# MEASUREMENTS OF ASPHALT LAYER DENSITIES CONCRETE IN PLACE BY NUCLEAR METHODS - INTERLABORATORY COMPARISON

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## Abstract

European Standard EN 12697-7:2014 [1] specifies a method for measuring the bulk density of pavement mixtures using a transmission type gamma radiation test bench. The standard contains approximate description of measurement procedure. Procedure is not described in details and there are several unknowns during measuring procedure if only European Standard EN 12697-7:2014 is used. Surprisingly precision data at the end of this standard is giving unrealistic good precision (Repeatability r = 0.007 Mg/m$^3$, Reproducibility R = 0.020 Mg/m$^3$). According to this data it seems that laboratory determination of bulk density of bituminous specimens according to EN 12697-6:2020 [2] is less precise (Repeatability r ≥ 0.017 Mg/m$^3$, Reproducibility R ≥ 0.022 Mg/m$^3$). From laboratory experience it can be claimed that precision data in European Standard EN 12697-7:2014 is unrealistic and not applicable for measurement of density of asphalt pavements. In American standard ASTM D2950/D2950M-14 [3] procedure of measurement is explained more in details and precision data seems realistic (Repeatability r = 0.070 Mg/m$^3$, Reproducibility R = 0.078 Mg/m$^3$). In order to get better insight in these problem two interlaboratory comparisons were performed in Slovenia. First interlaboratory comparison was performed in 2005, where 6 laboratories equipped with Troxler type nuclear devices were included. Calibration of all 6 devices was performed on the same 10 places with known densities. On test section each laboratory performed 30 measurements. When average of all was compared we obtained good precision data with averages variation from 2,323 Mg/m$^3$ to 2,336 Mg/m$^3$. But we must be aware that during real in place measurements each laboratory would select different places for calibration and also for measurement. That would lead to bigger differences between laboratories. Second interlaboratory comparison was performed in 2021 and results are still evaluated.

*Keywords: asphalt layer density, in place, nuclear devices, electromagnetic devices, interlaboratory comparison*

## 1 Introduction

Nuclear gauges are mostly used for measuring in-place density during the asphalt compaction process [4]. They are occasionally used for determination of asphalt pavement densities as part of quality control [5]. For this purpose in last year's companies mostly use less dangerous non-nuclear density bench [6]. In Slovenia all companies that perform measuring the bulk density of pavement mixtures in the past were using Troxler transmission type gamma radiation test bench. There are two types Troxler devices in Slovenia: older model (3411) with two sources of gamma radiation ($^{137}$Cs and Am-Be) and newer (Thin Lift Density Gauge Model 4640) with one source of gamma radiation ($^{137}$Cs). Important difference between devices is

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also their age. It is known that radioactive isotope of caesium has half-life time 30.17 years ± 0.03 years [7]. This practically means that after 30 years gamma radiation is reduced for 50 %, so also measurement is round two times less precise.

### 1.1 Purpose of interlaboratory comparisons

Interlaboratory comparisons are performed for different purposes, e.g. for:
- determining the performance of individual laboratories for certain tests or measurements and observing their continued performance,
- identifying problems in laboratories and initiating action,
- determining the effectiveness and comparability of new methods of investigation or measurement and the like to observe already introduced methods,
- ensuring additional trust from laboratory customers,
- identification of differences between laboratories,
- determining the executive characteristics of the method,
- determining the values of reference materials and assessing their suitability for use in certain investigation or measurement procedures.

In assessing the technical capacity of laboratories, a satisfactory result in interlaboratory comparison is considered as important evidence of the laboratory's ability to achieve reliable results.

## 2 Details of interlaboratory comparison in 2005

### 2.1 Participants

The interlaboratory comparison was led by ZAG representative with the participation of six asphalt laboratories from Slovenia (ZAG, Igmat, CGP, Primorje, B & A & M, PMA). Hereinafter, laboratories are numbered 1, 2, 3, 4, 5 and 6 (not in the same order as previously listed).
For interlaboratory comparison, the instructions of the International Organization for Standardization ISO / IEC Guide 43-1: 1997 (E).
Statistical analysis of all results transmitted to all participants without the laboratory label was performed by the ZAG laboratory.
For specific field measurements, the working instructions written in ZAG's document P.H.10-001 (Test methods) were followed, which contain an abbreviated description of the ASTM D2950-91 method for measuring the density of asphalt layers by the back-scatter radiation method.

## 2.2 Procedure

On 15th April 2005, measurements between the P 114 and P 131 profiles were carried out on the left carriageway of the AC Korenitka - Pluska. The binder layer AC 22 bin (PmB) was measured. Each of the participants performed the measurements with their own isotope probe.
First, the calibration procedure of all six probes was performed:
1. All participants measured the number of pulses on the standard 3 times in a row (the bar with the radioactive isotope is in the upper position). The average of the three measurements is the value of the standard (Std).
2. All participants measured the asphalt layer at the same ten measuring points (the rod with the radioactive isotope was in the lower position). Two measurements were performed at each site (one minute each time), with the probe rotated 180 degrees in the second measurement. For preliminary calculations, we considered the average of two

measurements in one place ($Imp_i$). If the measurements in one place differed in absolute terms by more than 4 %, additional measurements were performed at this location until the criteria were met. Calibration ratio is the ratio between the value of the standard and the measurement ($Imp_i$/Std).
3. Asphalt core cores were removed at the measurement sites and the density of removed asphalt cores (ri) was measured according to the SIST EN 12697-6 method (procedure B).
4. From the probe measurements and from the determination of the densities of asphalt cores, the trend line for each individual probe was calculated by regression.

The input data for the regression line are { $Imp_1$/Std, $Imp_2$/Std,... $Imp_{10}$/Std} on the x-axis and {$r_1$, $r_2$, ... $r_{10}$} on the y-axis. The calibration result is the slope of the regression line A and the section of the regression line on ordinate B. The equation of the line is as follows:

$$(r_i) = A \cdot (Imp_i/Std) + B \tag{1}$$

Each of the participants then performed a further 30 measurements with an isotope probe in the test field at their discretion. Further measurements of the density of the asphalt layer with outflow probes were performed as described in points 1 and 2. The results of the measurements were given as the density calculated on the basis of eqn (1).

# 3 Results and statistical analysis of all data

## 3.1 Calibration

Concrete results of input data for calibration procedure for one of participating laboratories are presented in Table 1. Additional information needed for calibration is measurement on Standard. For this laboratory Standard was Std=2065.

**Table 1** Example of data for calibration procedure for one laboratory

| Number of core | Core thick | No. of imp. | Calibrati. ratio | Bulk density EN 12697-6:2020 | Calculated bulk density |
|---|---|---|---|---|---|
| i | [mm] | [$Imp_i$]. | [$Imp_i$/Std] | [Mg/m$^3$] | [Mg/m$^3$] |
| 1 | 60.7 | 482.0 | 0.233 | 2.310 | 2.333 |
| 2 | 60.8 | 451.5 | 0.219 | 2.381 | 2.376 |
| 3 | 70.3 | 492.5 | 0.238 | 2.328 | 2.318 |
| 4 | 60.8 | 508.0 | 0.246 | 2.299 | 2.297 |
| 5 | 60.7 | 475.0 | 0.230 | 2.356 | 2.343 |
| 6 | 70.5 | 488.5 | 0.237 | 2.306 | 2.324 |
| 7 | 70.3 | 476.0 | 0.231 | 2.347 | 2.342 |
| 8 | 70.6 | 517.0 | 0.250 | 2.256 | 2.284 |
| 9 | 70.8 | 512.5 | 0.248 | 2.329 | 2.290 |
| 10 | 80.0 | 472.5 | 0.229 | 2.343 | 2.347 |

In Table 1 Calculated bulk density is product of calibration procedure that can be seen in Fig. 1. From Fig. 1 it can be seen that from eqn (1) calculated constant A has value -2,908 and B 3,012. It can be also seen that coefficient of determination ($r^2$) is relatively low ($r^2$= 0,681).

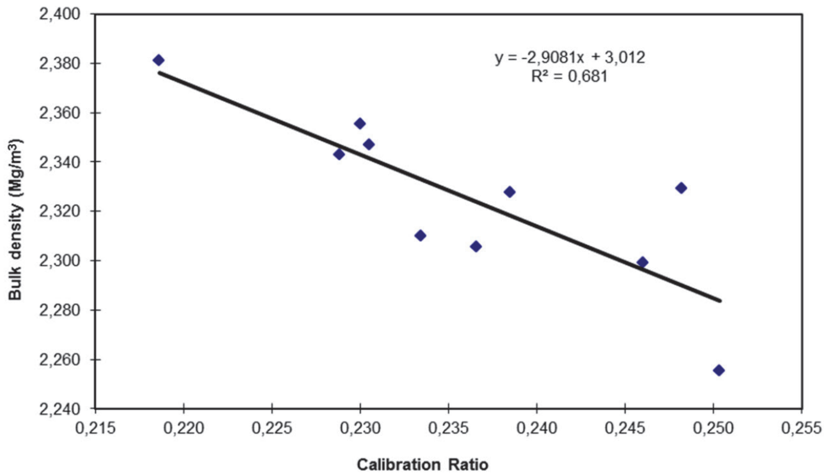


**Figure 1** Calibration of measurement device for one of participating laboratories

## 3.2 Results on test field

Concrete results of density measurements on test field are presented in Table 2. Additional information needed for calculation of Compaction degree and Void content were the same for all laboratories. In this case maximal density of asphalt mixture was 2.545 Mg/m$^3$ and bulk density of laboratory prepared specimen was 2.386 Mg/m$^3$.

**Table 2** Example of data for calibration procedure for one laboratory

| Number of core | No. of imp. | Calibrati. ratio | Calculated bulk density | Compaction degree | Void content |
|---|---|---|---|---|---|
| i | [Imp$_i$]. | [Imp$_i$/Std] | [Mg/m$^3$] | [%] | [v.- %] |
| 1 | 479.5 | 0.232 | 2.337 | 97.9 | 8.2 |
| 2 | 477.0 | 0.231 | 2.340 | 98.1 | 8.0 |
| 3 | 478.0 | 0.231 | 2.339 | 98.0 | 8.1 |
| ... | ... | ... | ... | ... | ... |
| 30 | 481.0 | 0.233 | 2.335 | 97.8 | 8.3 |
| Average | | | 2.336 | 97.9 | 8.2 |
| Standard deviation | | | 0.015 | 0.64 | 0.6 |

## 3.3 Statistical analysis

Statistical analysis of all results was performed as follows:
- The average value of all participating laboratories is selected for the recognized value

The following criterion was chosen to evaluate the results:
- Z-score - comparison of the deviation with the standard deviation, Eq. (2):

$$z = \frac{x - X}{s} \tag{2}$$

s - Standard deviation determined from laboratory results
x - Individual value of the laboratory
X - Arithmetic mean of all laboratories (may also be the value of the reference laboratory)

In Table 3 and Fig. 2 are results of density measurements:
• Statistical analysis of averages of all six laboratories on test field.

**Table 3** Average densities and standard deviations of asphalt layer determined from 30 measurements of all 6 participating laboratories

| **Laboratory number** | **1** | **2** | **3** | **4** | **5** | **6** |
|---|---|---|---|---|---|---|
| Average density of asphalt layer [Mg/m³] | 2.336 | 2.332 | 2.323 | 2.331 | 2.326 | 2.324 |
| Standard deviation [Mg/m³] | 0.015 | 0.018 | 0.018 | 0.021 | 0.018 | 0.030 |

Average results obtained from all six average density calculations:
• Average density of asphalt layer X = 2.329 Mg/m$^3$
• Standard deviation from all six average density calculations s = 0.005 Mg/m$^3$.

In Table 4 are results of Z-score calculations for average densities and average standard deviation of all six laboratories on test fields.

**Table 4** Z-scores of all 6 participating laboratories

| **Laboratory number** | **1** | **2** | **3** | **4** | **5** | **6** |
|---|---|---|---|---|---|---|
| Z-scores for average density of asphalt layer | 1.431 | 0.650 | -1.106 | 0.455 | -0.520 | -0.911 |
| Z-scores for standard deviation of 30 densities | -0.952 | -0.381 | -0.381 | 0.190 | -0.381 | 1.903 |

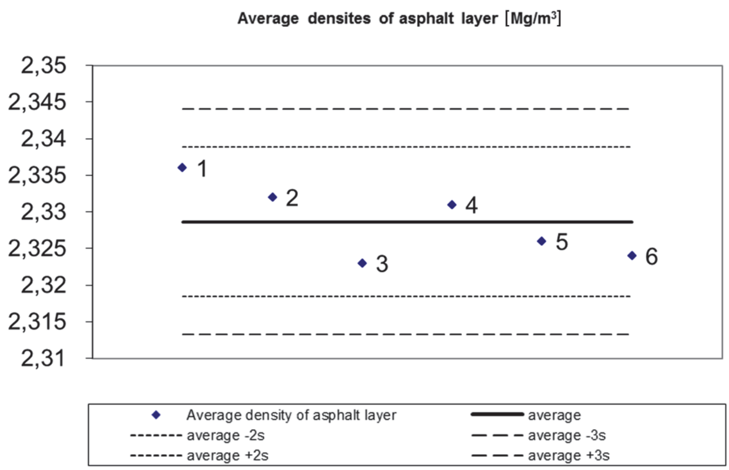


**Figure 2** Average densities and standard deviations of asphalt layer determined from 30 measurements of all 6 participating laboratories

### 3.4 Conclusions from results and statistical analysis of all data

According to the statistical criterion of z-scores with respect to the mean value of all participating laboratories, there is no inadequate result, as they are all in the range of z-scores from -2 to +2. Since the permissible range for interlaboratory comparability of Z-values is from -2 to +2, this is the measured volume mass of the asphalt layer between 2,323 $Mg/m^3$ and 2,336 $Mg/m^3$. With assumption that permitted interlaboratory difference between the two laboratories is the total range: 0.013 $Mg/m^3$ or 0.6 % of the average measured density of the asphalt layer, this result is lower than Reproducibility (R = 0.020 $Mg/m^3$) in European Standard EN 12697-7:2014. Standard deviation is 0.005 $Mg/m^3$.

## 4 Conclusions

In literature it was concluded that for asphalt samples with low to medium voids, the nuclear method and more exact EN 12697-6:2020 method are practically similar [5]. For asphalt samples with high air voids and having a relatively rough surface texture, the EN 12697-6:2020 method measured density values are higher than those determined by nuclear method. In our first interlaboratory comparison performed in 2005 6 laboratories equipped with Troxler type nuclear devices were included. Calibration of all 6 devices was performed on the same 10 places with known densities. On test section each laboratory performed 30 measurements.
First all data i.e. 10 densities, were used for calibration. On the test field measured volume mass of the asphalt layer were between 2.323 $Mg/m^3$ and 2.336 $Mg/m^3$. With assumption that permitted interlaboratory difference between the two laboratories is the total range: 0.013 $Mg/m^3$ or 0.6 % of the average measured density of the asphalt layer, this result is lower than Reproducibility (R = 0.020 $Mg/m^3$) in European Standard EN 12697-7:2014. Standard deviation is 0.005 $Mg/m^3$.
So we found out that results of measurement of asphalt pavement with nuclear method gave good Reproducibility, when all measurement devices were calibrated on the same places. But there is another point that we must be aware. In Table 1 it can be seen that differences between Bulk densities according more exact EN 12697-6:2020 method and nuclear method are huge even at the place of calibration. It can be assumed these differences are even bigger on the test fields. So we can conclude that nuclear methods have good reproducibility (it is precise), but results can be far from real bulk densities of asphalt pavements (it is not accurate).

## References


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