distance of not less than 16 m from the Nuclear Density Meter. For the definition of adequate shielding and/or for appropriate clearance distances for sources of greater activity, singly or combined, advice shall be obtained from the Radiation Safety Officer.

(c) Apply downward force to the handle of the source rod to ensure that the latch is firmly against the safe position location stop with the source in the shielded position.

(d) Initiate the four-minute count mode of the Nuclear Density Meter and at the completion of density standard count record the value. Repeat for a second four-minute count. Designate the two four-minute counts as $d_{1.1}$ and $d_{1.2}$.

(e) Calculate:

$$C_{1.1} = |d_{1.1} - d_{1.2}|, \qquad E_{1.1} = \left(\frac{d_{1.1} + d_{1.2}}{2}\right)$$

If $C_{1.1}$ is less than 0.008 $E_{1.1}$ then $d_{1.1}$ and $d_{1.2}$ can then be accepted as the first pair of valid density standard counts.

If $C_{1.1}$ exceeds the stated limit a third four-minute density count shall be taken. Designate this count as $d_{1.3}$.

(f) Calculate:

$$C_{1.2} = |d_{1.1} - d_{1.3}|, \qquad E_{1.2} = \left(\frac{d_{1.1} + d_{1.3}}{2}\right)$$

(g) Calculate:

$$C_{1.3} = |d_{1.2} - d_{1.3}|, \qquad E_{1.3} = \left(\frac{d_{1.2} + d_{1.3}}{2}\right)$$

If $C_{1.2}$ is less than 0.008 ($E_{1.2}$) and $C_{1.3}$ is less than 0.008 ($E_{1.3}$) then select the pair $d_{1.1}/d_{1.3}$ or $d_{1.2}/d_{1.3}$ with the smallest difference as the first pair of valid density standard counts.

If only one pair satisfies the stated limit accept this pair. If neither of the two pairs satisfies the stated limit the calibration shall be abandoned, and the Nuclear Density Meter checked for defects prior to re- commencing the calibration procedure.

Designate

$$E_{1.0} = \left(\frac{d_{1.1} + d_{1.2}}{2}\right) \; or \; \left(\frac{d_{1.1} + d_{1.3}}{2}\right) \; or \; \left(\frac{d_{1.2} + d_{1.3}}{2}\right)$$

(h) Place the Nuclear Density Meter on one of the standard density calibration blocks selected for calibration. The Nuclear Density Meter source rod shall be over the source access hole 'A' and the long axis of the Nuclear Density Meter shall be over the line connecting the two access holes.

NOTE: The correct location of the Nuclear Density Meter will be facilitated if the outline of its base at this location is marked on the upper surface of the standard density calibration block.

(i) Depress the Nuclear Density Meter source rod handle to the selected direct transmission depth.

If there is any resistance to penetration the Nuclear Density Meter source rod shall be returned to its shielded position. The Nuclear Density Meter shall be repositioned so that source rod handle can be depressed without any resistance. Ensure that the Nuclear Density Meter handle latch is correctly engaged against the location stop.

(j) Move the Nuclear Density Meter carefully towards the unoccupied source access hole until the source rod is against the side of the occupied source access hole so that there is minimal air gap between the source rod and the detector tubes.

(k) Initiate the four-minute count mode of the Nuclear Density Meter and at the completion of the count record the value.

NOTE: When the calibration is required in more than one direct transmission depth it is convenient to repeat Procedure 6.1(i) and 6.1(j) for those depths while the source rod is in the source access hole 'A'.

(l) Move the Nuclear Density Meter carefully to locate the source rod centrally in the source access hole.

(m) Return the source rod to its shielded position.

(n) Position the Nuclear Density Meter over the source access hole 'B' and repeat Procedure 6.1(h) to 6.1(k).

(o) Perform four-minute counts on each of the blocks, up to approximately one-half of the number of blocks required for the calibration.

(p) Repeat standard counts on the nylon reference block in accordance with Procedure 6.1(b) and 6.1(c).

(q) Initiate and record two four-minute density counts and designate the two four-minute standard counts as $d_{2.1}$ and $d_{2.2}$.

(r) Calculate:

$$C_{2.1} = |d_{2.1} - d_{2.2}|,$$

$$E_{2.1} = \left(\frac{d_{2.1} + d_{2.2}}{2}\right),$$

$$F_{2.1} = |E_{1.0} - E_{2.1}|$$

If $C_{2.1}$ is less than 0.008 ($E_{1.0}$) and $F_{2.1}$ is less than 0.013 ($E_{1.0}$) then $d_{2.1}$ and $d_{2.2}$ can be accepted as the second pair of valid density standard counts.

If $C_{2.1}$ and/or $F_{2.1}$ exceed the stated limits a third four- minute standard density count shall be taken. Designate this count as $d_{2.3}$.

(s) Calculate:

$$C_{2.2} = |d_{2.1} - d_{2.3}|,$$

$$E_{2.2} = \left(\frac{d_{2.1} + d_{2.3}}{2}\right),$$

$$F_{2.2} = |E_{1.0} - E_{2.2}|$$

and

$$C_{2.3} = |d_{2.2} - d_{2.3}|,$$

$$E_{2.3} = \left(\frac{d_{2.2} + d_{2.3}}{2}\right),$$

$$F_{2.3} = |E_{1.0} - E_{2.3}|$$

If $C_{2.2}$ and $C_{2.3}$ are less than 0.008 ($E_{1.0}$) and $F_{2.2}$ and $F_{2.3}$ are less than 0.013 ($E_{1.0}$) then select the pair $d_{2.1}/d_{2.3}$ or $d_{2.2}/d_{2.3}$ with the smallest difference as the second pair of valid standard density counts. If only one pair satisfies the stated limits accept this pair.

If neither of the two pairs satisfies the stated limits, the calibration shall be abandoned and the Nuclear Density Meter checked for defects.

(t) Perform four-minute density counts on the second half of the blocks, as described in Procedure 6.1(n) and 6.1(o), to complete the calibration.

(u) Repeat Procedure 6.1(b) and 6.1(c).

(v) Initiate and record two four-minute standard density counts on the nylon reference block and designate the two four-minute counts as $d_{3.1}$ and $d_{3.2}$.

(w) Calculate:

$$C_{3.1} = |d_{3.1} - d_{3.2}|,$$

$$E_{3.1} = \left(\frac{d_{3.1} + d_{3.2}}{2}\right),$$

$$F_{3.1} = |E_{1.0} - E_{3.1}|$$

If $C_{3.1}$ is less than 0.008 ($E_{1.0}$) and $F_{3.1}$ is less than 0.013 ($E_{1.0}$) then $d_{3.1}$ and $d_{3.2}$ can be accepted as the third pair of valid standard density counts.

If $C_{3.1}$ and/or $F_{3.1}$ exceed the stated limits and the second pair of valid standard density counts was accepted only after carrying out Clause 6.1(r) Para. 2 and 6.1(s) the calibration shall be abandoned.

If $C_{3.1}$ and/or $F_{3.1}$ exceed the stated limits and the second pair of valid standard density counts was accepted without carrying out Clause 6.1(r) Para. 2 and 6.1(s), a third four-minute standard density count shall be taken. Designate this count as $d_{3.3}$.

(x) Calculate:

$$C_{3.2} = |d_{3.1} - d_{3.3}|,$$

$$E_{3.2} = \left(\frac{d_{3.1} + d_{3.3}}{2}\right),$$

$$F_{3.2} = |E_{1.0} - E_{3.2}|$$

and

$$C_{3.3} = |d_{3.2} - d_{3.3}|,$$

$$E_{3.3} = \left(\frac{d_{3.2} + d_{3.3}}{2}\right),$$

$$F_{3.3} = |E_{1.0} - E_{3.3}|$$

If $C_{3.2}$ and $C_{3.3}$ are less than 0.008 ($E_{1.0}$) and $F_{3.2}$ and $F_{3.3}$ are less than 0.013 ($E_{1.0}$) then select the pair $d_{3.1}/d_{3.2}$ or $d_{3.2}/d_{3.3}$ with the smallest difference and satisfying the stated limits as the third pair of valid standard density counts. If only one pair satisfies the stated limits accept this pair. If neither of the two pairs satisfies the stated limits, the calibration shall be abandoned and the Nuclear Density Meter checked for defects.

7 DETERMINATION OF THE DENSITY CALIBRATION EQUATION

(a) Calculate the mean density standard count using the formula:

$$\bar{E} = \frac{E_{1.0} + [E_{2.1} + (E_{3.1}\ or\ E_{3.2}\ or\ E_{3.3})]\ or\ [(E_{2.2}\ or\ E_{2.3}) + E_{3.1}]}{3}$$

Where:

$\bar{E}$ is the mean standard count and $E_{1.0}$ and $E_{2.1}$, $E_{2.2}$, $E_{2.3}$ and $E_{3.1}$, $E_{3.2}$, $E_{3.3}$ were found by selecting pairs of valid density standard counts in accordance with Procedures 6.1(d) to 6.1(g) and 6.1(q) to 6.1(s) and 6.1(v) to 6.1(x).

(b) Calculate mean of the two four-minute density counts for each transmission depth on each standard density calibration block to the nearest 0.5. Calculate the count ratios to the nearest 0.0001 by dividing the mean density count by $\bar{E}$ obtained by Clause 7(a). Designate the count ratios as (CR_{dnk}) where 'd' designates the assigned density of the standard density calibration block, n designates the transmission depth and k is the number of the standard density calibration block.

(c) Produce, in table form, for each standard density calibration block and transmission depth, the block number, its assigned density (t/m³) and designated count ratio (CR_{dnk}).

(d) Calculate the calibration equation each standard density calibration block and transmission depth, expressing the relationship between the natural log (ln) of the count ratio and density (t/m³) at each particular depth.

(e) Calculate the slope and intercept of the equation, which is expressed in the following format and assign the equation a number.

Wet Density t/m³ = m x ln (Count Ratio) + b

Where:

m = slope

ln = natural log

b = intercept

7.1 CALIBRATION TO OBTAIN EXTENDED DENSITY RANGE (optional)

(a) Calibrate in accordance with Clause 6.1 above for the determination of the density calibration for an extended density range.

(b) Determine the extended density range calibration equation in accordance with Clause 7 above.

(c) Calculate the point of intercept of the two lines representing the normal operational range and the extended density calibration equations, in count ratio, to the nearest 0.0001. The point of intercept of the two lines is accepted when the calculated intercept density value is less than or equal to ± 0.050 t/m³ of the assigned density of the block common. This count ratio intercept is then converted to density to the nearest 0.001 t/m³ by substitution into the normal operational range and extended range density equations.

(d) There may be instances when the two equations do not result in an intercept, or when the calculated intercept density value is greater than ± 0.050 t/m³ of the assigned density of the block common to the normal operational range and extended range density range equations. In these instances, the intercept point is defined by the count ratio/density coordinate where the separation of the lines representing the two calibration equations is least, but not exceeding 0.010 t/m³ provided this value is within 0.050 t/m³ of the common block density.

(e) If the criteria described at 7.1 (d) cannot be satisfied then a recalibration of the extended density shall be carried out at this depth.

NOTE: It is permissible to adopt an extended range density equation where the separation of the lines is not the lowest possible value, provided the standard error of estimate ($S_{Y.X}$) is within the limiting value described in 9 (c).

7.2 VALID RANGE OF CALIBRATIONS EQUATIONS

The valid range of a density calibration equation is dependent on the range of calibration block densities used.

The valid range for this calibration equation can only be extended 0.050 t/m³ below the lowest calibration block density or 0.050 t/m³ above highest calibration block density.

7.3 VALIDITY OF CALIBRATIONS

(a) When a previous calibration exists, the current calibration equation/s is/are compared to the previous calibration equation/s using the current calibration count ratio range/s.

(b) When no previous calibration/s exist, dual calibrations shall be undertaken for the purpose of validation.

(c) An equally distributed range of at least ten count ratios, over the calibration range is produced. These ten count ratios are then substituted into both equations producing two series of calculated block densities.

(d) At each count ratio the density produced from the new calibration is subtracted from the density from the previous calibration or the dual calibration.

(e) The acceptance criteria are as follows:

- Acceptable when the calibrations for each depth have drifted in the same direction or show a similar trend with depth. If at any calibration depth this pattern or trend is not present, a repeat calibration shall be undertaken at the non- compliant depths only.
- Acceptable when dual calibrations are carried out and the equations differ from each other by less than or equal to 0.010 t/m³ over the calibration range.

8 STANDARD ERROR OF ESTIMATE ($S_{Y.X}$)

Calculate the standard error of estimate ($S_{Y.X}$) in t/m³, for each transmission depth using the following formulae or the equivalent spread sheet function:

$$S_{Y.X} = \sqrt{\frac{\Sigma[Y - \hat{Y}(X)]^2}{n-2}}$$

Where:

$S_{Y.X}$ = standard error of the estimate

X = count ratio

Y = assigned block density values, t/m³

$\hat{Y}$ = calculated block density values, t/m³

n = number of calibration blocks used

9 UNCERTAINTY OF MEASUREMENT

(a) Calculate the uncertainty of the calculated density in accordance with the ISO/IEC Guide 98 Uncertainty of measurement - Guide to the expression of uncertainty in measurement using the following:

$$\rho_w = m \times ln\left(\frac{DC}{SDC}\right) + b$$

Where:

m, b = calibration constants derived by curve fitting of block densities and density count ratios for the calibrated Nuclear Density Meter.

DC = density count

SDC = mean standard density count derived for the calibration

ρ_w = wet density, in t/m³

(b) The following factors shall be considered when estimating the uncertainty of measurement:

- The individual uncertainty of measurement derived from determination of the standard calibration block density (0.01 t/m³, estimated maximum).
- Variation in counts due to surface irregularities on the calibration block (5 counts, estimated maximum),
- Variation in counts due to changes in orientation of the Nuclear Density Meter on the calibration block (5 counts, estimated maximum),
- Variation in the depth of the radioactive source in relation to the Geiger-Mueller Tubes (0.003 x standard count),
- Block count variation due to random background radiation effects (0.02 x standard count, estimated maximum),
- The standard error of estimate ($S_{Y.X}$).
- Variation in standard density counts due to random background radiation effects (0.007 x standard count, estimated maximum),
- Block density variation due to change in temperature of blocks (0.005 t/m³, estimated maximum).

NOTE: The values shown in brackets above are estimated maximum values used by Main Roads Western Australia. These values have been established through testing and experience and may differ for other organisations.

(c) To be valid the Expanded Uncertainty (U_{95}) of the predicted wet density of a Nuclear Density Meter calibrated in accordance with this method shall be less than or equal to 0.050 t/m³ provided the following conditions are satisfied:

- A calibration equation shall be valid only if the standard error of estimate ($S_{Y.X}$), of the produced equation, is less than or equal to 0.035 t/m³.
- Equations that have a standard error of estimate greater than 0.035 t/m³ shall be rejected, the meter checked for faults and if required the calibration can be repeated. If the re-calibration still has a standard error of estimate greater than 0.035 t/m³, calibration equation shall not be reported.

10 REPORTING

Report the following:

(a) Identification of the Nuclear Density Meter.

(b) Identification of the nylon reference block.

(c) Date of calibration.

(d) Density calibration depth setting.

(e) The identification and assigned density for each standard calibration block.

(f) The operating range of each calibration equation in count ratios, to the nearest 0.0001 and in densities, to the nearest 0.001 t/m^3, for:

- Normal
- Normal/extended density

(g) The identification and assigned density of the block common to the normal operation and extended density.

(h) For each calibration depth the calibration equation identification number to be used. The calibration constants of the equations shall be reported to the nearest 0.000001.

(i) The uncertainty of the predicted density, to the nearest 0.001 t/m^3 with associated coverage factor.

(j) The standard error of estimate ($S_{Y.X}$) for each reported calibration equation, to the nearest 0.001 t/m^3

7 ISSUING AUTHORITY

Document Owner	Delegated Custodian
Manager Materials Engineering	Testing and Calibration Manager

8 REVISION STATUS RECORD

Page No.	Section	Revision Description / Reference
1	1. 1	Scope updated to include parameters for the normal operational range of the meter and extended density range.
1	4	Updated information detailing the density calibration blocks required for the “normal operational range” and the “extended density range”. Deleted *NOTE: Where an extended range of density calibration is required two regression equations (low and high density) maybe produced.* Added *NOTE: The extended density range calibration is optional and only necessary when calibrations are required to cover higher density pavement materials.*
4 - 5	7	Deleted text *“two methods are acceptable for the calculation of the regression equation”.* Deleted Method 2. Include Clause (f) “Calculate the Pearson product moment correlation coefficient” in Appendix A (Informative). Insert calibration to obtain extended density range (optional). Incorporate text for dual range calibrations under “calibration to obtain extended density range”.
All	8-10	Reorder and renumber Headings.

APPENDIX A

CALCULATION OF THE PEARSON PRODUCT MOMENT CORRELATION COEFFICIENT (R).

(Informative)

Calculate the Pearson product moment correlation coefficient (r) for each transmission depth using the following formulae or the equivalent spread sheet function:

$$r = \frac{n(\sum xy) - (\sum x)(\sum y)}{\sqrt{[n\sum x^2 - (\sum x)^2][n\sum y^2 - (\sum y)^2]}}$$

Where:
r = Pearson product moment correlation coefficient
x = natural logarithm of the density count ratio values
y = calibrated block density values, t/m³

Where:
r = Pearson product moment correlation coefficient
x = natural logarithm of the density count ratio values
y = calibrated block density values, t/m³