EAPIC

ESSAIS D'APTITUDE PAR ESSAI DE COMPARAISON

RESULTS PRESENTATION REPORT

N° 8.2.17

About the test

MEASUREMENT OF STIFFNESS

According to standard NF EN 12697-26 June 2012

CERTIFIED ISO 9001

January 29, 2019 by G. PIOT - Executive Cell of EAPIC
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Preamble

The road community remembers EAPIC 8.1.12 session about the stiffness modulus of bituminous mixtures in 2011-2012. Dealing with such an important characteristic for pavements design, the high observed values for reproducibility had then worried more than one of us.

Five years later, the test standard has already changed, offering a sixth measurement method, and the whole normative context continues to evolve: the new standards about tests, products, and pavement design, which concern this characteristic, will be published tomorrow. Therefore it was important to organise a new EAPIC session about this test method, to assess an eventual evolution of laboratory practices. Such is the subject of the present report.

For the organisation of this session, the Specialised Group wished to change some technical elements in comparison with 8.1.12 session. Thus, the selected mix formula aimed to simultaneously lead to a normal - but non excessive - scattering in the void percentage, and to modulus values typical of hot mixes currently used in road construction.

You then performed your tests and sent your results, most of you within the time limit. Unfortunately others have wished additional time, leading to additional internal cost for EAPIC. We chose not to charge these extra costs. Please do respect the deadlines when participating in coming sessions, so that no one is penalised.

The results of this session are now given to your appreciation. First observation: the two above mentioned technical goals are achieved. Then it can be noticed that, even if the participating laboratories are lightly less numerous (32 today against 38 in 2012), some methods are equally practised than they were six years ago (method A) and others are less practised (methods C and E). Method C followed by method A remain the most practised without any doubt. It is also observed that some laboratories chose to participate with several methods.

Precision values of methods A and C are better than they were, although their reproducibility remains high. Their repeatability values are clearly lower, partly supported by the fact that - for logistical reasons – the laboratories made the four replicas for each method on the same batch of test specimens. For other methods, precisions values could not be calculated because of too little results.

Regarding the averages of 15°C results, they are quite similar, except the one of method C which is lower.

These statements can provide different investigations which are not in our role. The main mission of EAPIC is indeed to set a report, as objective as possible, of laboratory practise in conditions representative of their current activity: mix design, checking, etc. However, the abundant information collected in this session may be anonymously forwarded to the National Commission for Road Tests to provide input for its work on these test methods.

Two novelties in conclusion. The present report gives the use of collected funds from participating laboratories. And finally, in accordance with the decision of Specialised Group at the end of last year, this report will be - as the previous one - translated into English and published on the IDRRIM website. So, the results of this session will be accessible in France and far beyond our borders.

Kind regards,

For EAPIC,

Michel SAUBOT

Summary

Session organization and data collection

Homogenized batches, comprised of four grain fractions, are supplied to participating laboratories. They must determine the modulus of the mixes according to standard NF EN 12697-26 of June 2012 and the conditions specified in the particular instructions of this series.

The selected formula is described as follows:

Fines	2.0 %
0/2	31.0 %
2/6	15.3 %
6/10	15.0 %
10/14	32.0 %
Bitumen 35/50	4.7 %

MVRe [mix maximum density] value has been determined by EAPIC Specialised Group according to NF EN 12697-5 (method A in water) and sent to all the laboratories.

MVRe value: 2.640 Mg/m^3

In order to limit the quantity of materials to be supplied, the test specimens of replica 1 were reused for replicas 2 to 4.

The campaign unfolded as follows:

- Shipping of samples in January / February 2018.
- Results delivery from participating laboratories at latest 15th of April 2018, except for those located outside continental France whose delivery date was postponed because of shipping time (delivery date at latest 15th of June 2018).
  The number of participants to this session is 32 laboratories which had the possibility of carrying out several methods.
  48 determination results forms were received, distributed as follow:
  - 16 results forms according to Annex A
  - 21 results forms according to Annex C 15°C
  - 3 results forms according to Annex D
  - 6 results forms according to Annex E
  - 2 results forms according to Annex F

  The requests for additional delays transmitted to the EAPIC Executive Unit have all been accepted.
- Publication of the result report in August 2018.

Preparation and shipping of samples
Support laboratory : Cerema Ouest – Department Laboratory of Angers

Materials

Each laboratory received one or two pallet(s) depending on the quantities of materials needed to carry out their tests.

Each pallet is composed of the following fractions:

- 2 bags of 25 kg of 10/14 ;
- 1 bag of 25 kg of 6/10 ;
- 1 bag of 25 kg of 2/6 ;
- 2 bags of 25 kg of 0/2 ;
- 1 bag of 25 kg de fines ;
- 1 bucket of 10 kg of bitumen.

Preparation

To carry out this campaign, 348 bags of aggregates were prepared and bagged by the Cerema Ouest – Department Laboratory of Angers:

- 106 bags of 25 kg of 10/14 ;
- 68 bags of 25 kg of 6/10 ;
- 68 bags of 25 kg of 2/6 ;
- 106 bags of 25 kg of 0/2.

Shipping of materials

The materials were sent by the Cerema Ouest - Department Laboratory of Angers.
The set of bags and bitumen buckets required for the campaign was set up on pallet before shipment. Only shipments for participants outside metropolitan France were made in wooden crates (due to shipping).

Verification of aggregates samples homogeneity
Support laboratory: Cerema East Central – Department Laboratory of Autun

In order to check that all the samples are homogeneous, the indication of Annex B of the ISO 13528 standard of December 2015, which gives a statistical method used in inter-laboratory comparison aptitude tests, are applied. This appendix is based on the comparison of the standard deviation inter-samples S_s to the standard deviation for the assessment of aptitude $\hat{\sigma}$. Samples are considered to be homogeneous if $S_s \leq 0.3 \times \hat{\sigma}$.

The homogeneity criteria considered by the "EAPIC Executive Cell" is the pre-dried aggregates density determined according to the standard NF EN 1097-6 annex A, of January 2014.

For each bag, the values of the inter-sample standard deviation are compared to the estimate of the homogeneity criterion 0.3 x $\hat{\sigma}$.

	MVR 0/2	**MVR 2/6**	**MVR 6/10**	**MVR 10/14**
Average	2.840	2.855	2.876	2.888
Origin of r and R	EAPIC 5th Campaign	NF EN 1097-6	NF EN 1097-6	NF EN 1097-6
r	0.022	0.019	0.019	0.019
R	0.074	0.042	0.042	0.042
0.3 x $\hat{\sigma}$	0.008	0.004	0.004	0.004
Standard deviation between samples S_s	0.0003	0.001	0.002	0.002
Validation $S_s \leq 0.3 \times \hat{\sigma}$	condition fulfilled	condition fulfilled	condition fulfilled	condition fulfilled

Criteria is fulfilled for each test.

Therefore, it can be concluded that samples are sufficiently homogeneous.

Complementary tests for the characterization of bitumen samples
Support laboratory: Cerema East Central – Department Laboratory of Autun

Additionnal tests were carried out on bitumen samples in order to verify their characteristics and homogeneity:

- The needle penetration, determined according to the standard NF EN 1426 of June 2007 ;
- The softening point, determined according to the standard NF EN 1427 of June 2007.
- The dynamic complex modulus G*, at 15°C and 10 Hz, determined according to NF EN 14770

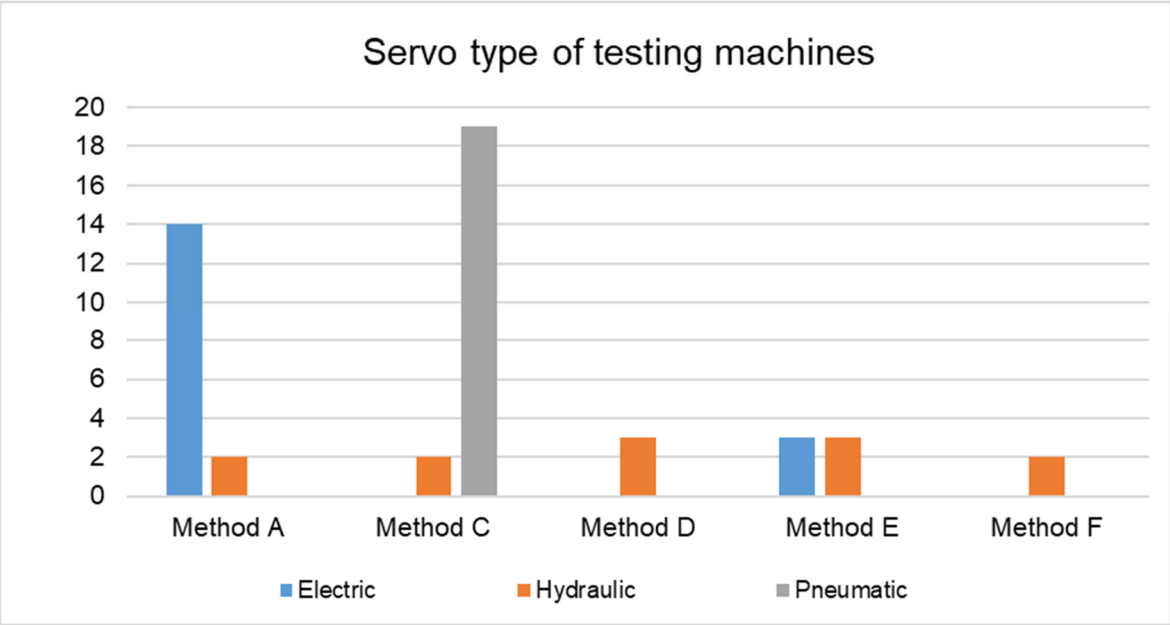
The results of 5 pots randomly selected are shown in the following table:

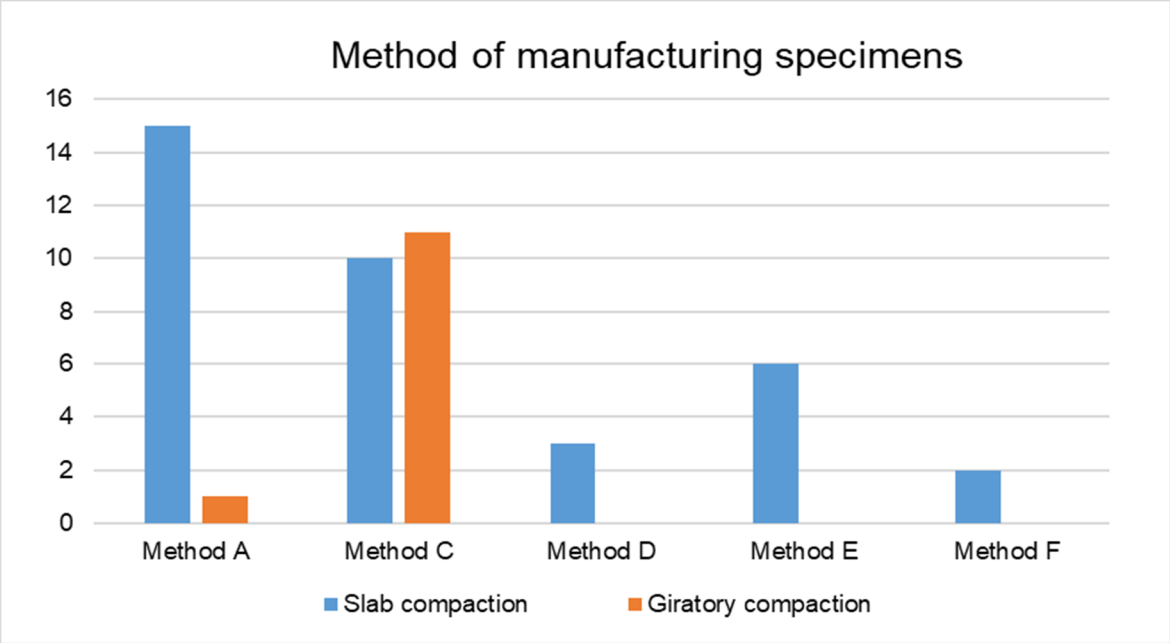
	1	2	3	4	5	Average	r
Penetrability (0,1 mm)	41	42	42	41	42	41.6	2
TBA (°C)	53.8	53.4	53.4	53.8	53.6	53.6	1
G* (kPa)	$3.25.10^4$	$3.17.10^4$	$3.01.10^4$	$3.22.10^4$	$3.21.10^4$	$3.17.10^4$	$3.03.10^3$

(*) the r of G was taken from the 2017 BNPé round robbin test report « Flexural creep stiffness (Bending Beam Rheometer - BBR) and complex shear modulus & phase angle (Dynamic Shear Rheometer - DSR) tests on paving grade bitumen » page 90/105*
This value of r was obtained for the following test conditions: Temeprature of 15°C, Frequency of 1.59 Hz and realized with a DSR with a plateau diameter of 8 mm.

The measured values satisfy the repeatability criteria.

Materials


Servo type of testing machines
20
18
16
14
12
10
8
6
4
2
0
Method A
Method C
Method D
Method E
Method F
Electric
Hydraulic
Pneumatic


Method of manufacturing specimens
16
14
12
10
8
6
4
2
0
Method A
Method C
Method D
Method E
Method F
Slab compaction
Giratory compaction

Data processing

Data processing is based on series of standards ISO 5725 « Application of statistics – Accuracy (trueness and precision) of measurement methods and results ».
The treatment is performed using an Excel table. Results are checked later using the XLSTAT software.

Graphic representations

The raw results are represented in histogram diagrams.

Histograms express the results obtained by the participating laboratories. The corrected average (after removing the outliers) is placed on the graph.

Statistical tests

On the raw results, the following statistical tests are applied:

- Cochran test (intra-laboratory variability): detection of variance outliers, in the statistical sense of the results in a laboratory.
- Simple Grubbs test or possibly double (between laboratories variability): detection of averages outliers, among the population of laboratories.

Results exceeding the critical value at 1 % are reported as outliers and removed from the statistical treatment that retains only the corrected data.
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Z-Score

Z-Score is the number of standard deviations that are above or below the population average. Z-Score is calculated from the following formula:
$z = (|X - \mu| / \sigma) * 2$
where :

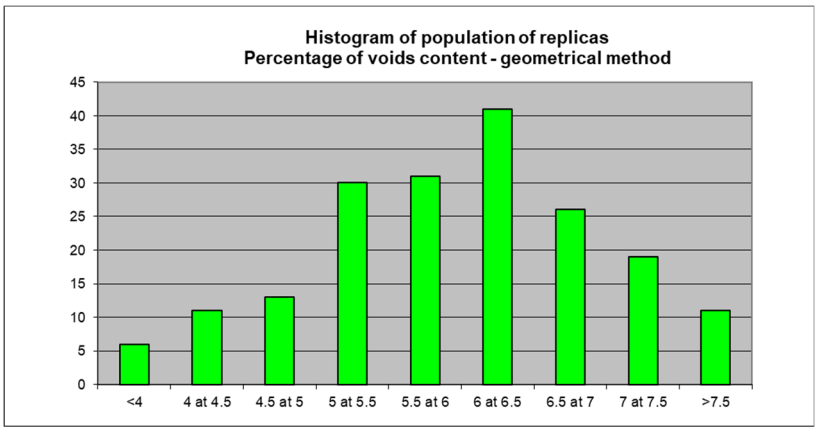
z is z-score
X is the laboratory average
μ is the average of population
σ is the standard deviation of the population.

Determination of air void contents from the bulk density (NF EN 12697-6 procedure D August 2012)

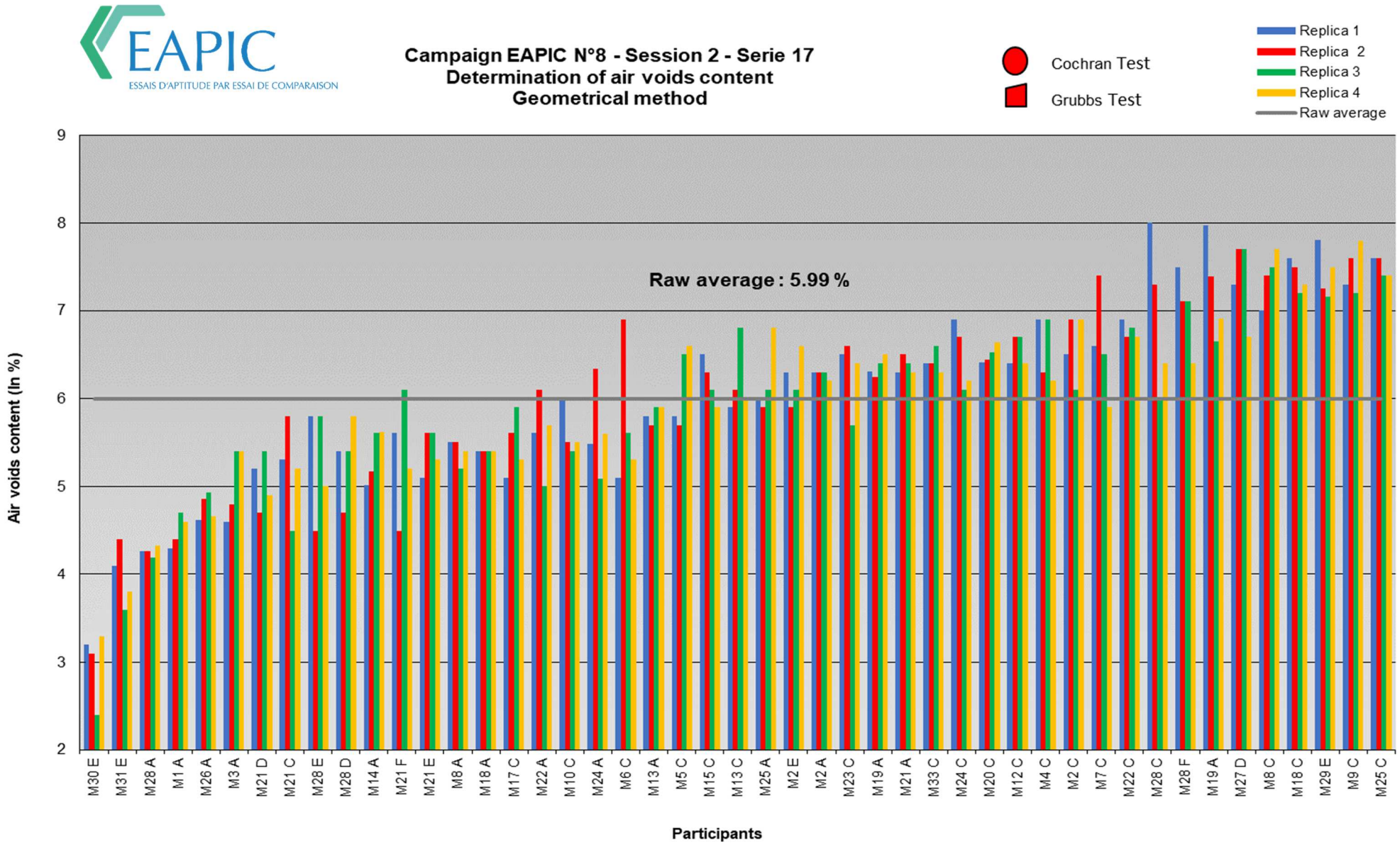
Graphical representation

		Raw data	Results rejected by statistical tests	Standard (NF EN 12697-8)
	Number of results taken in account	47	/	
In %	Average m	5.99		
	Standard deviation repeatability	0.39		
	Repeatability r	1.092		r = 1.1
	Standard deviation Reproducitibility	1.045		
	Reproducitibility R	2.925		R = 2.2

Raw data



Raw data validated by XLSTAT software:
m = 5.993


EAPIC
ESSAIS D'APTITUDE PAR ESSAI DE COMPARAISON
Campaign EAPIC N°8 - Session 2 - Serie 17
Determination of air voids content
Geometrical method
Cochran Test
Grubbs Test
Replica 1
Replica 2
Replica 3
Replica 4
Raw average
Raw average : 5.99 %
Air voids content (In %)
9
8
7
6
5
4
3
2
M30 E
M31 E
M28 A
M1 A
M26 A
M3 A
M21 D
M21 C
M28 E
M28 D
M14 A
M21 F
M21 E
M8 A
M18 A
M17 C
M22 A
M10 C
M24 A
M6 C
M13 A
M5 C
M15 C
M13 C
M25 A
M2 E
M2 A
M23 C
M19 A
M21 A
M33 C
M24 C
M20 C
M12 C
M4 C
M2 C
M7 C
M22 C
M28 C
M28 F
M19 A
M27 D
M8 C
M18 C
M29 E
M9 C
M25 C
Participants

Average difference on the raw data

Difference less than 1 standard deviation

Code résults	Average deviation	Z-Score
M5 C	0.16	0.30
M13 A	0.17	0.32
M15 C	0.21	0.40
M13 C	0.21	0.40
M25 A	0.21	0.40
M2 E	0.23	0.44
M6 C	0.27	0.51
M2 A	0.28	0.54
M23 C	0.31	0.59
M24 A	0.37	0.71
M19 A	0.37	0.71
M21 A	0.38	0.73
M22 A	0.39	0.75
M10 C	0.39	0.75
M33 C	0.43	0.83
M24 C	0.48	0.92
M20 C	0.51	0.98
M17 C	0.52	0.99

Difference more than 1 standard deviation

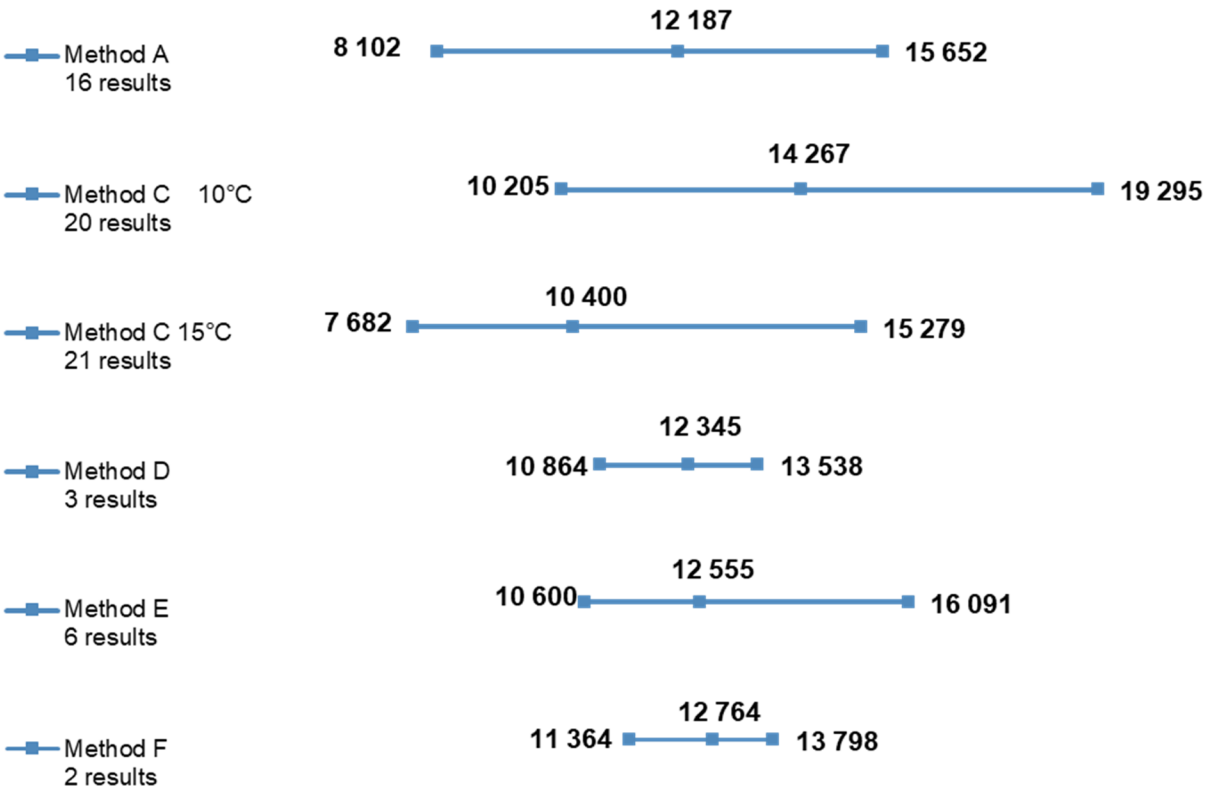
Code résults	Average deviation	Z-Score
M12 C	0.56	1.07
M4 C	0.58	1.11
M8 A	0.59	1.14
M18 A	0.59	1.14
M21 E	0.59	1.14
M2 C	0.61	1.16
M7 C	0.61	1.16
M21 F	0.64	1.23
M14 A	0.65	1.24
M28 D	0.67	1.28
M28 E	0.72	1.38
M22 C	0.78	1.50
M21 C	0.79	1.52
M28 C	0.93	1.78
M21 D	0.94	1.81
M3 A	0.94	1.81
M28 F	1.03	1.98

Difference more than 2 standard deviation

Code résults	Average deviation	Z-Score
M26 A	1.23	2.35
M19 A	1.24	2.37
M27 D	1.36	2.60
M8 C	1.41	2.69
M18 C	1.41	2.69
M29 E	1.44	2.75
M9 C	1.48	2.84
M1 A	1.49	2.86
M25 C	1.51	2.88
M28 A	1.73	3.30
M31 E	2.02	3.86
M30 E	2.99	5.73

Determination of complex modulus (NF EN 12697-26 June 2012)

Range of raw data of modulus values (in MPa) according to the method

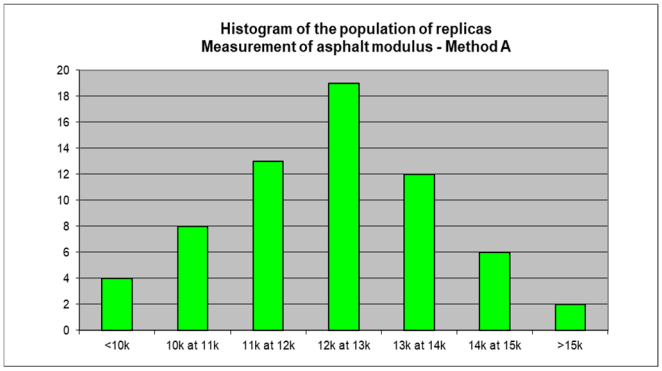


Method A – 2PB-TR / 2PB-PR (15°C – 10Hz)

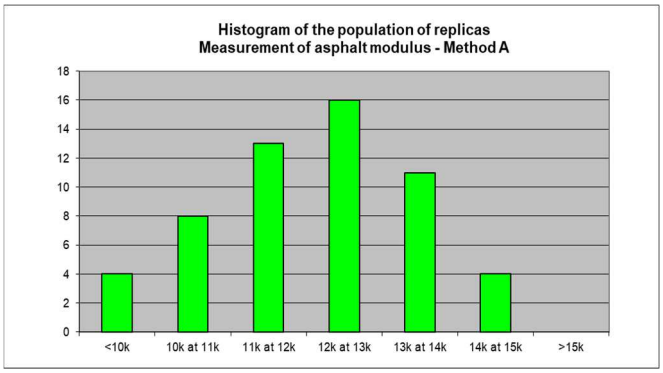
Graphical representation

		Raw data	Results rejected by statistical tests	Corrected data	Standard NF EN 12697-26 Method A	Results of campaign 8.1 Corrected data
In MPa	Number of results taken in account	16	Cochran M1A M21A	14		
	Average m	12 187		11 939		
	Standard deviation repeatability	228		131		
	Repeatability r	639		367	r = 335	r = 377
	Standard deviation Reproducitibility	1 707		1 608		
	Reproducitibility R	4 780		4 501	R = 2 740	R = 6 289

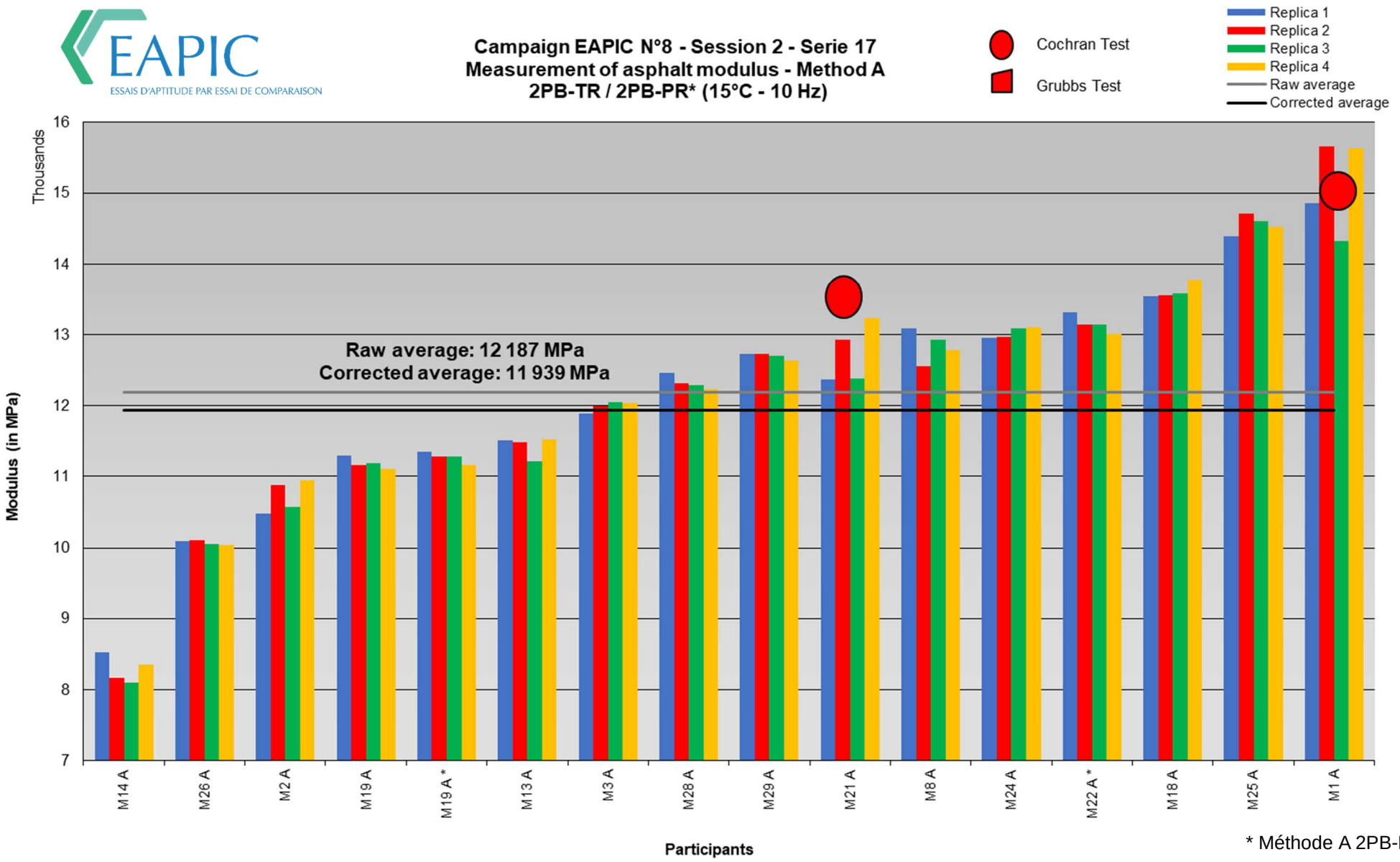
Raw data



Corrected data



Raw data validated by XLSTAT software:
m = 12 186.56 MPa



* Méthode A 2PB-PR

Average difference on the raw data – Method A (15°C – 10 Hz)

Difference less than 1 standard deviation

Code résults	Average deviation	Z-Score
M28 A	136.75	0.16
M3 A	196.75	0.23
M29 A	511.32	0.60
M21 A	539.57	0.63
M8 A	651.13	0.76
M13 A	757.12	0.89
M24 A	842.07	0.99

Difference less than 2 standard deviations

Code résults	Average deviation	Z-Score
M19 A	921.12	1.08
M22 A	962.25	1.13
M19 A	1005.25	1.18
M18 A	1423.25	1.67
M2 A	1467.81	1.72

Difference more than 2 standard deviations

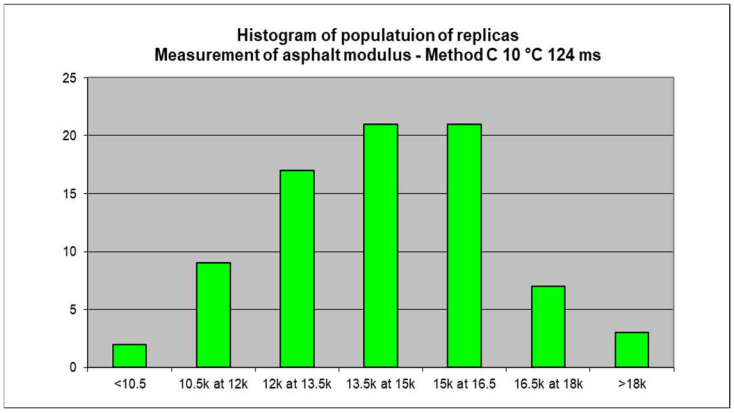
Code résults	Average deviation	Z-Score
M26 A	2112.93	2.48
M25 A	2369.38	2.78
M1 A	2926.13	3.43
M14 A	3900.87	4.57

Method C – IT-CY (10°C – 124 ms)

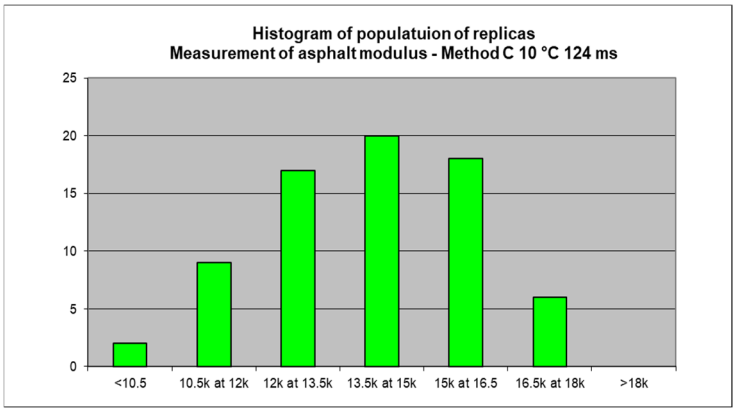
Graphical representation

		Raw data	Results rejected by statistical tests	Corrected data	Standard NF EN 12697-26 Method A
	Number of results taken in account	20	Cochran M18C M10C	18	
In MPa	Average m	14 267		13 960	
	Standard deviation repeatability	324		231	
	Repeatability r	906		648	r = 335
	Standard deviation Reproducitibility	2 077		1 880	
	Reproducitibility R	5 816		5 266	R = 2 740

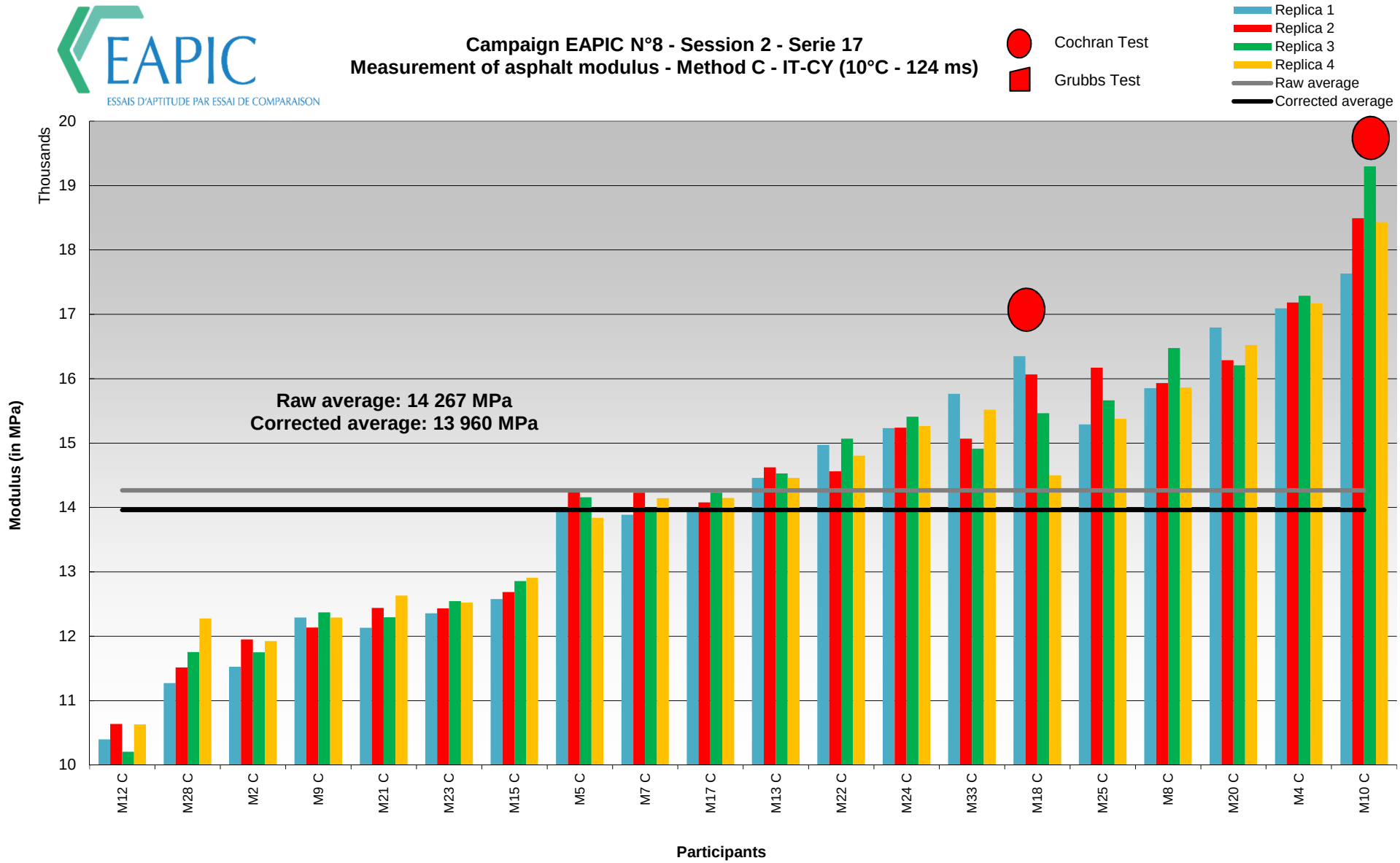
Raw data



Corrected data



Raw data validated by XLSTAT software:
m = 14 267.11 MPa


EAPIC
ESSAIS D'APTITUDE PAR ESSAI DE COMPARAISON
Campaign EAPIC N°8 - Session 2 - Serie 17
Measurement of asphalt modulus - Method C - IT-CY (10°C - 124 ms)
Cochran Test
Grubbs Test
Replica 1
Replica 2
Replica 3
Replica 4
Raw average
Corrected average
Raw average: 14 267 MPa
Corrected average: 13 960 MPa
Thousands
Modulus (in MPa)
20
19
18
17
16
15
14
13
12
11
10
M12 C
M28 C
M2 C
M9 C
M21 C
M23 C
M15 C
M5 C
M7 C
M17 C
M13 C
M22 C
M24 C
M33 C
M18 C
M25 C
M8 C
M20 C
M4 C
M10 C
Participants

Average difference on the raw data – Method C (10°C - 124 ms)

Difference less than 1 standard deviation

Code résults	Average deviation	Z-Score
M17 C	161.11	0.16
M7 C	208.05	0.20
M5 C	223.17	0.21
M13 C	249.27	0.24
M22 C	583.77	0.56
M24 C	1017.89	0.98

Difference less than 2 standard deviations

Code résults	Average deviation	Z-Score
M33 C	1048.08	1.01
M18 C	1325.83	1.28
M25 C	1357.58	1.31
M15 C	1511.23	1.46
M8 C	1762.64	1.70
M23 C	1803.55	1.74
M21 C	1894.42	1.82
M9 C	1995.86	1.92

Difference more than 2 standard deviations

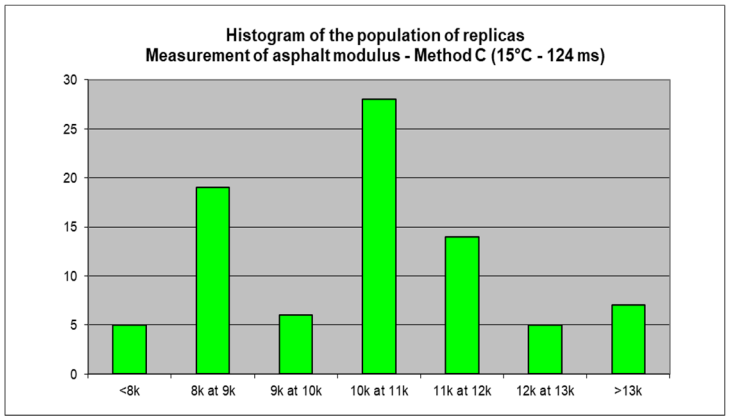
Code résults	Average deviation	Z-Score
M20 C	2185.20	2.10
M2 C	2479.61	2.39
M28 C	2562.98	2.47
M4 C	2914.89	2.81
M12 C	3799.55	3.66
M10 C	4194.39	4.04

Method C – IT-CY (15°C – 124 ms)

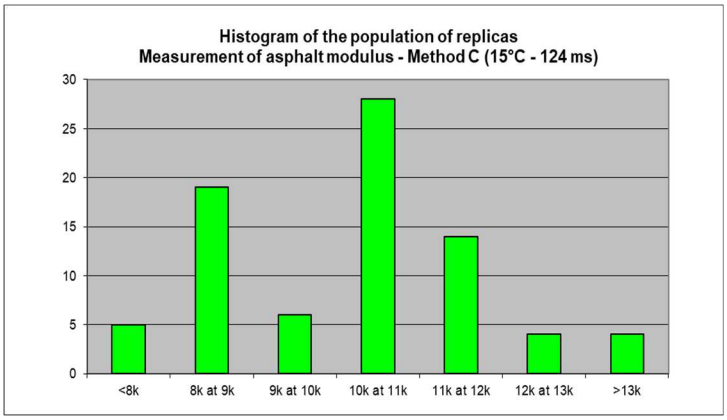
Graphical representation

		Raw data	Results rejected by statistical tests	Corrected data	Standatd NF EN 12697-26 Method A	Results of campaign 8.1 Corrected data
In MPa	Number of results taken in account	21	Cochran M10C	20		
	Average m	10 400		10 240		
	Standard deviation repeatability	218		188		
	Repeatability r	610		526	r = 335	r = 1 187
	Standard deviation Reproducitibility	1 883		1 775		
	Reproducitibility R	5 272		4 971	R = 2 740	R = 8 265

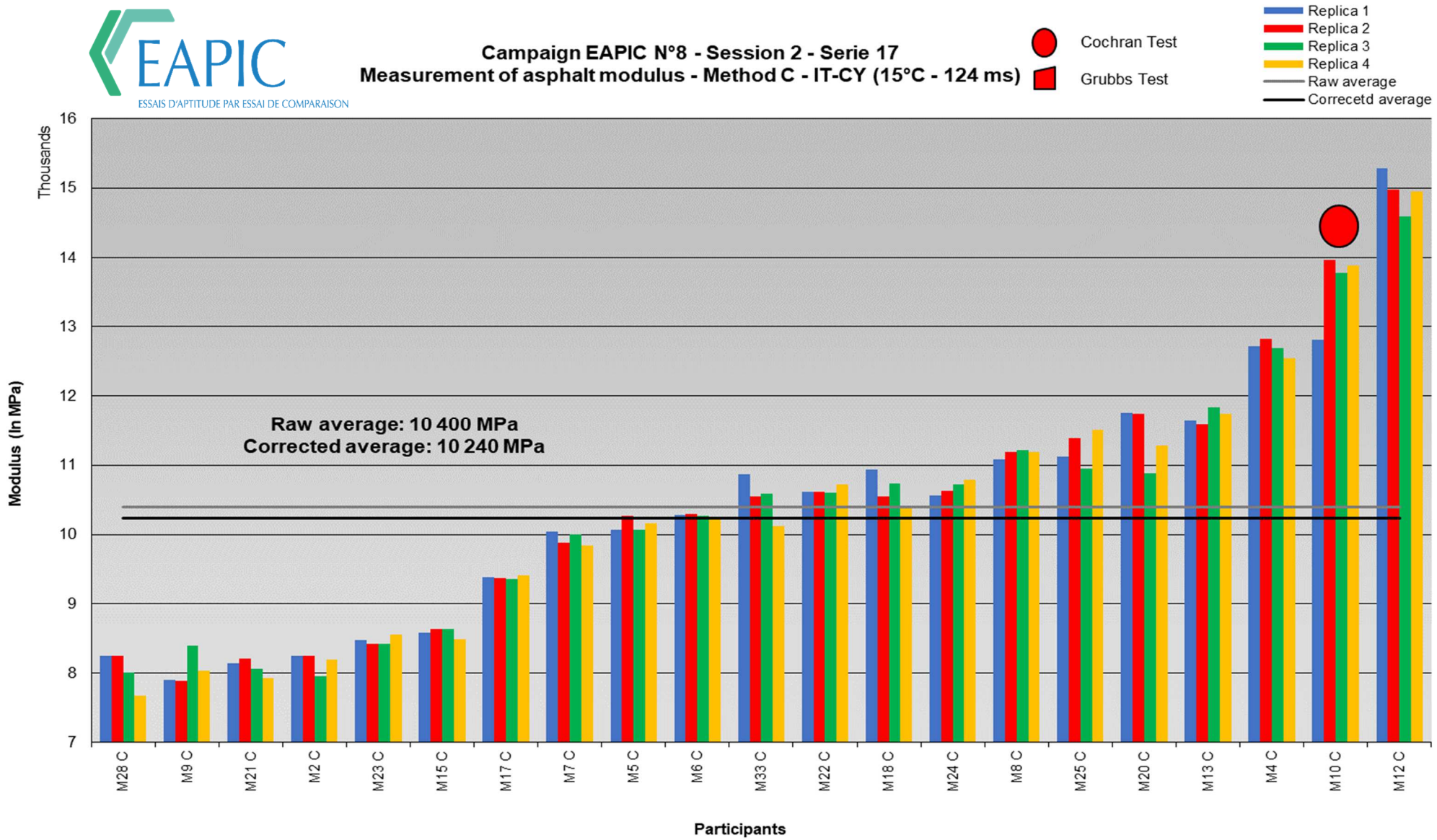
Raw data



Corrected data



Raw data validated by XLSTAT software:
m = 10 400.29 MPa


EAPIC
ESSAIS D'APTITUDE PAR ESSAI DE COMPARAISON
Campaign EAPIC N°8 - Session 2 - Serie 17
Measurement of asphalt modulus - Method C - IT-CY (15°C - 124 ms)
Cochran Test
Grubbs Test
Replica 1
Replica 2
Replica 3
Replica 4
Raw average
Correcetd average
Thousands
Modulus (In MPa)
16
15
14
13
12
11
10
9
8
7
Raw average: 10 400 MPa
Corrected average: 10 240 MPa
M28 C
M9 C
M21 C
M2 C
M23 C
M15 C
M17 C
M7 C
M5 C
M6 C
M33 C
M22 C
M18 C
M24 C
M8 C
M25 C
M20 C
M13 C
M4 C
M10 C
M12 C
Participants

Average difference on the raw data – Method C (15°C – 124 ms)

Difference less than 1 standard deviation

Code résults	Average deviation	Z-Score
M33 C	129.59	0.14
M6 C	130.10	0.14
M22 C	239.15	0.25
M18 C	254.15	0.27
M5 C	256.35	0.27
M24 C	275.97	0.29
M7 C	458.28	0.49
M8 C	764.90	0.81
M25 C	838.53	0.89

Difference less than 2 standard deviations

Code résults	Average deviation	Z-Score
M20 C	1013.84	1.08
M17 C	1023.16	1.09
M13 C	1299.22	1.38
M15 C	1817.78	1.93

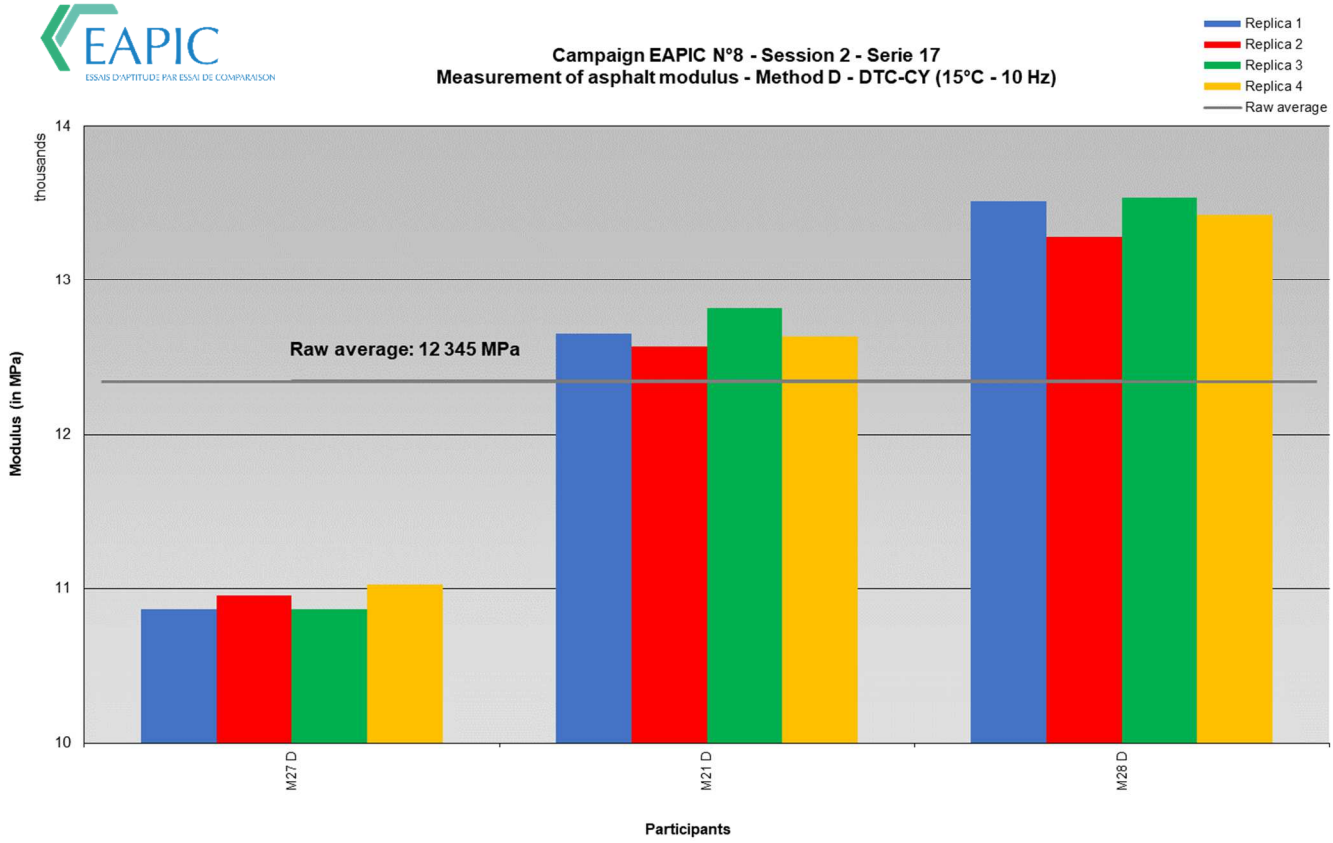
Difference more than 2 standard deviations

Code résults	Average deviation	Z-Score
M23 C	1929.47	2.05
M2 C	2237.78	2.38
M4 C	2292.53	2.43
M21 C	2312.91	2.46
M9 C	2344.35	2.49
M28 C	2353.28	2.50
M10 C	3208.84	3.41
M12 C	4546.72	4.83

Method D – DTC-CY (15°C – 10 Hz)

Graphical representation

Number of results: 3
Average: 12 345 MPa

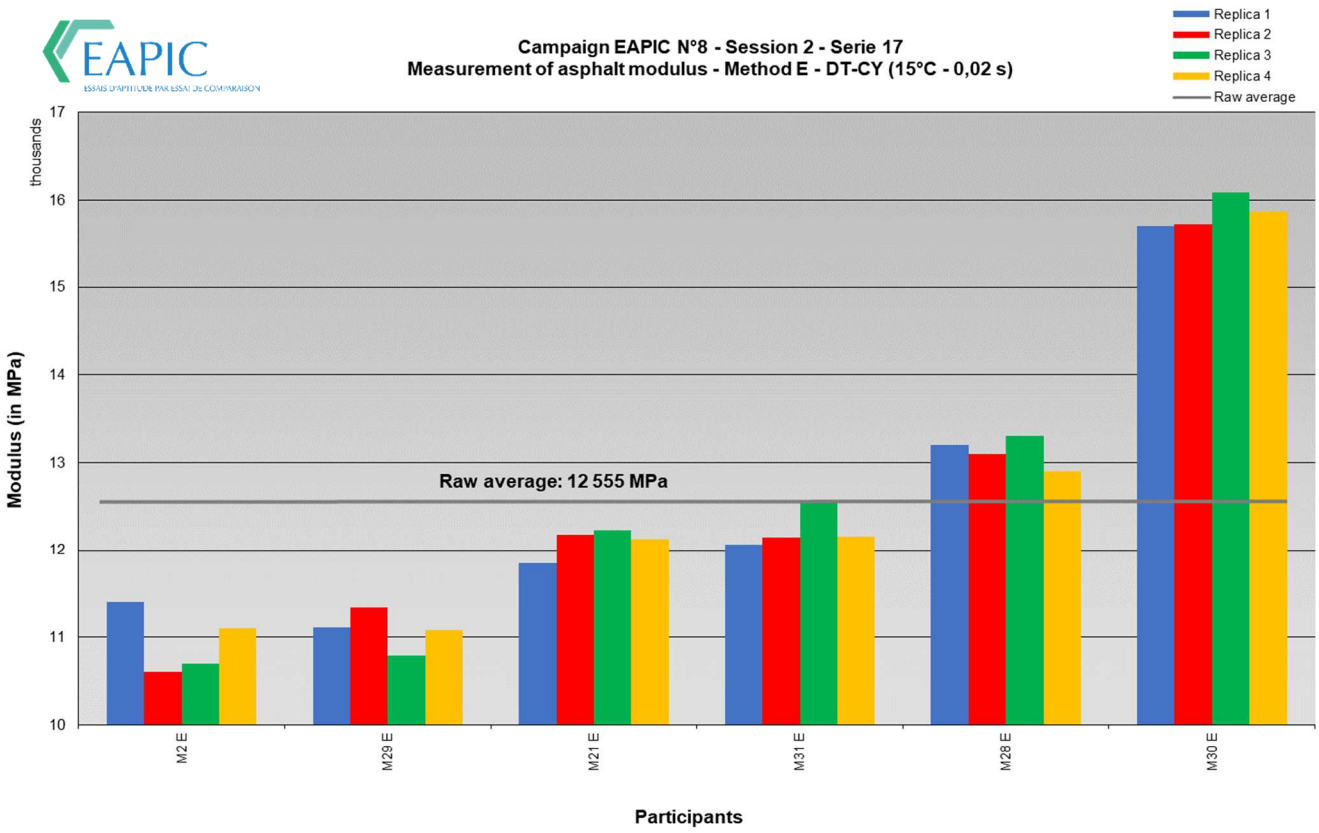


Raw data validated by XLSTAT software:
m = 12 344.78 MPa

Method E - DT-CY (15°C – 0.02 s)

Graphical representation

Number of results: 6
Average: 12 555 MPa

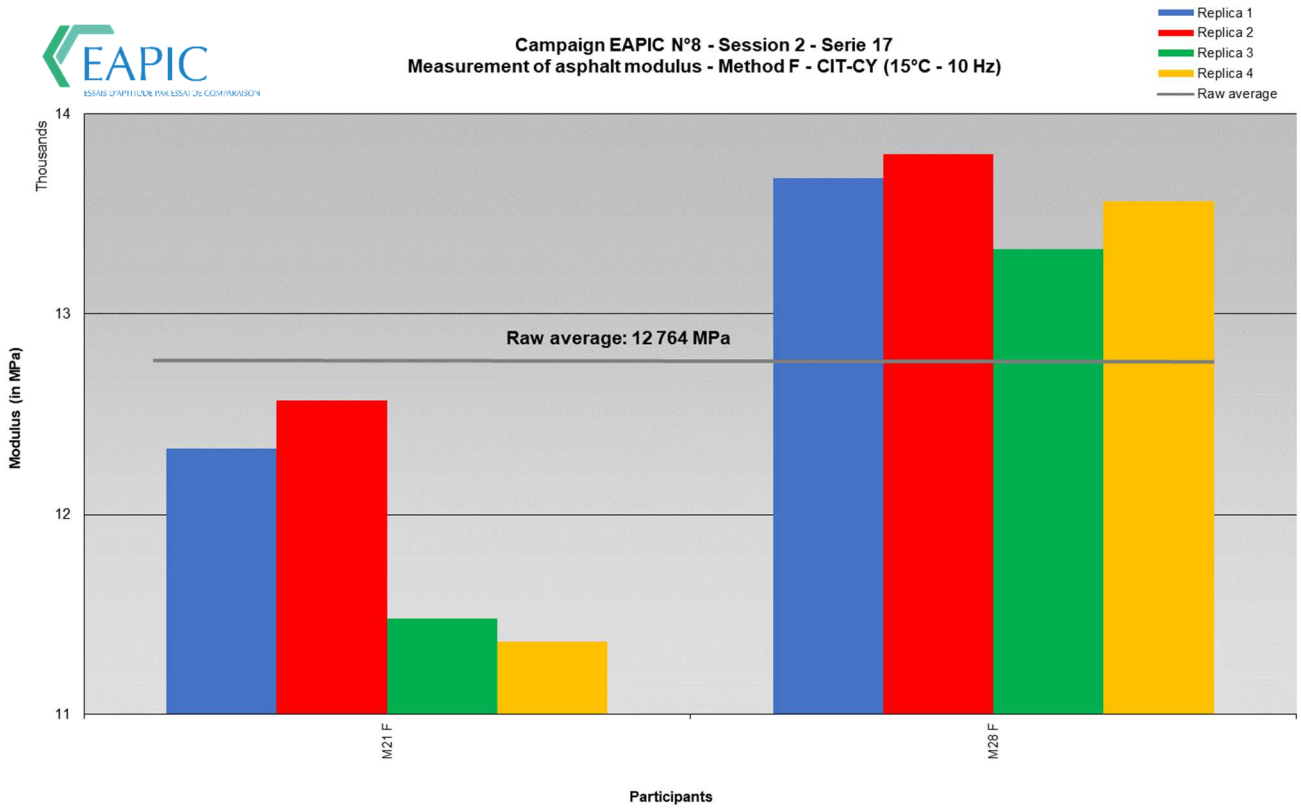


Raw data validated by XLSTAT software:
m = 12 554.58 MPa

Method F - CIT-CY (15°C – 10 Hz)

Graphical representation

Number of results: 2
Average: 12 764 MPa



Raw data validated by XLSTAT software:
m = 12 763.81 MPa

Organisation of EAPIC

The Specialized Group « Inter Comparison Aptitude Tests (EAPIC) » is placed under the aegis of the Operational Committee for Qualification Inter-Laboratory Comparison (COQC) of the Institute of Roads, Streets and Infrastructures for Mobility (IDRRIM) chaired by Didier DESMOULINS (acting) (assistant : Anaïs FERMINE)

The ***Specialized Group*** relies on the ***Executive Cell*** to organize the test campaign. The logistic for the preparation of sample is provided by the ***Support Laboratory.***

EAPIC Specialized Group

General Secretary : Michel SAUBOT

Members :

BADROUILLET Christophe
FAUCON-DUMONT Stéphane
PERIGOIS Stéphanie
PIOT Géraldine
PRIEZ Christophe
SOME Ciryle

EAPIC Executive Cell

Cerema IDF- Site de Sourdun : PIOT Géraldine & SOME Ciryle

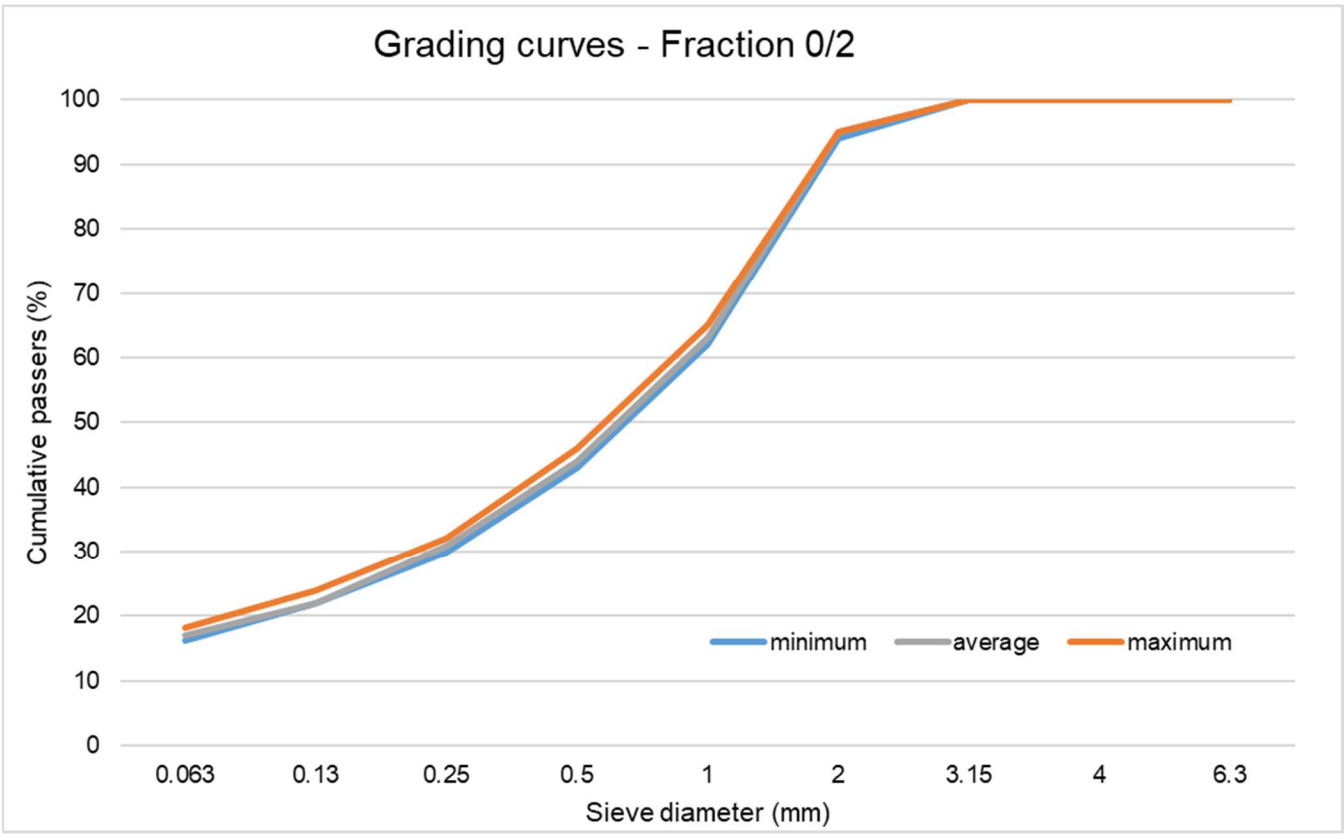
EAPIC Support Laboratories

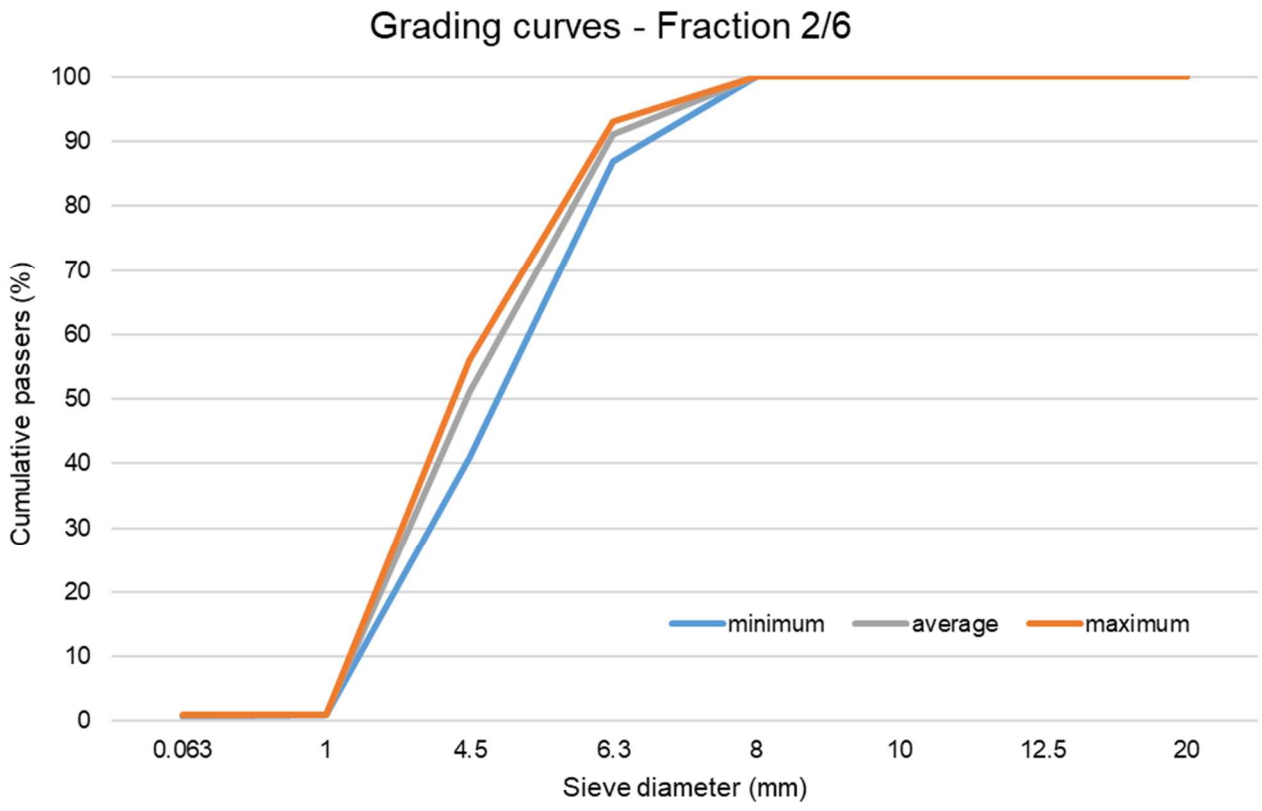
Cerema Ouest Département Laboratoire d'Angers : PERIGOIS Stéphanie
Cerema East Central Département Laboratoire d'Autun : BADROUILLET Christophe

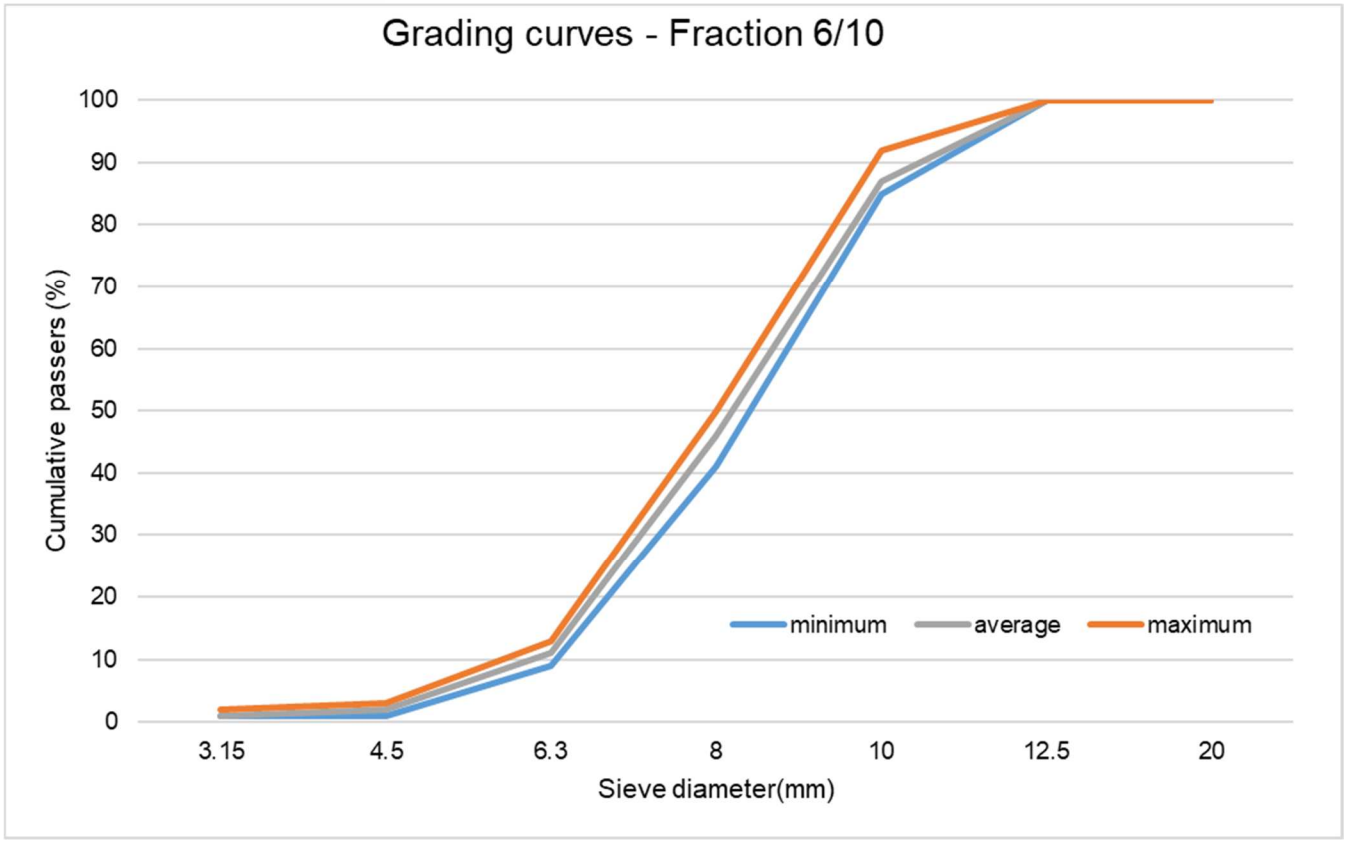
Annexes

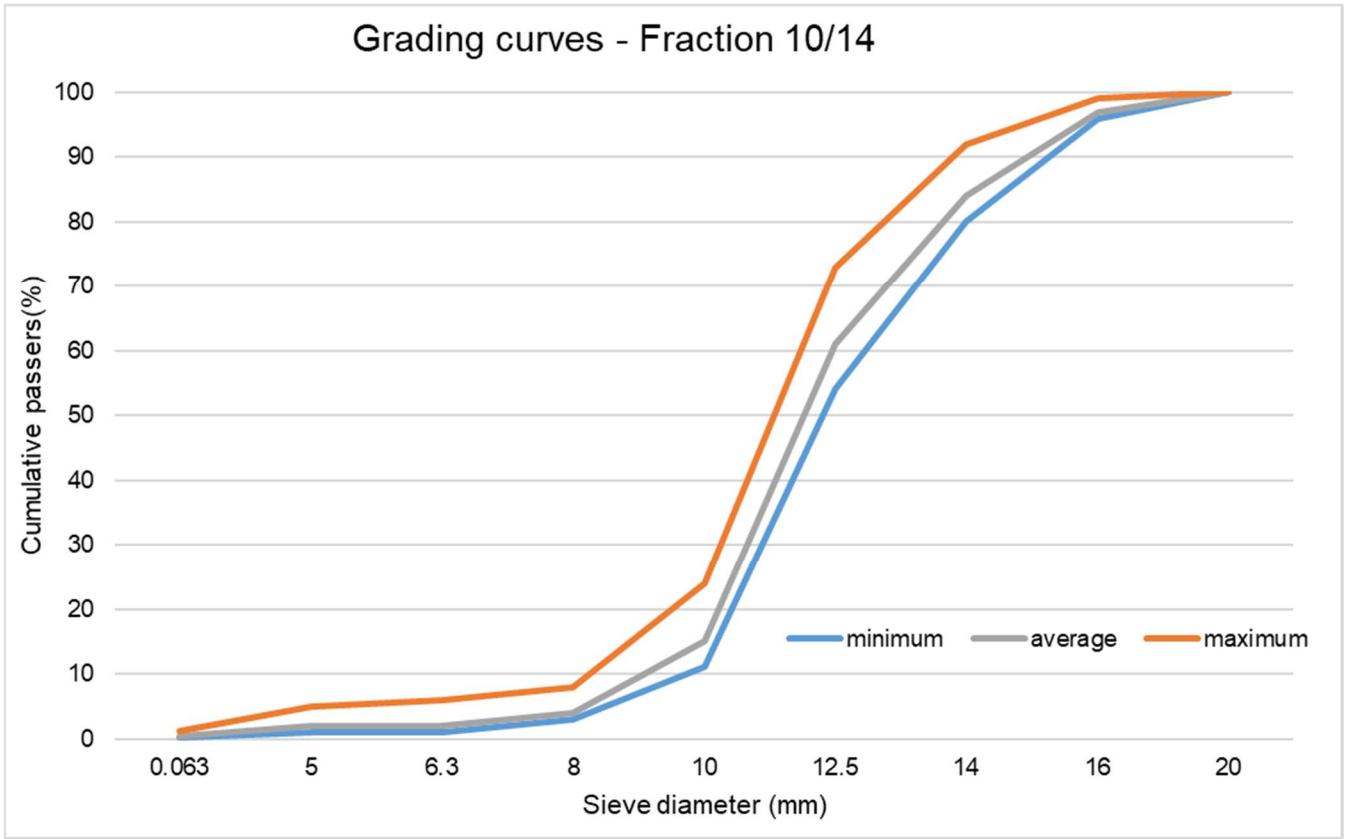
- Grading curves
- Determination of air void contents by gamma rays
- Balance sheet

Grading curves






Grading curves - Fraction 6/10
Cumulative passers (%)
100
90
80
70
60
50
40
30
20
10
0
3.15
4.5
6.3
8
10
12.5
20
Sieve diameter(mm)
minimum
average
maximum

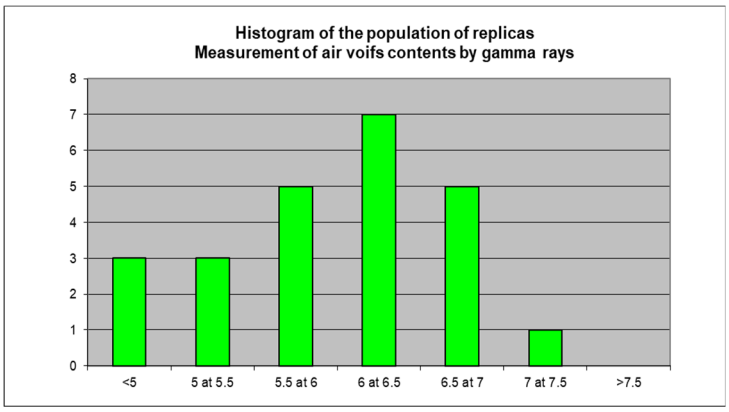

Grading curves - Fraction 10/14
Cumulative passers(%)
100
90
80
70
60
50
40
30
20
10
0
0.063
5
6.3
8
10
12.5
14
16
20
Sieve diameter (mm)
minimum
average
maximum

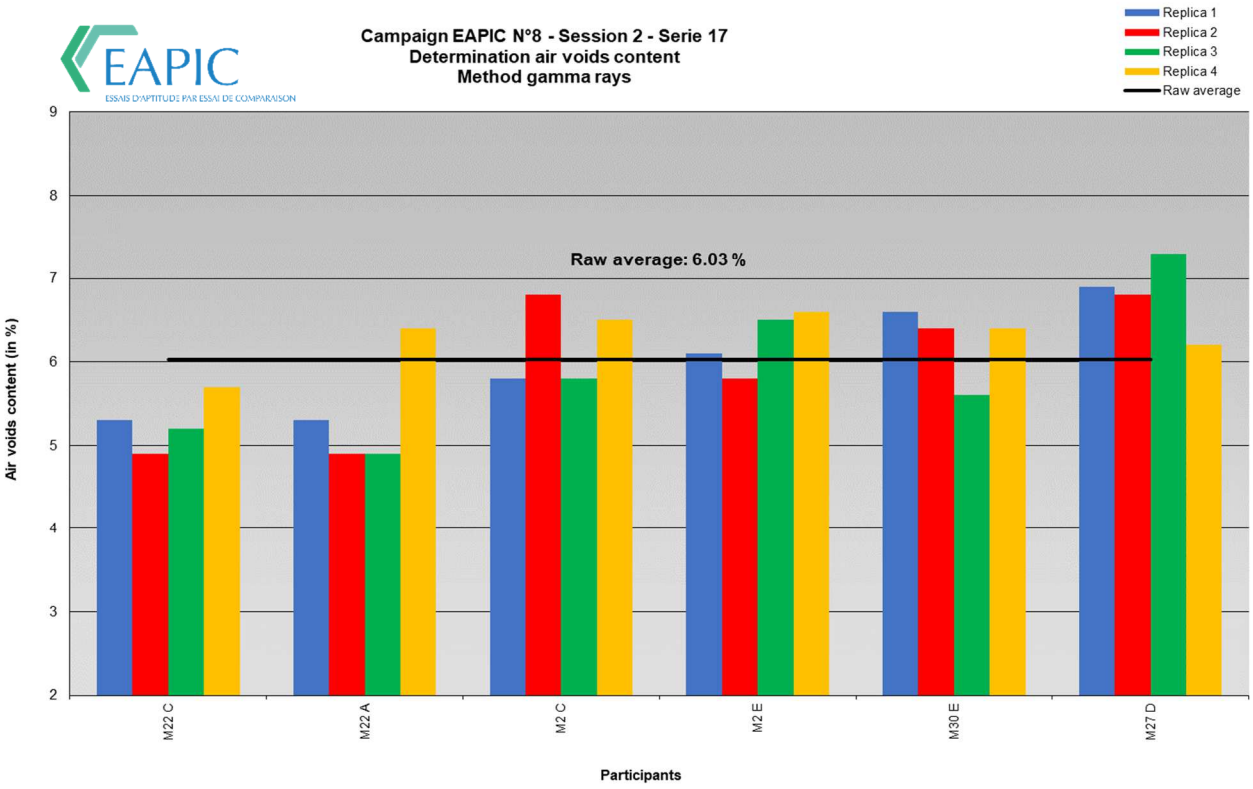
Determination of air voids content by gamma rays

Graphical representation

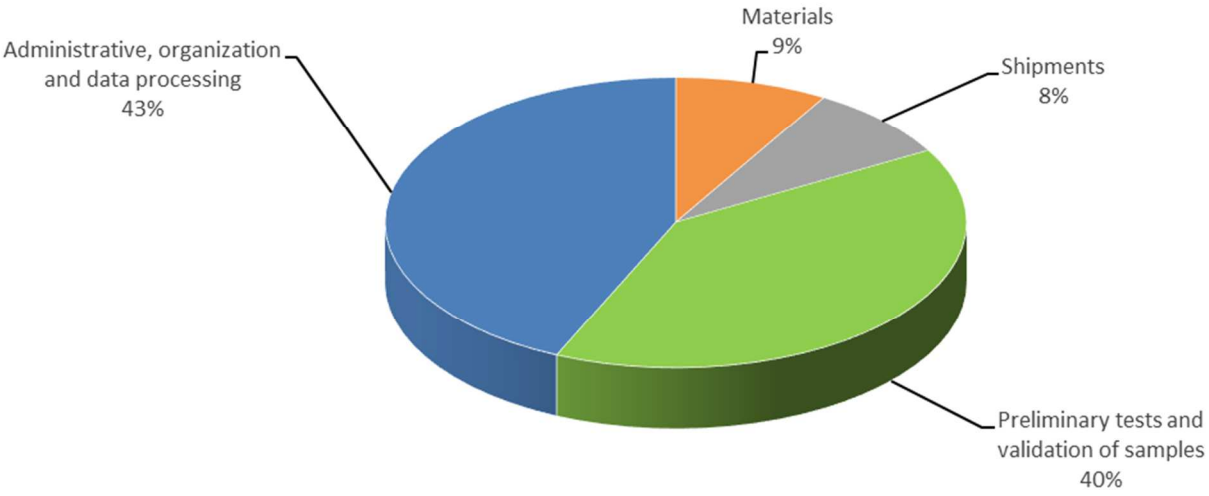
Number of laboratories: 6
Average: 6.03 %

Raw data





Balance sheet



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Tracer ensemble les voies durables de la mobilité