

## RESULTS PRESENTATION REPORT

N° 2.2.16

About the test

### DETERMINATION OF AIR VOID CONTENTS

According to standard NF EN 12697-31 August 2007



10 October 2017 par G. PIOT – Executive Cell of EAPIC  
CEREMA - Dter IDF - Laboratoire Eco Matériaux  
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77487 PROVINS Cedex

## *Preamble*

In EAPIC's history, session n°2.1, performed in 2005, impressed everyone's mind. Ours anyway: the results scattering was higher than void ranges defined to specify one type of bituminous mix or another. Fear and Trembling !

A study had then been launched by French LCPC. A subsample of laboratories that had participated in aptitude test 2.1 had then been defined. A new metrological device at the time, had been operated to measure internal angles of all used testing equipments. Let us remind for young people that, at that period, there was less diversity in the type of GC [gyratory compactors]. It was concluded that the internal angle value had absolutely to be better controlled to get a chance to improve test precision.

About 10 years later, the internal angle measurement as a way of checking metrological compliance of gyratory compactors has become a habit and a part of the standard. That's why we convinced ourselves that a new experiment had to be tried. Especially in view of the fact that several types of equipment are now existing. These different types are part of the machines population which participated in the present inter-comparison test. An analysis by type is proposed in the annex of this report. You will notice by reading the graphics illustrating this report that the situation is substantially improved. It is true that conventionally true value of MVRe [mix maximum density] had been given to you. This removed one source of dispersion.

In addition to this experiment, metrological data entrusted to us about your machines have been anonymously processed. They will be sent to experts of French mirror commission for tests standards. The aim is to improve the standard.

You will have noticed that « Chairman's message » has become « Preamble ». The reason is that the Quality system review, aimed to move to a potential accreditation of COQC's activities, led to rename the function of the Inter-Comparison Aptitude Tests [EAPIC] specialized group.

Kind regards,

For EAPIC,



Jean-Eric POIRIER

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## Session Organization and Data Collection

Homogenized batches, comprised of three grain fractions, are supplied to participating laboratories. These have to determine the air void content according to NF EN 12697-31 August 2007.

The selected formula is described as follows:

Fines	2,7 %
0/2	32 %
2/6	19 %
6/10	41 %
Bitumen	5,3 %

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: 2,615 Mg/m<sup>3</sup>

The campaign unfolded as follows:

- Shipping of samples in January / February 2017.
- Results delivery from participating laboratories at latest 15th of March 2017, except for those located outside continental France whose delivery date was postponed because of shipping time (delivery date at latest 20th of May 2017).  
The number of participants to this session is 38 laboratories, with 50 registered GC machines.  
The report provides 45 GC results, 2 laboratories having been unable to give results for physical and/or technical damage and 1 laboratory with 3 GC unable to send its results on time.  
All requests for further delay sent to EAPIC executive unit have been accepted with ultimate deadline 20th of May 2017.  
(For information: 58% results have been sent within the initial deadline of 15th March 2017.)
- Publication of the result report in August 2017.

## *Preparation and shipping of samples*

### *Materials*

Each laboratory received one pallet per GC machine registered in the test campaign.

The pallet is composed of the following fractions:

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

### *Preparation*

To carry out this campaign, 300 bags of aggregates were prepared and bagged by the Cerema Ouest – Department Laboratory of Angers:

- 100 bags of 25 kg of 6/10 ;
- 100 bags of 25 kg of 2/6 ;
- 100 bags of 25 kg of 0/2.



*Bagging workshop*



*Pallets storage*

### ***Shipping of materials***

The materials were sent by the Cerema Ouest - Department Laboratory of Angers. The set of bags and bitumen pots required for the campaign was set up on pallet before shipment.



## Verification of aggregates homogeneity by Cerema Ouest - Laboratory department of Angers (support laboratory)

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 " are:

- The pre-dried aggregates density determined according to the standard NF EN 1097-6 annex A, of January 2014 ;
- 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.

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

	MVR 0/2	MVR 2/6	MVR 6/10	penetration	TBA
<b>Average</b>	2,830	2,874	2,870	39,2	52,0
<b>Origin of r and R</b>	EAPIC 5th Campaign	NF EN 1097-6	NF EN 1097-6	NF EN 1426	NF EN 1427
<b>r</b>	0,026	0,019	0,019	2	1,0
<b>R</b>	0,008	0,042	0,042	3	2,0
<b><math>0,3 \times \hat{\sigma}</math></b>	0,0078	0,0043	0,0043	0,30	0,20
<b>Standard deviation between samples <math>S_s</math></b>	0,0006	0,0029	0,0020	0,20	0,14
<b>Validation <math>S_s \leq 0,3 \times \hat{\sigma}</math></b>	condition fulfilled	condition fulfilled	condition fulfilled	condition fulfilled	condition fulfilled

Criteria is fulfilled for each test.

Therefore, it can be concluded that samples are sufficiently homogeneous

## *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:

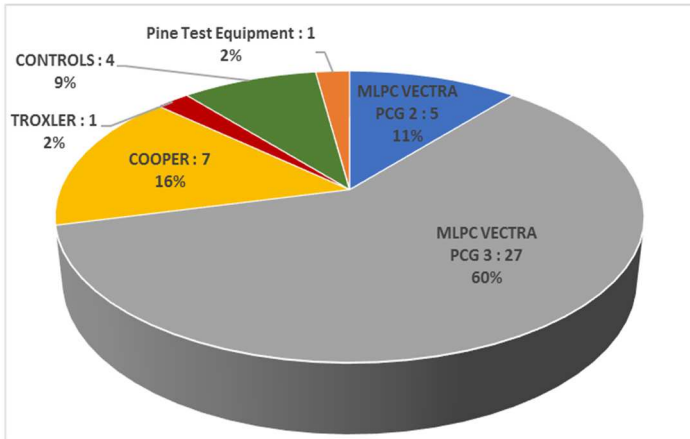
- Cochran test (intra-laboratory variability): detection of variance outliers, in the statistical sense of the results in a laboratory.
- Simple Grubbs test or possibly double (between laboratories variability): detection of averages outliers, among the population of laboratories.

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

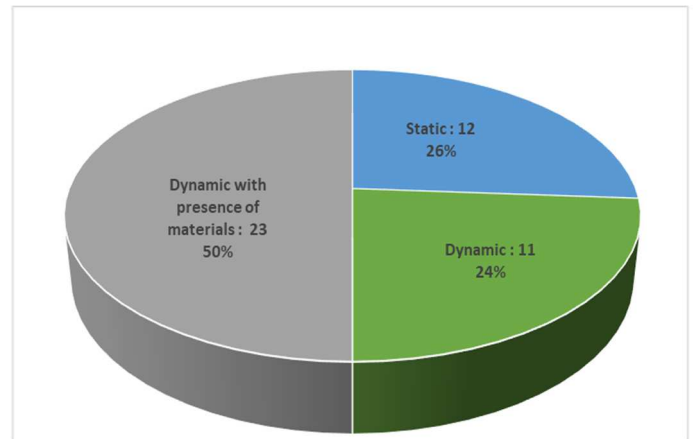


# Materials and metrology

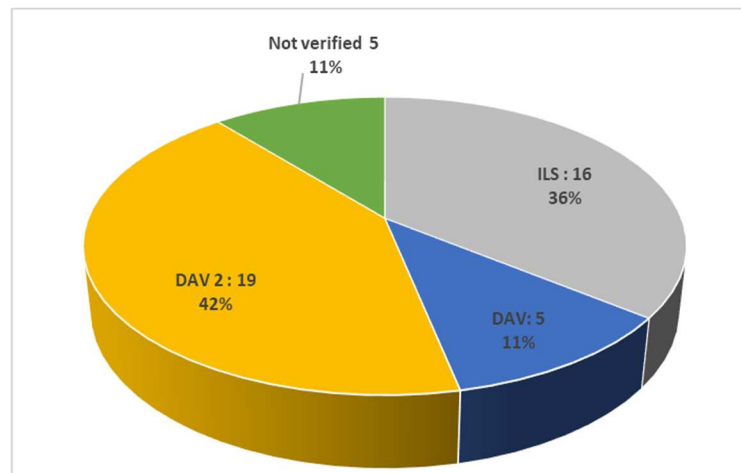
## Inter-Comparison Aptitude Test (EAPIC) 2<sup>nd</sup> Campaign – 2<sup>nd</sup> Session – Série n° 16



**Brand**



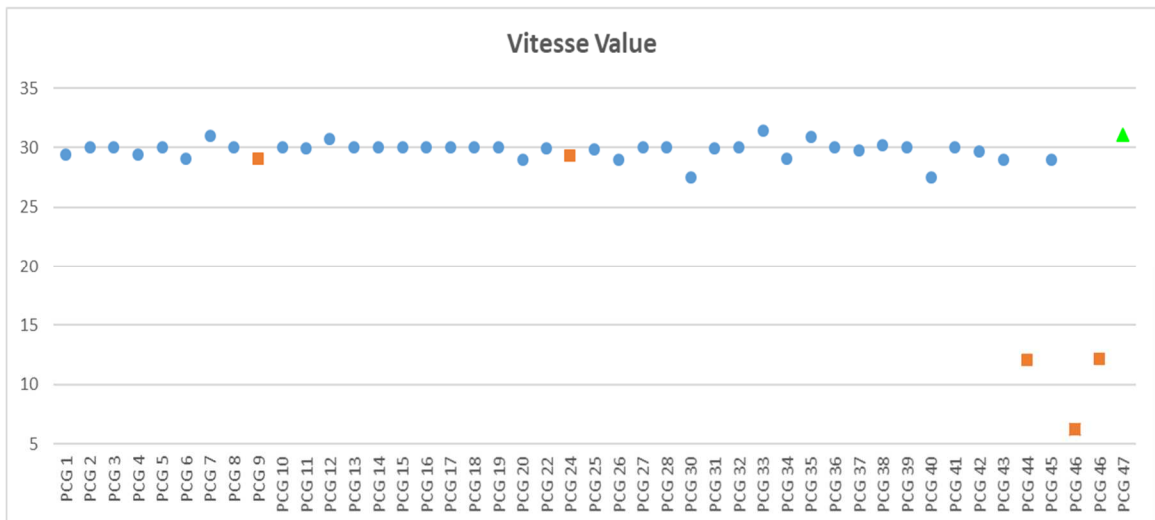
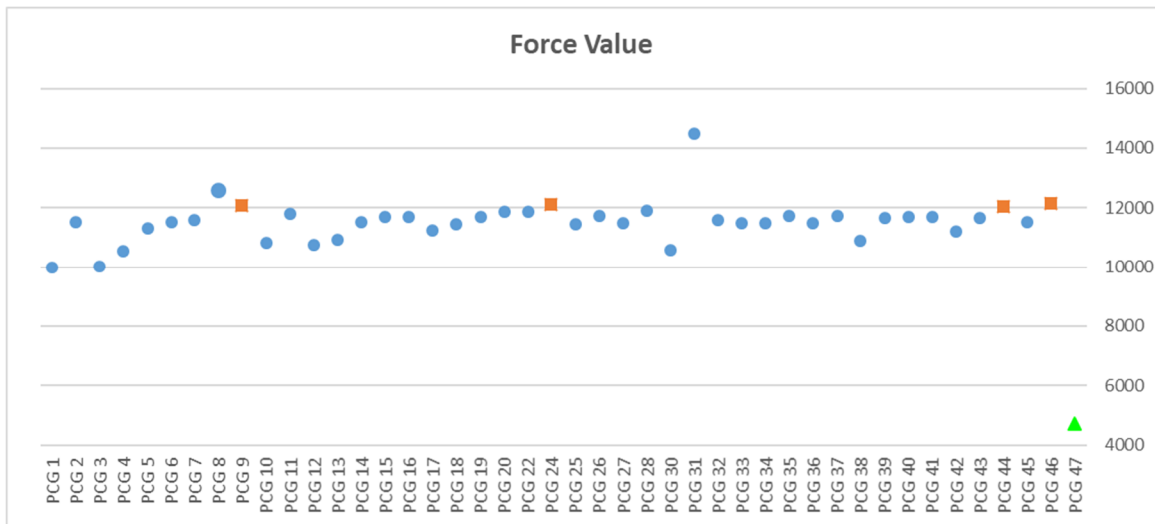
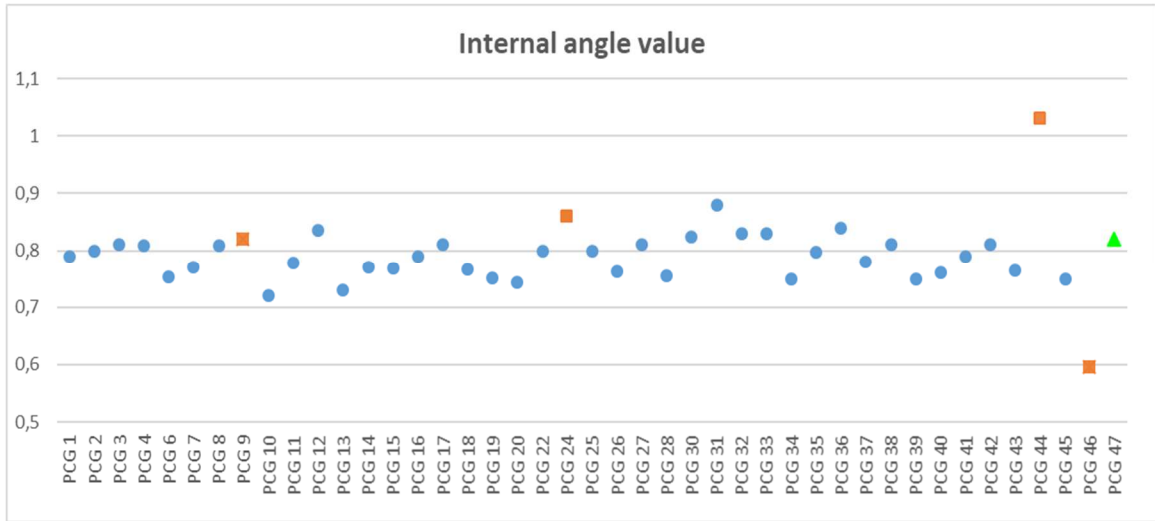
**Verification of the force**



**Verification of the internal angle**

**Legend**

- Molds diameter 100 mm ▲
- Molds diameter 150 mm ●
- Molds diameter 160 mm ■



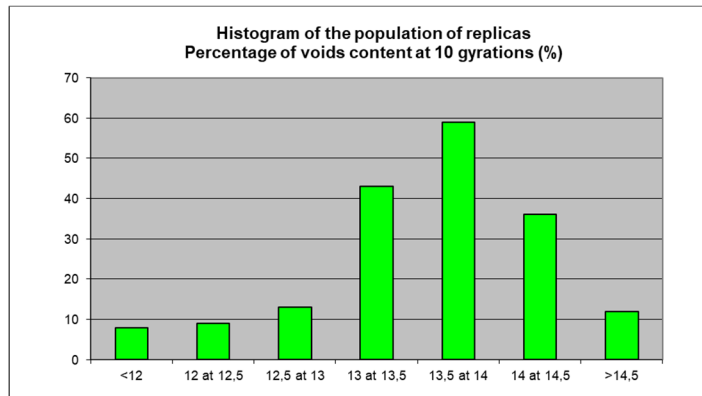
# Determination of air void contents (NF EN 12697-31)

## Values at 10 gyrations

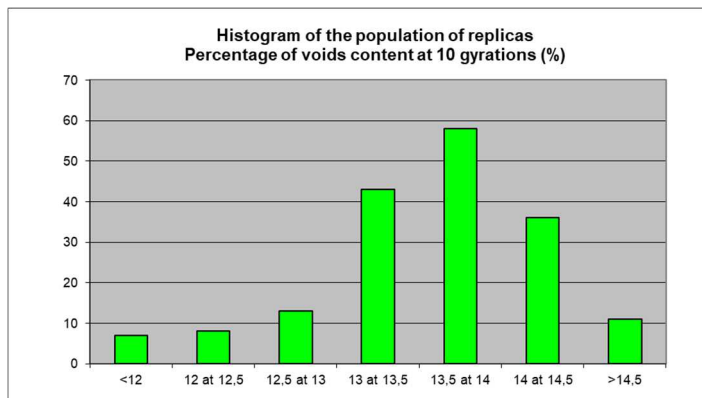
### Graphical representations

		Raw data	Results rejected by statistical tests	Corrected data	Values from the accuracy experiment of Dec. 1996	Results of campaign 2.1
In %	Number of results taken in account	45	Cochran PCG 1	44		
	Average m	13,62		13,63		
	Standard deviation repeatability	0,372		0,313		
	Repeatability r	1,043		0,876	r = 0,89	
	Standard deviation Reproducibility	0,746		0,728	R = 1,53	
	Reproducibility R	2,088		2,040		

Raw data

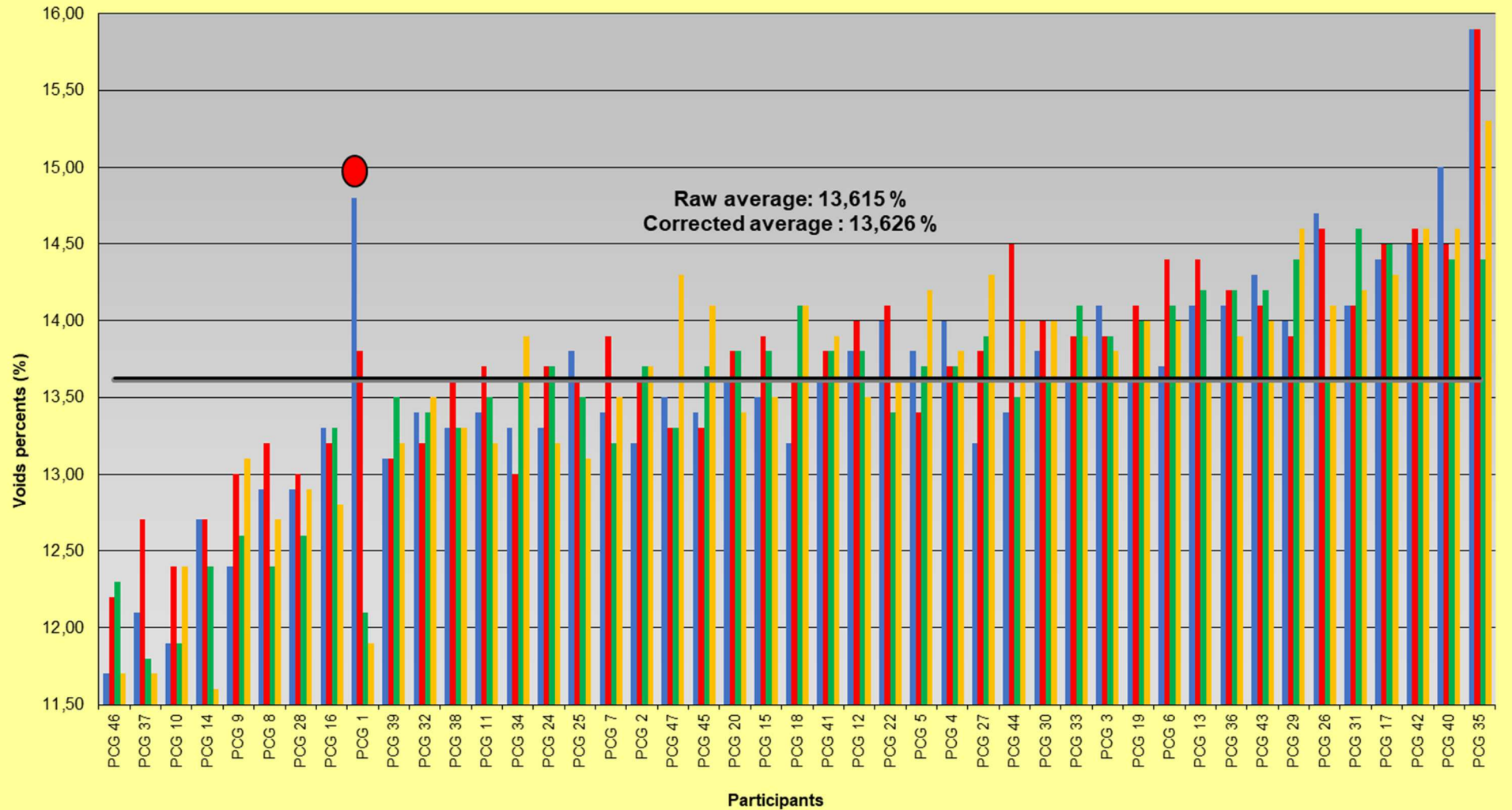


Corrected data



Raw data validated by XLSTAT software:  
m = 13,615

**Campaign EAPIC N°2 - Session 2 - Serie 16**  
**Determination of air void contents**  
**Values at 10 gyrations**  
**(according to NF EN 12697-31)**



*Average difference on the data raw at 10 gyrations*

**Difference less than 1 standard deviation**

Code results	Average deviation	Z-Score
PCG 45	0,01	0,03
PCG 47	0,01	0,04
PCG 20	0,04	0,09
PCG 15	0,06	0,16
PCG 2	0,06	0,17
PCG 25	0,11	0,31
PCG 7	0,11	0,31
PCG 18	0,14	0,36
PCG 24	0,14	0,38
PCG 41	0,16	0,43
PCG 22	0,16	0,43
PCG 12	0,16	0,43
PCG 5	0,16	0,43
PCG 11	0,16	0,44
PCG 34	0,16	0,44
PCG 4	0,19	0,50
PCG 27	0,19	0,50
PCG 44	0,24	0,63
PCG 30	0,24	0,63
PCG 32	0,24	0,65
PCG 38	0,24	0,65
PCG 33	0,26	0,70
PCG 3	0,31	0,83
PCG 19	0,31	0,83

**Difference less than 2 standard deviation**

Code results	Average deviation	Z-Score
PCG 39	0,39	1,05
PCG 6	0,44	1,17
PCG 13	0,46	1,24
PCG 1	0,46	1,25
PCG 16	0,46	1,25
PCG 36	0,49	1,31
PCG 26	0,49	1,31
PCG 43	0,54	1,44
PCG 29	0,61	1,64
PCG 31	0,64	1,71

**Difference more than 2 standard deviations**

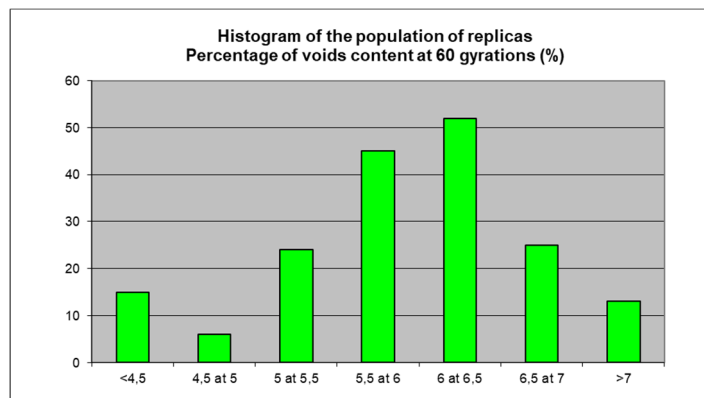
Code results	Average deviation	Z-Score
PCG 28	0,76	2,06
PCG 17	0,81	2,18
PCG 8	0,81	2,19
PCG 9	0,84	2,26
PCG 42	0,94	2,52
PCG 40	1,01	2,72
PCG 14	1,26	3,41
PCG 10	1,46	3,94
PCG 37	1,54	4,15
PCG 46	1,64	4,41
PCG 35	1,76	4,74

## Values at 60 gyrations

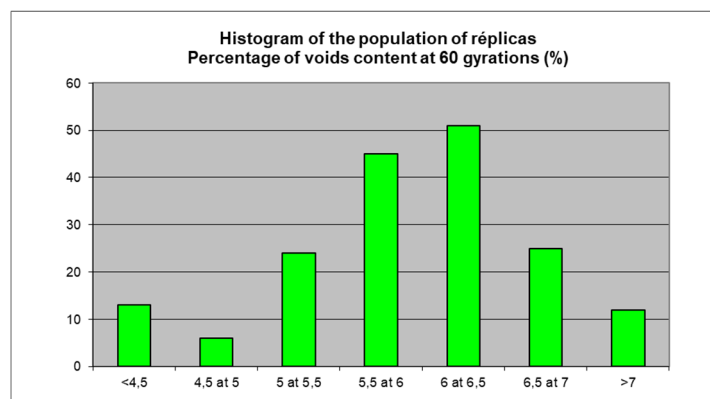
### Graphical representations

		Raw data	Results rejected by statistical tests	Corrected data	Standard NF EN 12697-31	Results of campaign 2.1
In %	Number of results taken in account	45	Cochran PCG1	44	r = 0,950	r = 0,90
	Average m	5,95		5,96		
	Standard deviation repeatability	0,362		0,307		
	Repeatability r	1,014		0,861	R = 1,384	R = 3,92
	Standard deviation Reproducibility	0,849		0,839		
	Reproducibility R	2,378		2,350		

Raw data











Corrected data

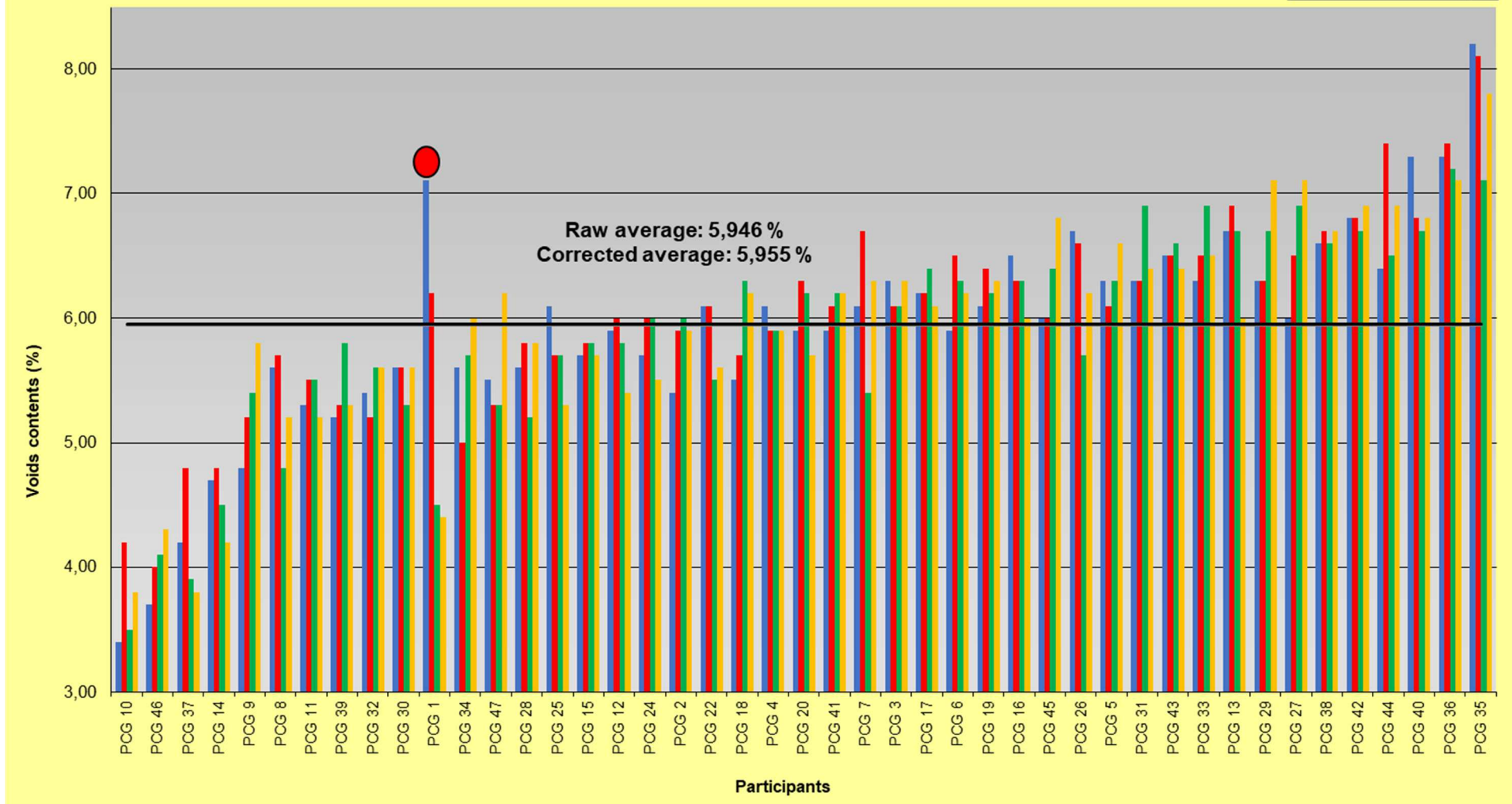


Raw data validated by XLSTAT software:  
m = 5,946

**Campaign EAPIC N°2 - Session 2 - Serie 16**  
**Determination of air void contents**  
**Values at 60 gyrations**  
**(According to NF EN 12697-31)**

 Cochran Test  
 Grubbs Test

 Replica 1  
 Replica 2  
 Replica 3  
 Replica 4  
 Raw average  
 Corrected average



*Average difference on the raw data at 60 gyrations*

**Difference less than 1 standard deviation**

Code results	Average deviation	Z-Score
PCG 4	0,00	0,01
PCG 18	0,02	0,05
PCG 20	0,08	0,19
PCG 22	0,12	0,28
PCG 2	0,15	0,34
PCG 24	0,15	0,34
PCG 41	0,15	0,36
PCG 12	0,17	0,40
PCG 7	0,18	0,42
PCG 15	0,20	0,46
PCG 26	0,22	0,52
PCG 25	0,25	0,58
PCG 3	0,25	0,60
PCG 6	0,28	0,66
PCG 17	0,28	0,66
PCG 19	0,30	0,72
PCG 16	0,33	0,78
PCG 28	0,35	0,81
PCG 45	0,35	0,84
PCG 47	0,37	0,87
PCG 34	0,37	0,87
PCG 5	0,38	0,89
PCG 1	0,40	0,93
PCG 30	0,42	0,99

**Difference less than 2 standard deviation**

Code results	Average deviation	Z-Score
PCG 32	0,50	1,17
PCG 31	0,53	1,25
PCG 39	0,55	1,29
PCG 43	0,55	1,31
PCG 11	0,57	1,35
PCG 33	0,60	1,42
PCG 8	0,62	1,46
PCG 13	0,63	1,48
PCG 9	0,65	1,52
PCG 29	0,65	1,54
PCG 27	0,68	1,60
PCG 38	0,70	1,66

**Difference more than 2 standard deviations**

Code results	Average deviation	Z-Score
PCG 44	0,85	2,01
PCG 42	0,85	2,01
PCG 40	0,95	2,25
PCG 36	1,30	3,08
PCG 14	1,40	3,29
PCG 37	1,77	4,17
PCG 35	1,85	4,37
PCG 46	1,92	4,53
PCG 10	2,22	5,24

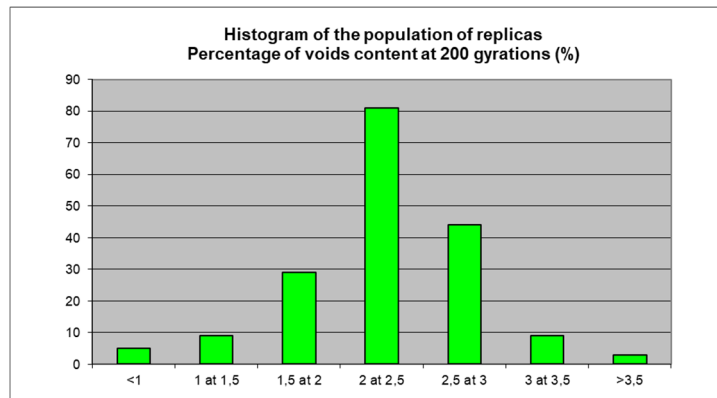


## Values at 200 gyrations

### Graphical representations



		Raw data	Results rejected by statistical tests	Values from the accuracy experiment of Dec. 1996	Results of campaign 2.1
In %	Number of results taken in account	45	RAS	r = 1,04	r = 1,08
	Average m	2,304			
	Standard deviation repeatability	0,216			
	Repeatability r	0,606			
	Standard deviation Reproducibility	0,541			
	Reproducibility R	1,514			






Raw data

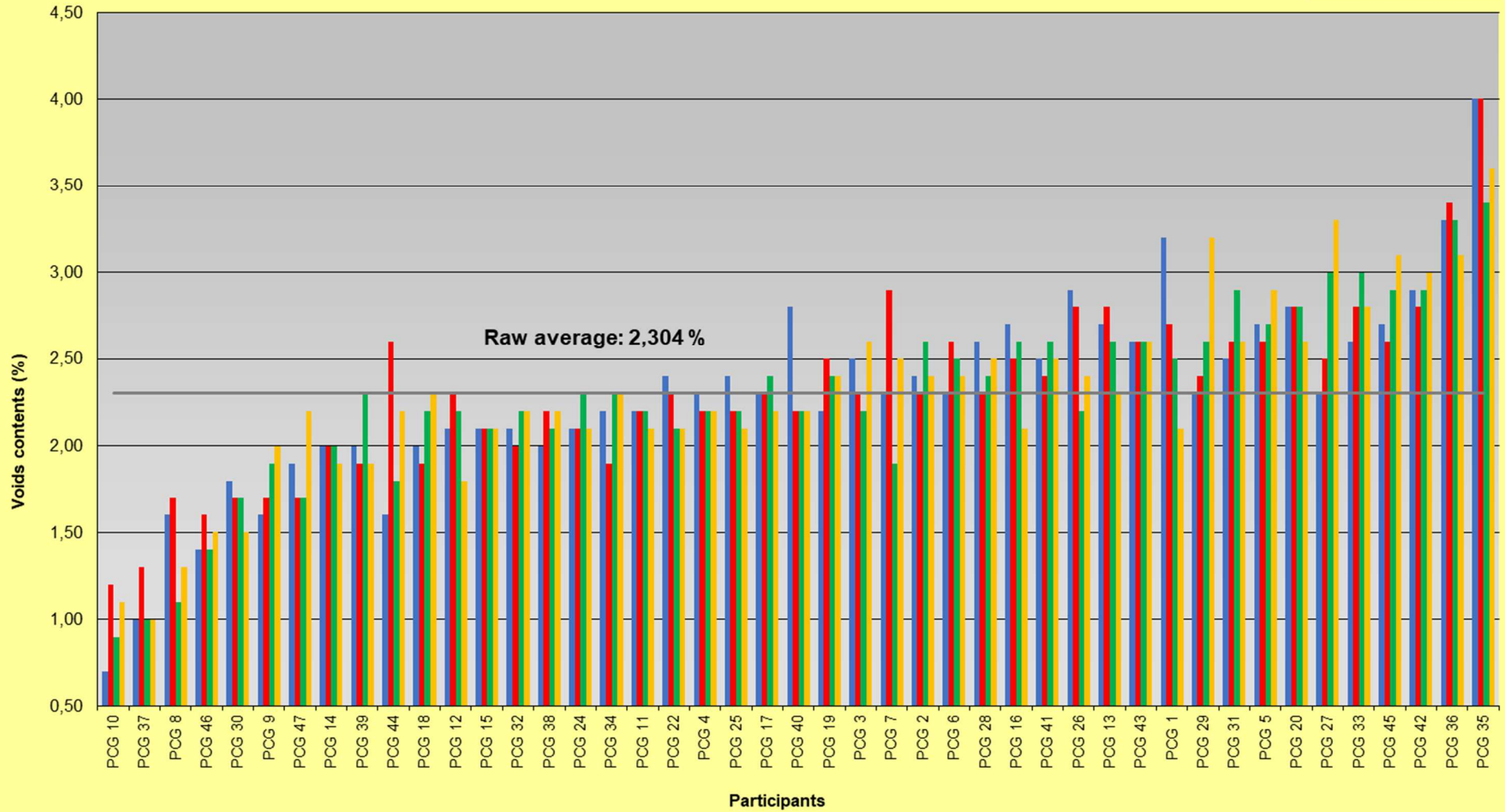


Raw data validated by XLSTAT software:  
m = 2,3039

**Campaign EAPIC N°2 - Session 2 - Serie 16**  
**Determination of air void contents**  
**Values at 200 gyrations**  
**(According to NF EN 12697-31)**

 Cochran Test  
 Grubbs Test

 Replica 1  
 Replica 2  
 Replica 3  
 Replica 4  
 Raw average



*Average difference on the raw data at 200 gyrations*

**Difference less than 1 standard deviation**

Code results	Average deviation	Z-Score
PCG 17	0,00	0,01
PCG 40	0,05	0,17
PCG 19	0,07	0,26
PCG 4	0,08	0,29
PCG 25	0,08	0,29
PCG 22	0,08	0,29
PCG 3	0,10	0,36
PCG 7	0,10	0,36
PCG 2	0,12	0,45
PCG 11	0,13	0,48
PCG 34	0,13	0,48
PCG 6	0,15	0,54
PCG 28	0,15	0,54
PCG 24	0,15	0,57
PCG 26	0,16	0,60
PCG 16	0,17	0,63
PCG 32	0,18	0,66
PCG 38	0,18	0,66
PCG 41	0,20	0,73
PCG 15	0,20	0,76
PCG 12	0,20	0,76
PCG 18	0,20	0,76
PCG 44	0,25	0,94

**Difference less than 2 standard deviations**

Code results	Average deviation	Z-Score
PCG 39	0,28	1,03
PCG 13	0,30	1,10
PCG 43	0,30	1,10
PCG 1	0,32	1,19
PCG 29	0,32	1,19
PCG 14	0,33	1,22
PCG 31	0,35	1,28
PCG 5	0,42	1,56
PCG 47	0,43	1,59
PCG 20	0,45	1,65
PCG 27	0,47	1,75
PCG 33	0,50	1,84
PCG 9	0,50	1,87
PCG 45	0,52	1,93

**Difference more than 2 standard deviations**

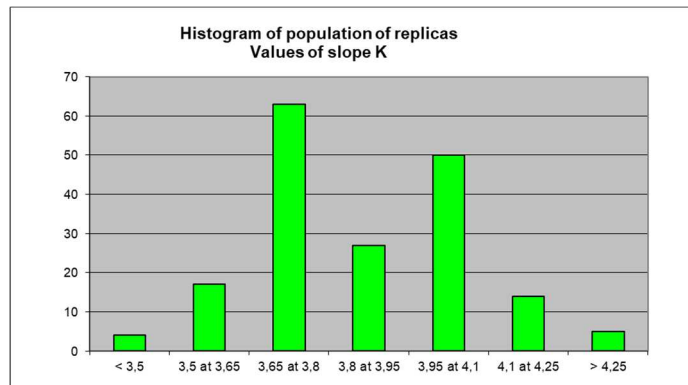
Code results	Average deviation	Z-Score
PCG 42	0,60	2,21
PCG 30	0,63	2,33
PCG 46	0,83	3,07
PCG 8	0,88	3,26
PCG 36	0,97	3,60
PCG 37	1,23	4,55
PCG 10	1,33	4,92
PCG 35	1,45	5,36

## Détermination of the slope K

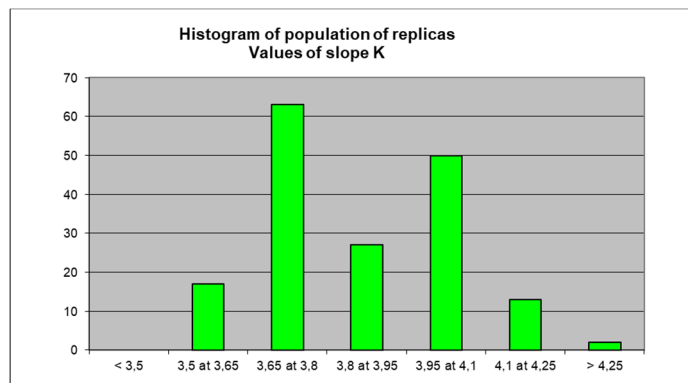
### Graphical representations

	Raw data	Results rejected by statistical tests	Corrected data
Number of results taken in account	45	Cochran PCG 10 PCG 14	43
Average m	3,867		3,869
Standard deviation repeatability	0,075		0,062
Repeatability r	0,209		0,174
Standard deviation Reproducibility	0,214		0,179
Reproducibility R	0,599		0,500

Raw data

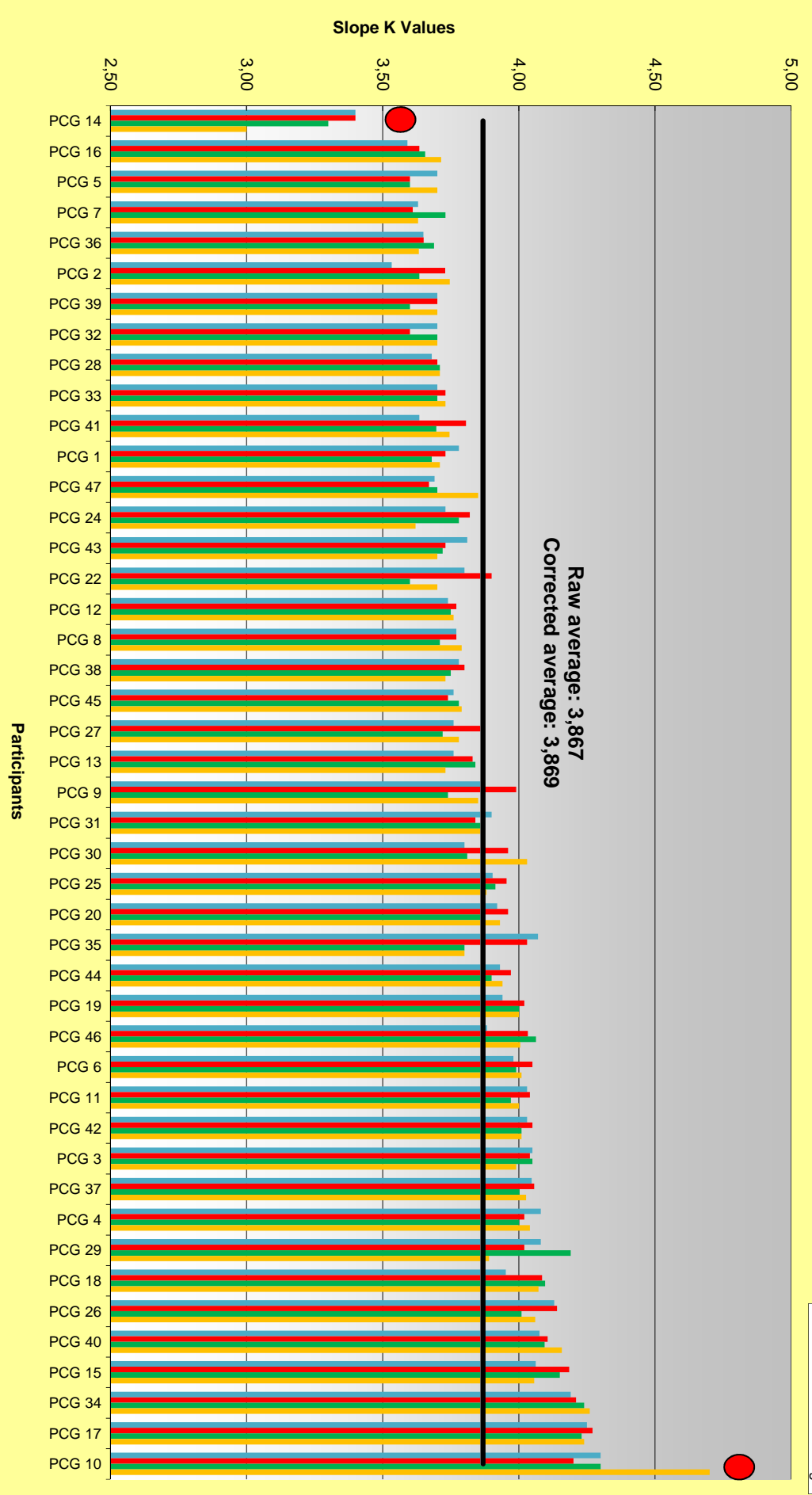


Corrected data



Raw data validated by XLSTAT software:  
m = 3,867

**Campaign EAPIC N°2 - Session 2 - Serie 16**  
**Determination of slope K**



● Cochran Test  
 ■ Grubbs Test

Replica 1  
 Replica 2  
 Replica 3  
 Replica 4  
 Raw average  
 Corrected average

*Average difference on the raw data*

**Difference less than 1 standard deviation**

Code results	Average deviation	Z-Score
PCG 31	0,00	0,02
PCG 9	0,01	0,07
PCG 30	0,03	0,31
PCG 25	0,05	0,43
PCG 20	0,05	0,49
PCG 35	0,06	0,54
PCG 44	0,07	0,64
PCG 13	0,08	0,72
PCG 27	0,09	0,81
PCG 45	0,10	0,93
PCG 38	0,10	0,96

**Difference less than 2 standard deviations**

Code results	Average deviation	Z-Score
PCG 8	0,11	1,00
PCG 12	0,11	1,05
PCG 22	0,12	1,10
PCG 19	0,12	1,15
PCG 43	0,13	1,19
PCG 46	0,13	1,21
PCG 24	0,13	1,21
PCG 47	0,14	1,31
PCG 6	0,14	1,31
PCG 1	0,14	1,33
PCG 11	0,14	1,34
PCG 41	0,15	1,37
PCG 33	0,15	1,42
PCG 42	0,16	1,48
PCG 3	0,17	1,55
PCG 37	0,17	1,55
PCG 28	0,17	1,56
PCG 4	0,17	1,57
PCG 29	0,18	1,66
PCG 18	0,18	1,72
PCG 39	0,19	1,80
PCG 32	0,19	1,80
PCG 2	0,21	1,93
PCG 36	0,21	1,99

**Difference more than 2 standard deviations**

Code results	Average deviation	Z-Score
PCG 5	0,22	2,03
PCG 7	0,22	2,03
PCG 26	0,22	2,04
PCG 16	0,22	2,04
PCG 40	0,24	2,26
PCG 15	0,25	2,30
PCG 34	0,36	3,35
PCG 17	0,38	3,56
PCG 10	0,51	4,75
PCG 14	0,59	5,54

## 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 Thierry KRETZ (assistant: Anaïs FERMINE)

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

### **EAPIC Specialised group**

**General Secretary:** POIRIER Jean-Eric

**Members:**

BADROUILLET Christophe  
FAUCON-DUMONT Stéphane  
PERIGOIS Stéphanie  
PIOT Géraldine  
SAUBOT Michel  
SOME Cyrille

### **EAPIC Executive Cell**

**Cerema IDF- Sourduin:** PIOT Géraldine & SOME Cyrille

### **EAPIC Support Laboratory**

**Cerema Ouest - Laboratory Department of Angers:** PERIGOIS Stéphanie

## *Annexes*

- Determination of air void contents (60 gyrations) according to type of machine
  - MLPC – VECTRA PCG 2
  - MLPC – VECTRA PCG 3
  - COOPER
  - Other types PCG (CONTROLS – TROXLER – Pine Test Equipment)
- Determination of air void contents at 60 gyrations: Results analysis by Mandel's test statistics
  - Inter-laboratory consistency h
  - Intra-laboratory consistency k



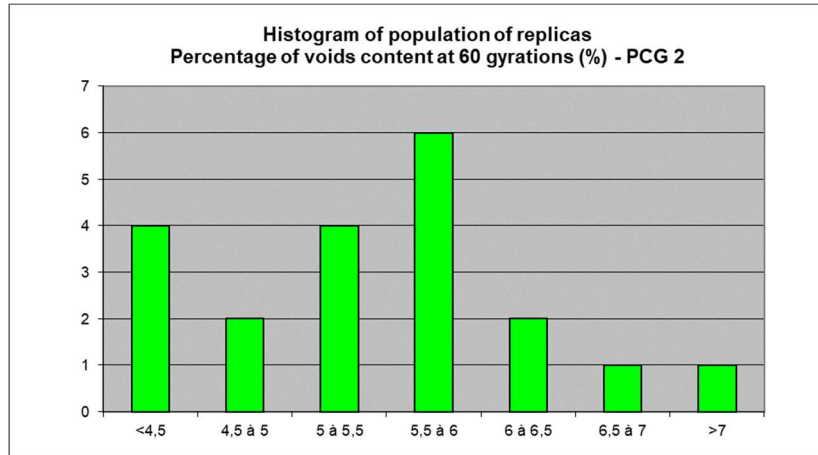
## Determination of air voids content at 60 gyrations

« MLPC VECTRA – PCG 2 »

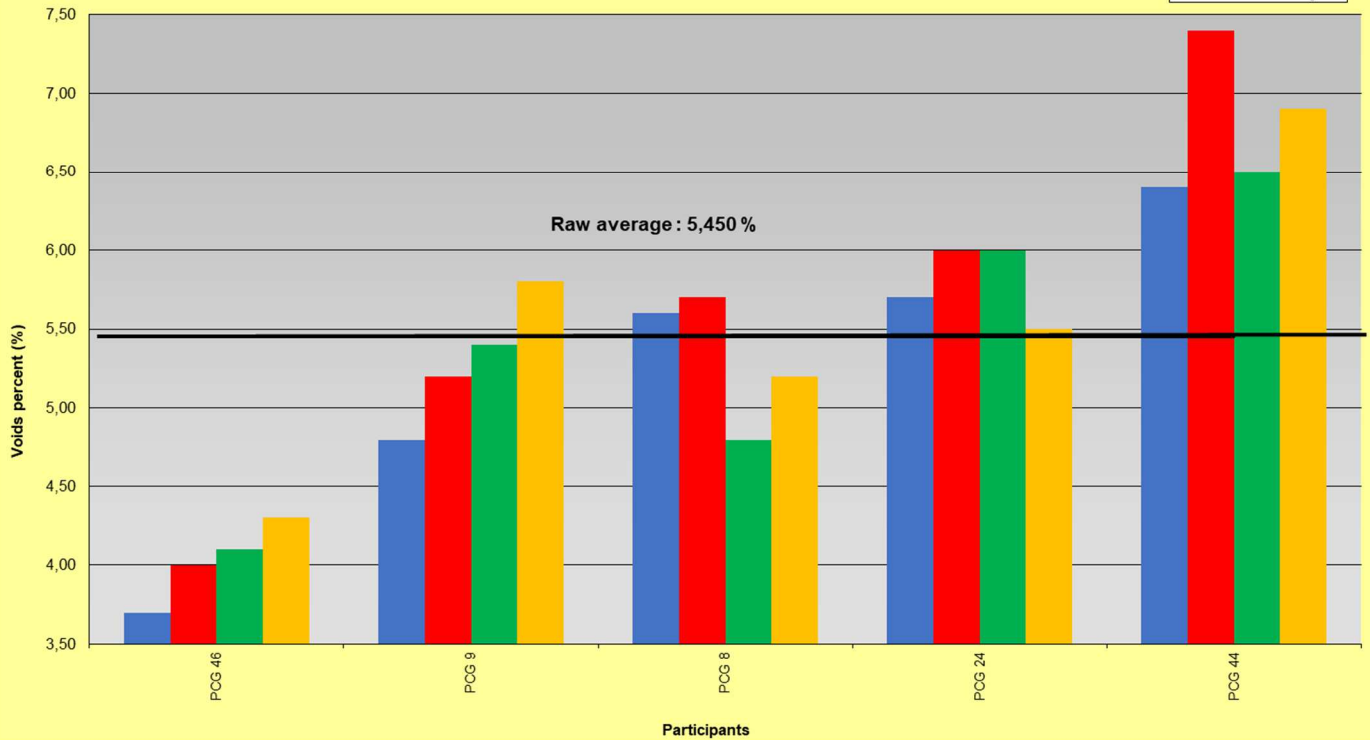
Number of results: 5

Average m: 5,45 %

### Raw data



**Campaign EAPIC N°2 - Session 2 - Serie 16**  
Determination of air voids contents  
Values at 60 gyrations - PCG 2  
(According to NF EN 12697-31)

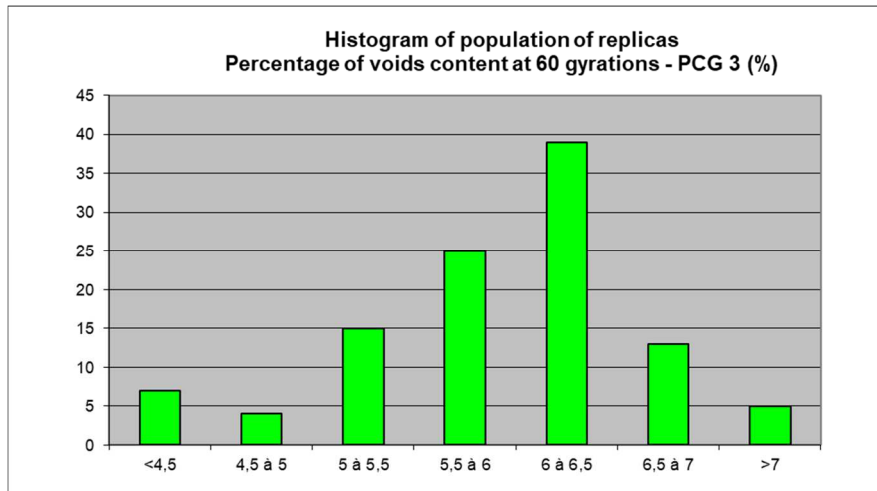


Machine « MLPC VECTRA – PCG 3 »

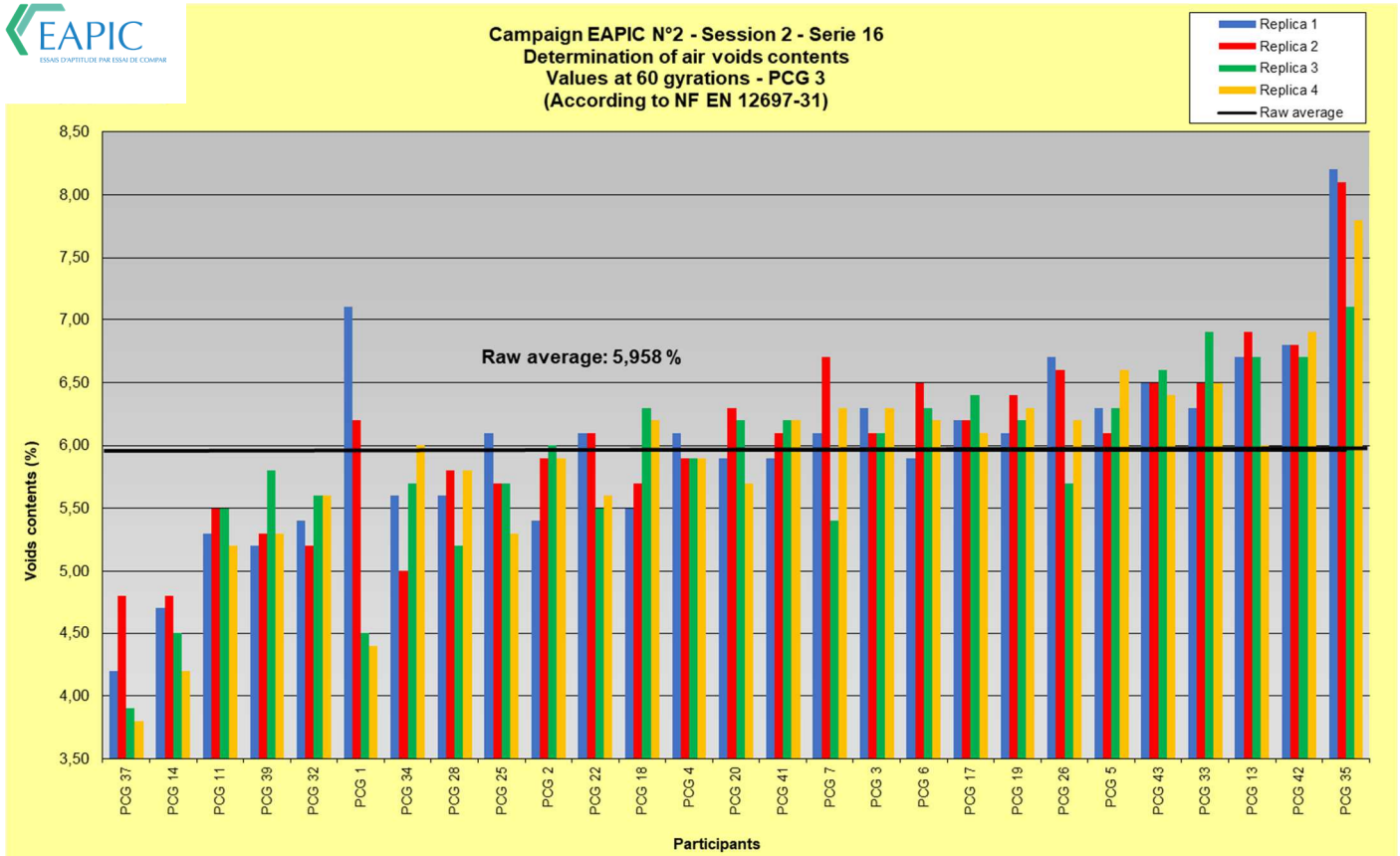
Number of results: 27

Average m: 5,958 %

Raw data



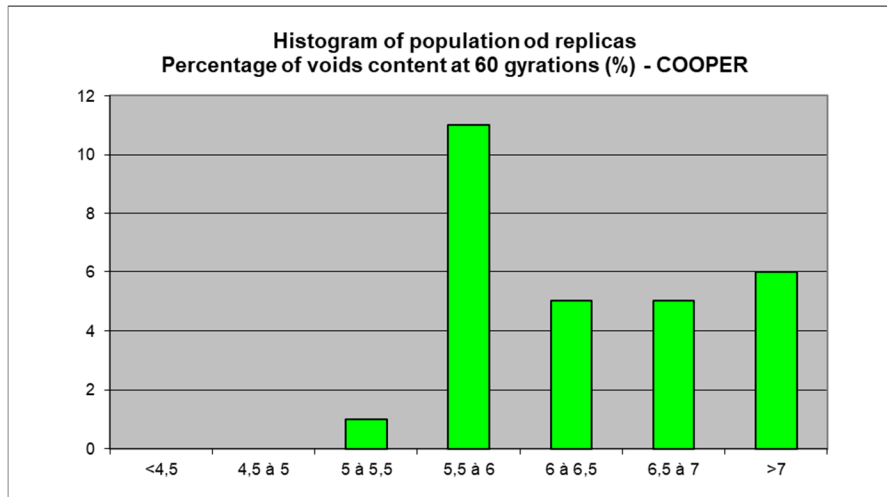
Campaign EAPIC N°2 - Session 2 - Serie 16  
Determination of air voids contents  
Values at 60 gyrations - PCG 3  
(According to NF EN 12697-31)



*Machine « Cooper »*

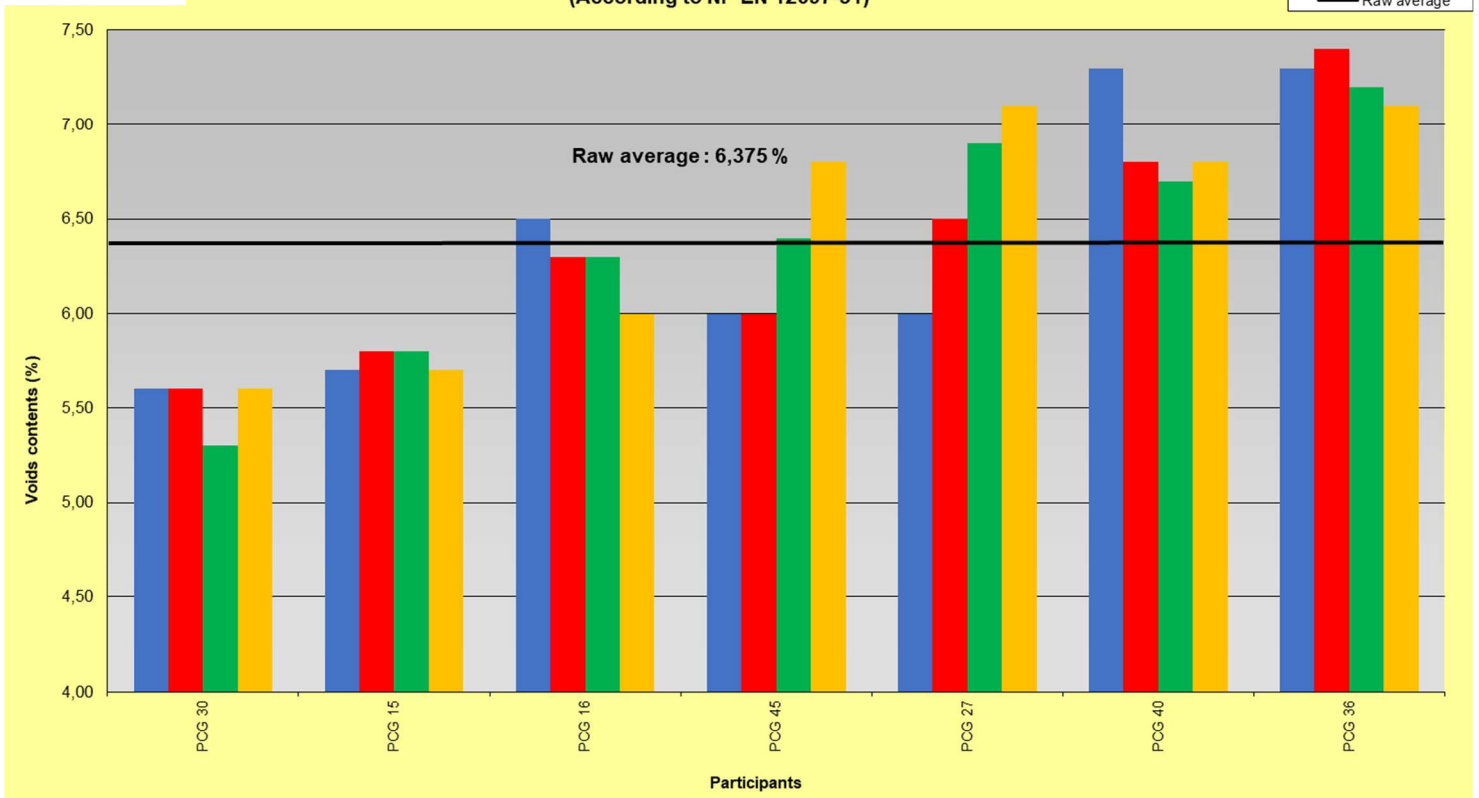
Number of results: 7  
Average m: 6,375 %

Raw data



**Campaign EAPIC N°2 - Session 2 - Serie 16**  
Determination of air voids contents  
Values at 60 gyrations - COOPER  
(According to NF EN 12697-31)

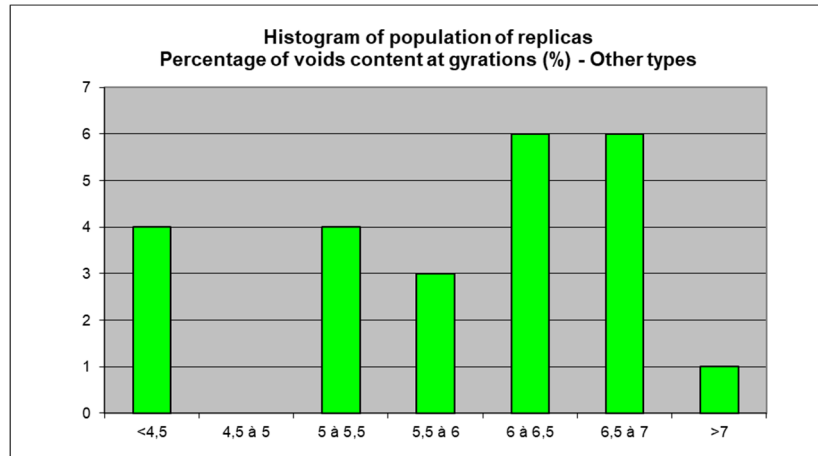
- Replica 1
- Replica 2
- Replica 3
- Replica 4
- Raw average



### Other types PCG (CONTROLS – TROXLER – Pine Test Equipment)

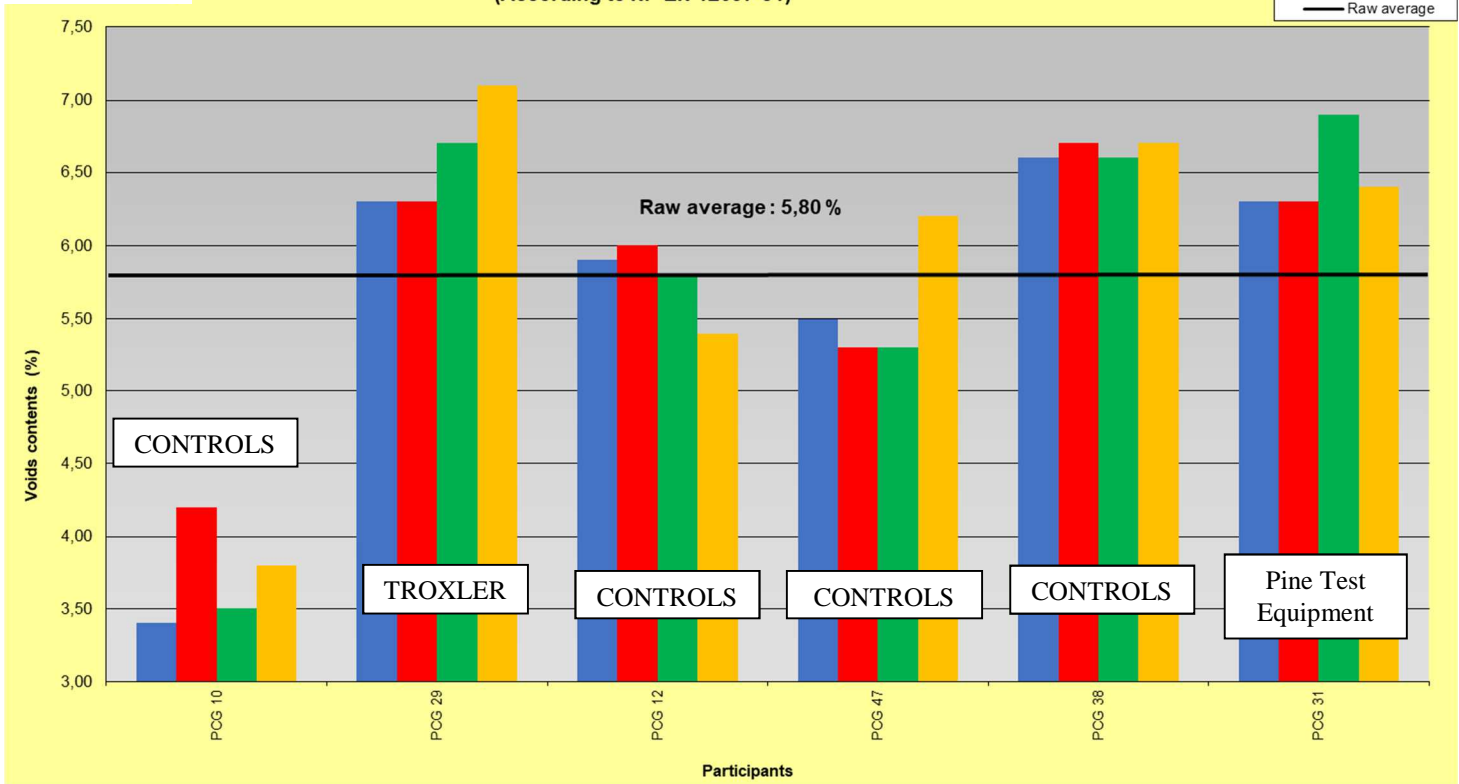
Number of results: 6  
Average m: 5,80 %

Raw data

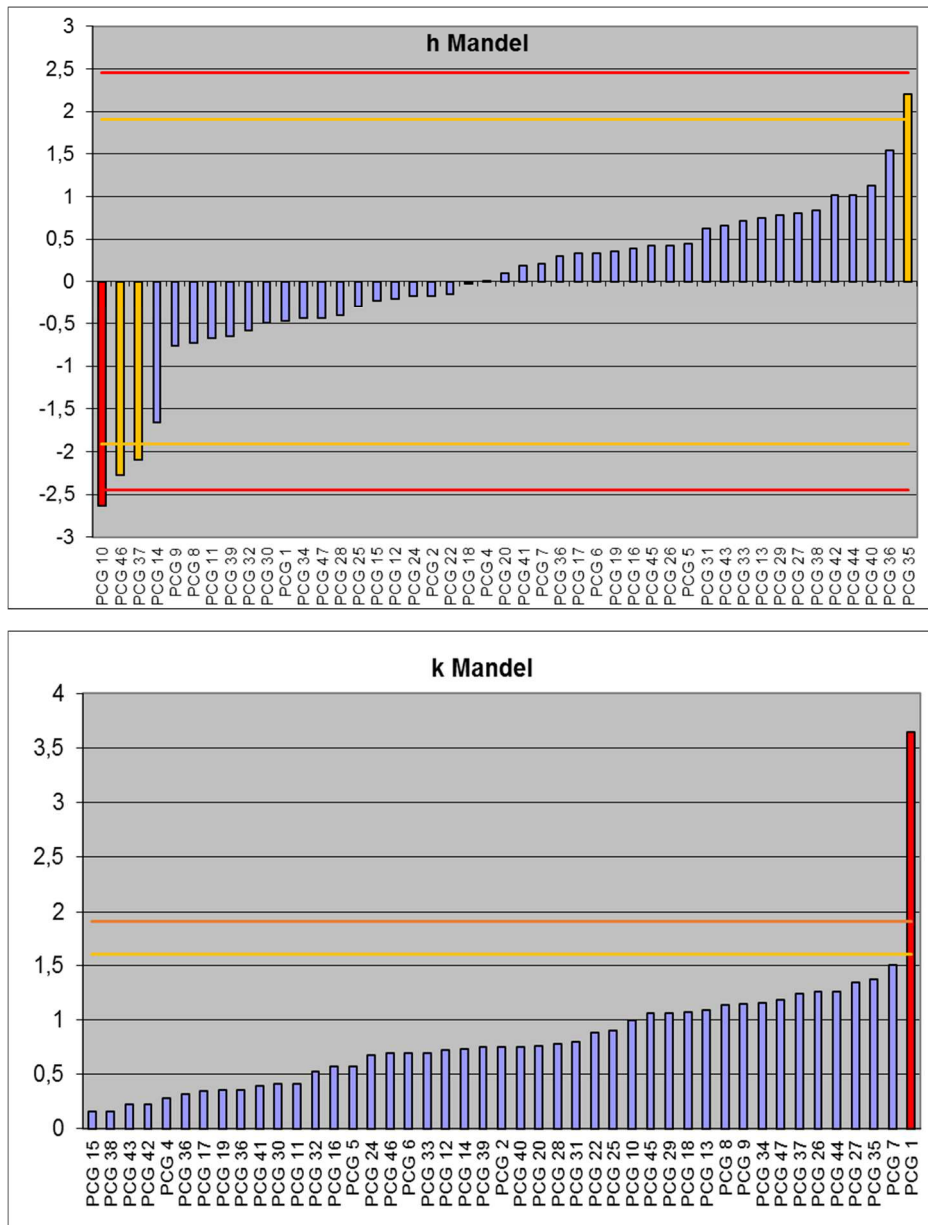


**Campaign EAPIC N°2 - Session 2 - Serie 16**  
Determination of air voids contents  
Values at 60 gyrations - Other types  
(According to NF EN 12697-31)

- Replica 1
- Replica 2
- Replica 3
- Replica 4
- Raw average



*Determination of air voids content at 60 gyrations - Mandel tests of consistency Inter-laboratories h and Intra-laboratory k – According to NF ISO 5725-2*



The consistency tests h and k lead to a graphical representations by histogram of the data and make it possible to detect visually inconsistent data.

- h: deviation from the average value
  - critical value 1% |2,45|
  - critical value 5% |1,91|
- k: deviation from the average standard deviation
  - critical value 1% |1,91|
  - critical value 5% |1,60|

In red are indicated values above the critical value 1 %  
 In yellow are indicated values above the critical value 5 % and below the critical value 1 %