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ESSAIS D'APTITUDE PAR ESSAI DE COMPARAISON

EAPIC CAMPAIGN, SERIAL 14
WATER SENSITIVITY TEST

IDRRIM

INSTITUT DES ROUTES, DES RUES ET DES INFRASTRUCTURES POUR LA MOBILITÉ

EAPIC CAMPAIGN, SERIAL 14
WATER SENSITIVITY TEST

This inter-comparison campaign assessed, under real-life conditions, the performance of 37 laboratories to evaluate the water sensitivity of asphalt mixes according to Method B (Duriez i/C) and/or A (ITSR) of the Standard NF EN 12697-12. It allowed to check consistency in the performance of measurements of maximum and bulk density of asphalt mixes.

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"*Water is the enemy of pavements.*"[1] Its effect on asphalt mixes is characterised by progressive deterioration of the bitumen-aggregate bonding and a decrease in the asphalt mix cohesion, resulting in a decline in performance and reduce the durability of pavements.

To assess the water sensitivity of asphalt mixes, standard NF EN 12697-12[2] proposes 3 methods:

- Method A, the Indirect Tensile Strength Ratio (ITSR), uses the indirect tensile strength of cylindrical specimens.
- Method B, the i/C ratio, uses the simple compressive strength of cylindrical specimens.
- Method C assesses adhesive properties by filtration one hour after mixing.

According to the French methodology of formulation of bituminous mixtures, assessing the water sensitivity using a Gyratory Compactor (GC) represents the first level of mix design. It is therefore an essential test performed by most laboratories in mix design, in particular for CE marking.

In this campaign, the objective of the EAPIC (inter-comparison proficiency testing) working group is threefold:

- To evaluate, by inter-comparison, the ability of laboratories to perform this test;
- To compare the results obtained according to Methods A and B of Standard NF EN 12697-12;
- To evaluate, as necessary, the laboratories consistency of performance measurements of the maximum and bulk densities of asphalt mixes.

This campaign involved 37 laboratories, 22 of which for Method B alone and 15 for both methods A and B.

The objective of this article is to analyse the possible causes of the variability of results both in the preparation of specimens, and in the performance of tests.

COMPOSITION OF THE MIXTURE

The formulation selected for this campaign was asphalt concrete AC 10 Surf 50/70 with an i/C close to the specification limit (70%) defined in the French foreword to NF EN 13108-1[3].

The used aggregate consisted of three granular fractions (0/4, 4/6.3 and 6.3/10) from the Marcigny-sous-Thil quarry (21). The grading curves of each of these fractions and of the recomposed formulation are shown in **Figure 1**.

The water absorption rates, measured according to Standard NF EN 1097-6[4] for each of these fractions, were 0.8%, 0.7% and 0.7%, respectively.

The binder was a 50/70 class bitumen from the Lavéra refinery (13).

The mix formula, shown in Table 1, was selected following a preliminary water sensitivity and GC compaction capacity study. GC compaction showed that the air void content at 60 gyrations was 10.1% (Figure 2). The results of the preliminary water sensitivity study led to an i/C ratio of 72%, which is an appropriate value for a water sensitivity study.

–Table 1–
Composition of the mixture.

Materials	Mass percentage
Sand 0/4	44.8%
Fine aggregate 4/6.3	13.3%
Fine aggregate 6.3/10	37.1%
Bitumen 50/70	4.8%

Several batches of samples were formed, each containing 3 granular fractions (0/4, 4/6.3 and 6.3/10). Ten of these batches were randomly selected for verification of homogeneity before sending the materials to the various laboratories. The homogeneity criterion was checked on the pre-dried density of the aggregates in accordance with Standard NF ISO 13528[5]. In addition, homogeneity was also checked using colorimetric measurements carried out on 10 randomly-selected batches of fraction 6.3/10.

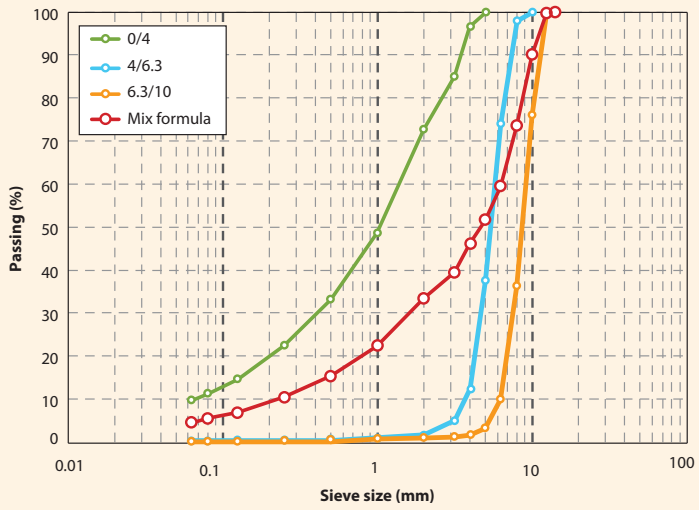
MAXIMUM DENSITY OF THE PREPARED ASPHALT MIX

The maximum densities (ρ_{mv}) of each of the four replicates were determined according to NF EN 12697-5 Method A (in water)[6]. The results obtained from each laboratory for each replicate are shown in Figure 3. Four laboratories were discarded based on the Cochran and Grubbs statistical tests (NF ISO 5725-2[7]).
Precision data (Table 2) show that the repeatability (r) and the reproducibility (R) of the corrected data are lower than those obtained during the EAPIC campaign no. 2-1-0038. This reflects the improved laboratory practice in this test.

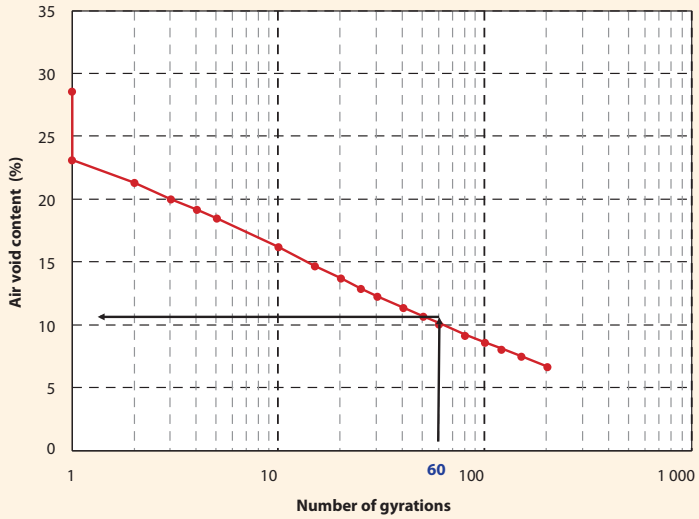
–Table 2–
Data of the reliability of ρ_{mv} of this campaign compared to a campaign from 2006.

	ρ_{mv} (Mg/m³) EAPIC no. 10-1-014 (2015)[9]	ρ_{mv} (Mg/m³) EAPIC no. 2-1-003 (2006)[8]
Number of laboratories	29	26
r	0.016	0.020
R	0.034	0.044

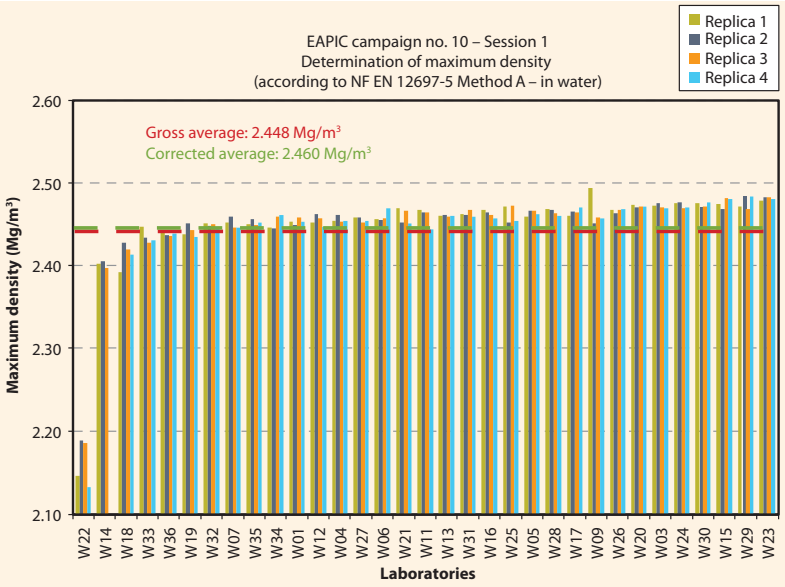
–Figure 1–
Grading curve of the granular skeleton.



–Figure 2–
Air void content assessed with the GC.



–Figure 3–
Maximum density of asphalt mixes.



WATER SENSITIVITY ACCORDING TO METHOD B (DURIEZ TEST)

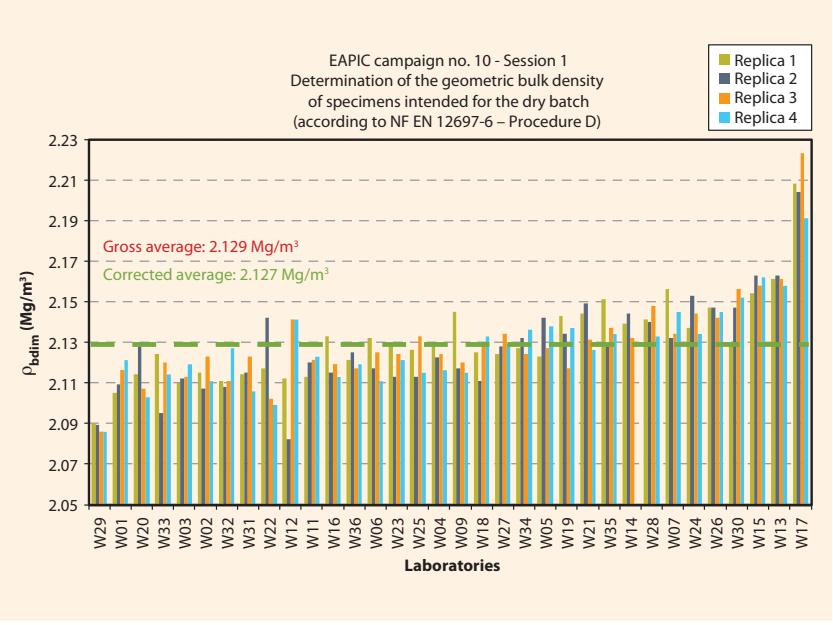
DESCRIPTION OF THE TEST PROTOCOL

Water sensitivity (i/C) of asphalt mixes was determined in accordance with Method B of Standard NF EN 12697-12 and the additional instructions of its national foreward:

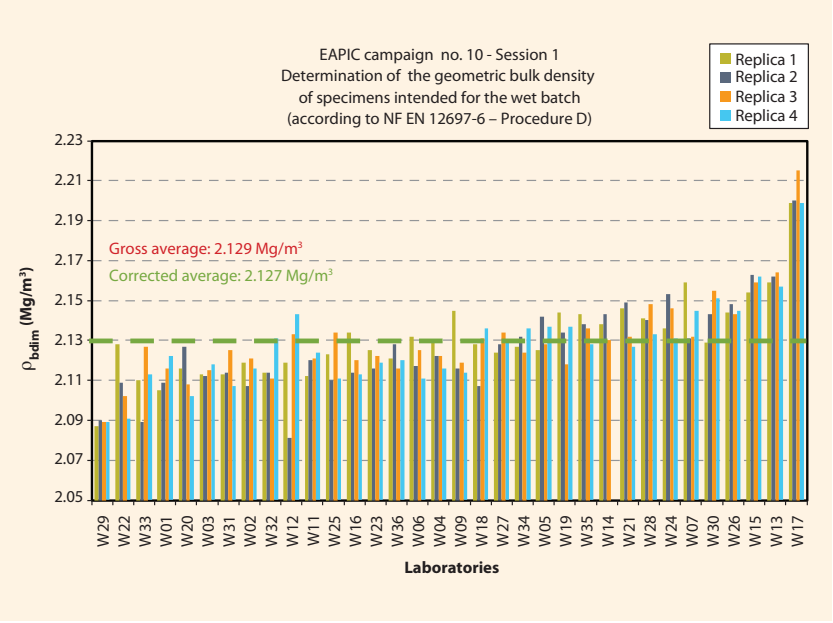
- Diameter: 80 (± 2) mm;
- Number of test specimens per batch: ≥ 5;
- Mass of the specimens: 1,000 (± 2) g;
- Compaction: at 60 kN/300 s by double-effect compression, half an hour to two hours after filling the moulds;
- Determination of the geometric bulk density (ρ_{bdim}) of specimens in accordance with standard NF EN 12697-6 procedure D[10];
- Validation of the complete series of fabricated specimens: ρ_{bdim} constant at ± 1%.

The test specimens were divided into two batches with very close bulk density, one batch being stored in water at 18°C for 7 days (wet batch), and the other stored in air for 7 days at 18°C and 50% relative humidity (RH) (dry batch). The specimens are then subjected to compression test to break at a controlled rate between 45 and 65 mm/min. The i/C ratio expressed as a percentage was then calculated, as the ratio of the average compressive strength of the batch of wet specimens to simple compression to the strength of the batch of dry specimens.

–Figure 4–
ρ_{bdim} values of specimen batches intended for dry storage.



–Figure 5–
ρ_{bdim} values of specimen batches intended for storage in water.



BULK DENSITY OF THE PREPARED SPECIMENS

The results of the average geometric bulk density obtained for each batch are shown in Figures 4 (dry batch) and 5 (wet batch) (ρ_{bdim} values measured according to Standard NF EN 12697-6 method D). Two laboratories were discarded based on the Cochran and Grubbs statistical tests[8].
The corrected precision data r and R are presented in Table 3.

–Table 3–
ρ_{bdim} reliability data of the batches of this campaign.

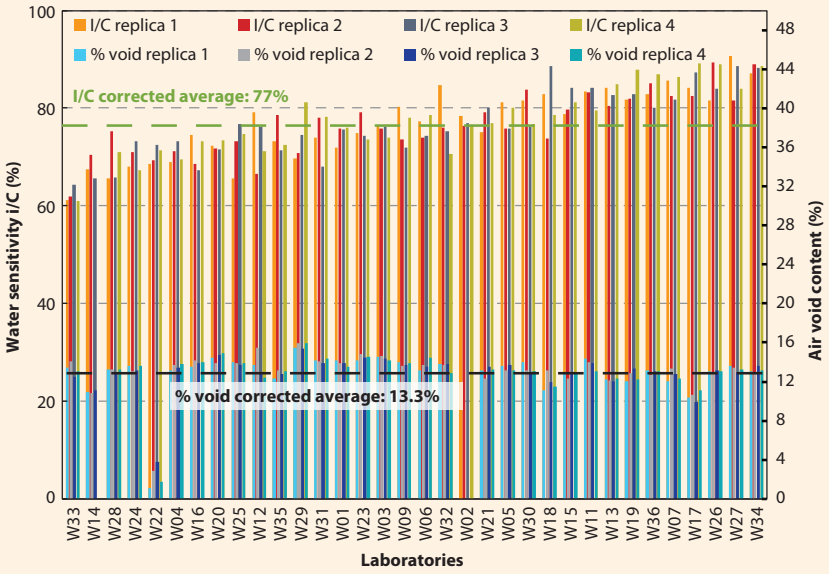
	ρ_{bdim} (Mg/m³) EAPIC no. 10-1-014 (2015)[9]
Number of laboratories	32
r	0.025
R	0.048

They are identical for the dry and wet batches. To our knowledge, there are no comparison data for repeatability and reproducibility of this test.
Based on the maximum (ρ_{mv}) and bulk (ρ_{bdim}) densities of each replicate, we can determine the percentage of voids using the following equation (Equation 1):

$$\% \text{ void} = 100 \times \left(1 - \frac{\rho_{bdim}}{\rho_{mv}}\right)$$

RESULTS AND INTERPRETATION

The results obtained for each replica are presented in Figure 6. The obtained i/C water sensitivity values vary from 62% to 91%, around an average value of 77%. They are highly scattered, but no laboratory was statistically discarted. The obtained precision values were higher than those in Standard NF EN 12697-12 for Method B (Table 4).
The percentages of average voids content calculated using Equation 1 vary from 10% to 16%, around an average value of 13.3% (corrected values after eliminating 4 laboratories based on the results of actual density, which is included in the calculation of the of voids content).



–Figure 6–
i/C water sensitivity and voids content for each replica.

	i/C (%) EAPIC no. 10-1-014 (2015)[9]	i/C (%) NF EN 12697-12[2]
Number of laboratories	34	14
r	9.4	7.8
R	19	13.4

–Table 4–
Precision data of the i/C test of water sensitivity in this campaign versus Standard 12697-12.

Their moderate scatter (standard deviation 1.0%) should be linked to the unique method of preparation of the specimens. However, there is no apparent connection between the percentage of air void content and the i/C test of water sensitivity.

WATER SENSITIVITY ACCORDING TO METHOD A (ITSR)

DESCRIPTION OF THE TEST PROTOCOL

The water sensitivity ITSR of asphalt mixes was determined according to Method A of Standard NF EN 12697-12.

The choice of specimen preparation method was left to the participating laboratories, among the following three methods proposed by the standard:

- Gyratory compaction;
- Roller compaction using a plate compactor followed by coring;
- Impact compaction (Marshall).

During this campaign, 7 laboratories prepared the specimens by GC, 6 by impact compacting and 2 by plate coring.

After demoulding, the geometric bulk density (ρ_{bdim}) of specimens was determined according to standard NF EN 12697-6 procedure D[10].

The prepared specimens were divided into two batches of very close bulk density. The test specimens intended for the wet batch were stored in water at 40°C for 72 hours, while those intended for the dry batch were stored in ambient temperature between 15°C and 25°C. After the storage stage, followed by conditioning at 15°C for at least 2 hours, the indirect tensile strength of the specimens in each batch was measured. The ITSR ratio was then calculated, as the ratio of indirect tensile strength of the batch of wet specimens and that of the batch of dry specimens.

BULK DENSITY OF THE PREPARED SPECIMENS

The average results obtained for each batch are shown in Figures 7 and 8 for the dry batch and wet batch, respectively. One laboratory was discarded based on Cochran and Grubbs statistical tests[7, 11]. The obtained repeatability and reproducibility are indicated in Table 5.

RESULTS AND INTERPRETATION

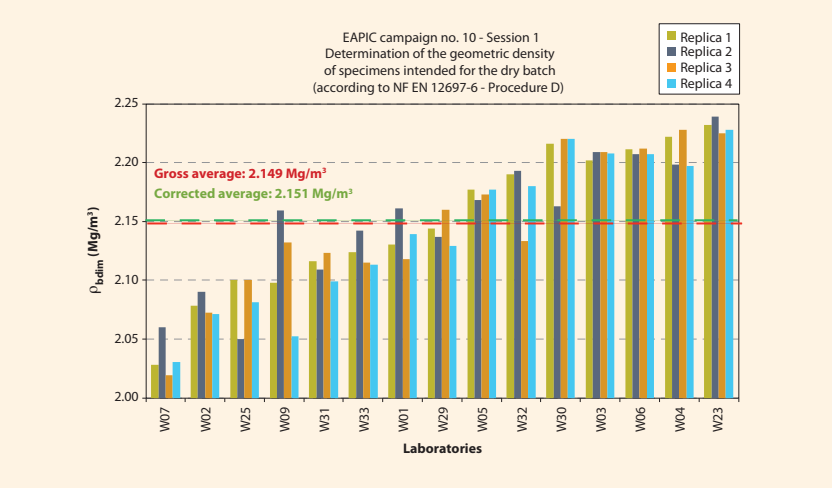
The results obtained for each replica are presented in Figure 9.

The obtained ITSR values varied from 27% to 84%, around an average value of 58%. They were very highly scattered, but no laboratory was discarded by the statistical tests. The repeatability of the obtained results was slightly lower than the value given in Standard EN 12697-12 for Method A (Table 6).

Reproducibility was much higher. This degradation may be partly attributed to the different preparation methods used.

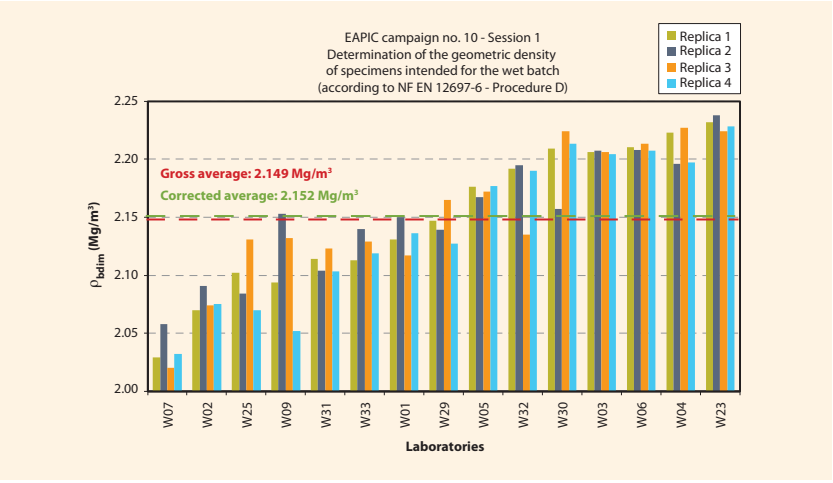
–Figure 7–

ρ_{bdim} values of specimen batches intended for dry storage.



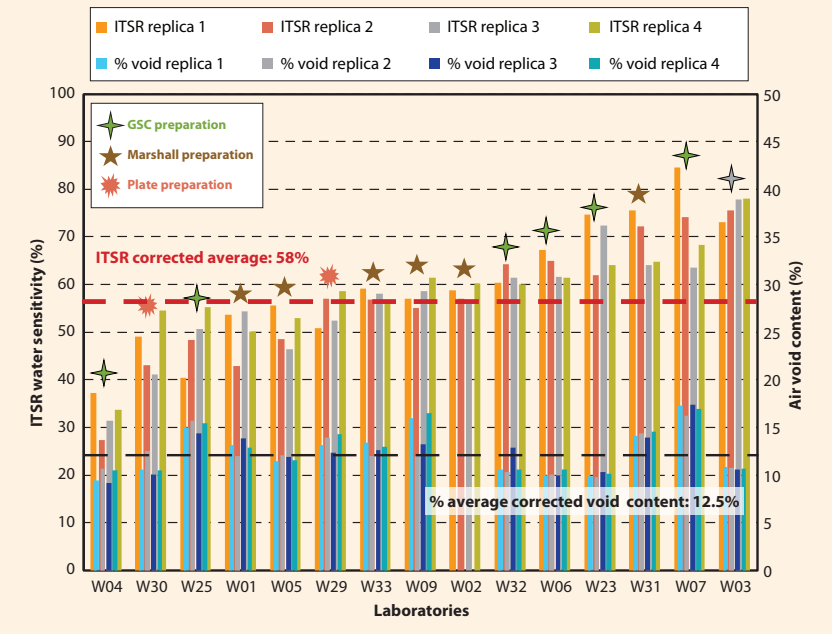
–Figure 8–

ρ_{bdim} values of specimen batches intended for storage in water.



–Figure 9–

ITSR water sensitivity and air void content for each replica.



–Table 5–

Reliability data of the ρ_{bdim} values of this campaign.

	Reliability	ρ_{bdim} (Mg/m³) EAPIC no. 10-1-014 (2015)[9]
Number of laboratories		14
Dry batch	r	0.045
	R	0.172
Wet batch	r	0.046
	R	0.168

–Table 6–

Reliability data of ITSR water sensitivity in this campaign versus Standard 12697-12.

	ITSR (%) EAPIC no. 10-01-14 (2015)[9]	ITSR (%) NF EN 12697-12[2]
Number of laboratories	15	-
r	13.2	15
R	33.8	23

The average voids content based on Equation 1 varied from 9% to 17.4% around an average value of 12.5% and a standard deviation of 2.2% (Figure 9). However, no apparent relationship was noticeable between the voids content and the water sensitivity according to ITSR.

INTERPRETATION OF THE RESULTS OBTAINED ACCORDING TO METHODS A AND B

COMPARISON OF THE WATER SENSITIVITY RESULTS

The average of the i/C results (77%) was considerably higher than the ITSR results (58%). It follows that Method A is significantly more severe than Method B under the conditions of this experiment and at this value level.

However, the results scattering is very high, as evidenced by the precision values:

- The repeatability values (r = 9.4 and 13.2) are fairly close to the values indicated in the test standard (r = 7.8 and 15).
- The reproducibility values (R = 19 and 33.8) are markedly higher that the values indicated in the test standard (R = 13.4 and 23).

Thus, under this campaign conditions, we note that Method A is approximately 1.4 times more scattered than Method B with regard to repeatability and 1.8 times more scattered in terms of reproducibility. While repeatability shows acceptable control of this test by the various laboratories, the high reproducibility raises a problem, in particular for the approval of products within the scope of mix design and CE marking.

This may be partly due to the method and, for Method A, to flexibility given in the specimen preparation.

Accordingly, an improvement in this method is possible from the outset by imposing the method of specimen preparation.

The ITSR analysis according to preparation method **(Figure 10)** shows that specimen preparation by GC caused increased scatter compared to the Marshall and plate compaction method. The lowest scatter was obtained using the roller compaction method; however, this method was used by only two laboratories.

COMPARISON OF CRUSHING STRENGTH VALUES

On average, the compressive strength values obtained using Method B are 5 times higher than those obtained by indirect traction according to Method A. These results are displayed in **Figures 11** for each batch of specimens. The obtained r and R scatter values for both methods are provided in **Table 7**.

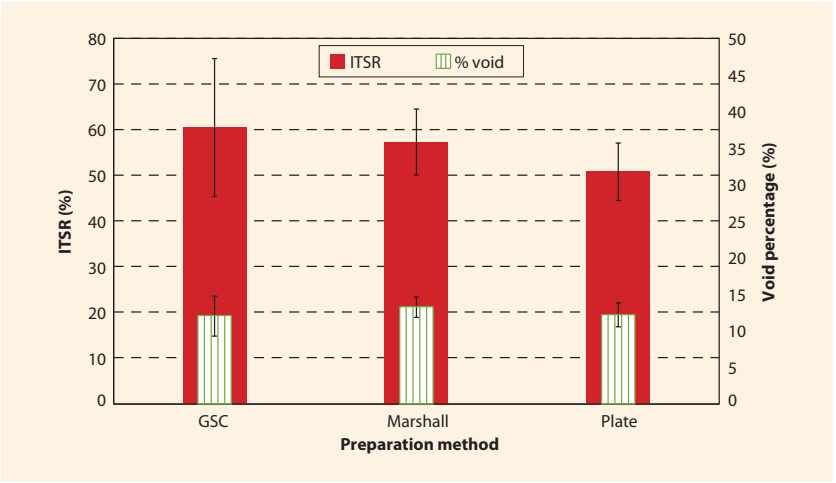
The variation coefficients CV (CV = standard deviation/average, in %) for repeatability (CVr) and reproducibility CVR) were evaluated. These results show very high scatter in terms of the reproducibility of the strength values obtained for Method A, which could partly explain the high scatter obtained for the water sensitivity according to ITSR.

CONCLUSION AND RECOMMENDATIONS

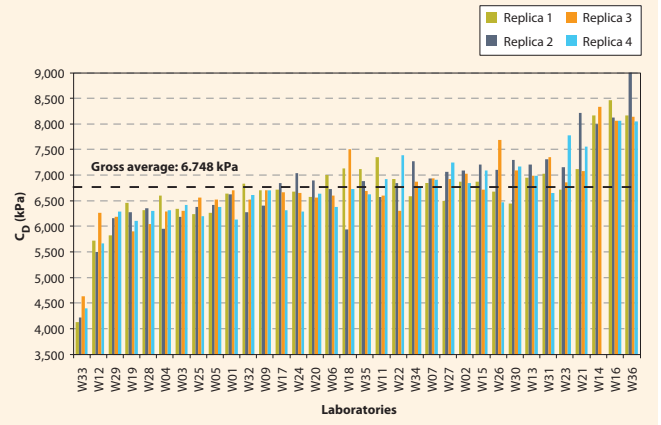
This campaign enabled assessing:

- The performance of laboratories to carry out the i/C (Duriez) and ITSR water sensitivity tests;
- The performance consistency of all the laboratories concerning the measurements of maximum and bulk densities of the asphalt mixes.

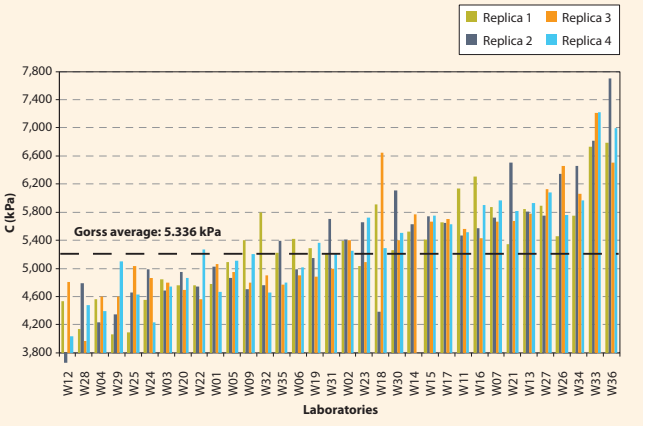
–Figure 10–
Impact of the specimen preparation method on the tests of water sensitivity by the ITSR method and the voids content of asphalt mixes.



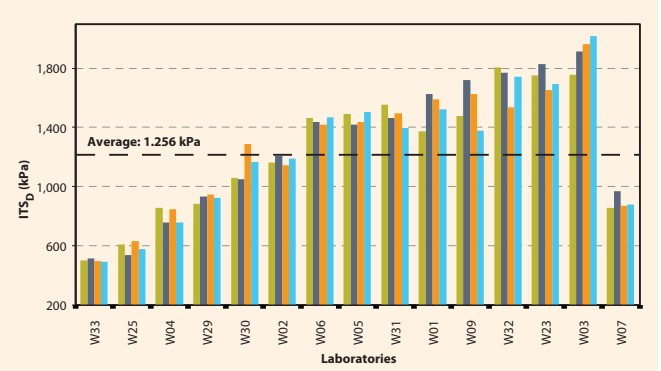
–Figures 11–
The strength values measured during the crushing tests (Method A and Method B for determining water sensitivity).



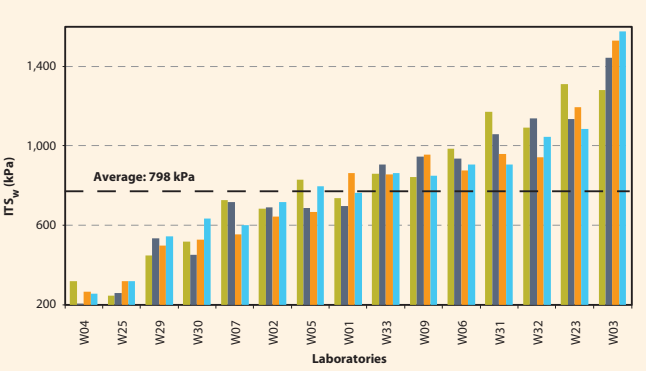
a) Compaction resistance of dry batches – Method B.



b) Compaction resistance of wet batches – Method B.



c) Indirect traction resistance of dry batches – Method A.



d) Indirect traction resistance of wet batches – Method A.

–Table 7–
Comparison of the precision data for the strength values of this campaign.

	Precision	Method B		Method A	
		Strength in kPa	CV	Strength in kPa	CV
Dry batch	r	856	5%	224	6%
	R	2,103	11%	1,232	35%
Wet batch	r	975	7%	210	9%
	R	2,034	14%	926	42%

The average of the results obtained in the i/C test, under these conditions, is 1.4 times higher than the average obtained in the ITSR test.
Moreover, the results of the water sensitivity test are highly scattered, irrespective of the method used. We also note a scatter value in terms of reproducibility that is higher than the one provided in the standard for i/C and for ITSR. The reproducibility value may raise problems, in particular for the approval of products within the scope of mix design and CE marking.

With regard to the measurement of maximum density, we have noted a clear decrease of the scattering compared to the previous EAPIC campaign no. 2-1-003. This study may serve as basis for proposing steps for collective improvement to reduce the scatter of Method A (ITSR):
• Selecting a unique compacting process for method A and/or targeting percentages of voids for Method A;
• Defining the storage time of expanded asphalt mixes under controlled temperature before moulding the specimens. ■

The EAPIC working group

The EAPIC dedicated group is a component of the Operational Committee for Inter-Laboratory Comparison Qualification (COQC) of IDRRIM. Currently, it has 7 members coming from administrative and professional positions. Its main activity is organising inter-comparison tests in which laboratories can demonstrate their performance levels in carrying out routine pavement tests under real-life conditions.

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