Comité français pour les techniques routières

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Cftr - info

EAPIC
to prove the skills of the laboratories

Summary: *The quality process requires laboratories to prove their skills. Among the means available, inter-comparison experiments are tools which are commonly accepted. They were expected by the profession in the field of road materials, but issues related with volumes and the guarantee of sample homogeneity had delayed their application. The EAPIC (Essai d'Aptitude Par Inter-Comparaison, Ability experiment by Inter-Comparison) attempted to meet this expectation. This information note describes the EAPIC process, a specialized group of the Qualification-Certification sector committee of the CFTR. The EAPIC is in charge of organizing 'round robin tests' in the field of road materials (asphalt mixtures, materials treated with hydraulic binders, aggregates…). The test campaigns allow the laboratories to check the quality of their results, by comparison with 'real' values, when they exist, or average conventional values obtained by all laboratories. If applicable, statistical processing then shows values different from current results.*

Six sessions were organized, including determination of binder content and sieve size analysis of asphalt samples, repeated three times. Two hundred participations were recorded for all these sessions. The main results for repeatability, reproducibility, and if applicable difference against 'real' values, are presented.

1 - Introduction

Over the last few years, road-building professions have focused on better control over quality assurance.

The first step taken was to standardize tests in road construction and determine values for method repeatability and reproducibility. The second step resulted in the certification or accreditation of an increasing number of road construction laboratories.

The third consists in validating for specific tests the performance of laboratories, allowing them to demonstrate reliability to their clients and still improve result quality through corrective action. The method is based on inter-comparison ability tests (round robin tests). In addition to this objective, inter-comparison experiments improve test methods, confirm or improve reproducibility values, while appreciating the effectiveness of new methods.

In the field of road infrastructures, test campaigns of this type are organized regularly for hydro carbonated binders, cements and a few other products. In spite of strong demand, there are no inter-comparison experiments for materials treated with hydro carbon or hydraulic binders, probably because of the 'weight' of the organisation required.

The EAPIC specialized group attempted to resolve this shortcoming. It was implemented late 2001, and ran the first test campaign in 2002. Since then, EAPIC has led the following campaigns:

- Determination of soluble binder content of an asphalt mixture, as per the European standard and associated sieve size analysis,
- Measurement of maximum density of a bituminous mixture and gyratory shear press compaction,
- Los Angeles and Micro-Deval experiments,
- Methylen blue test and flow-test for fine aggregate.

This note describes the structure of the EAPIC and its operation, and presents a summary based on examples of results of the test campaigns led.

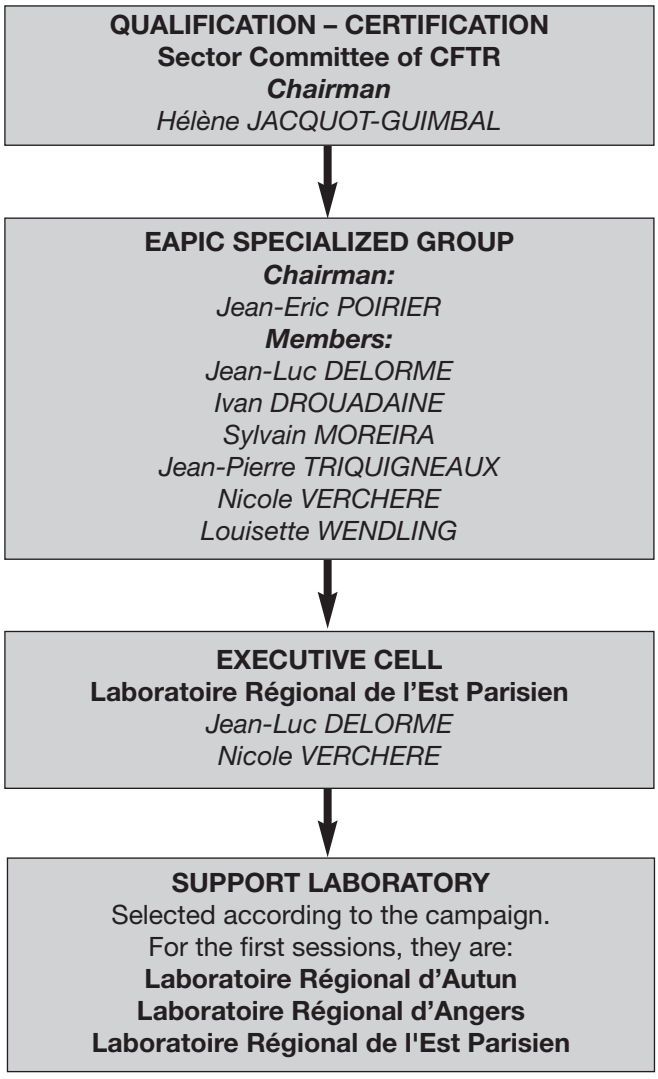
2 - The E.A.P.I.C specialized group

The 'Essais d'aptitudes par inter-comparaison' (inter-comparison collaborative experiments) specialized group reports to the Comité Sectoriel Qualification – Certification (Qualification-Certification Sector Committee) of the CFTR.

It is composed of technical experts and qualiticians from the public and private road construction community.

This group aims at organizing inter-comparison experiments on the ability of laboratories in the field of road construction: asphalt mixtures, materials treated with hydraulic binders.

The specialized group is the decision authority, supported by an executive cell in charge of organizing the experiments. The logistic support linked with the preparation and procurement in materials or test specimens required for leading the inter-comparison experiments is ensured by support laboratories under the liability of the executive cell.



The ***specialized group*** is entrusted with choosing and planning the test campaigns, supervising their performance, validating the organization procedures, selecting the support laboratories and approving and publishing the results.
It also approves the amounts set for financial contributions for participation in the tests and remuneration of the works.

The ***executive cell*** is in charge of organizing and coordinating the test campaigns. It monitors test performance and result processing. It issues calls for applicants, prepares procedures, ships objects to be tested, collects results, processes them and communicates them to each participant within the rules of confidentiality set.

Support laboratories, selected by the specialized group, store the stocks of materials, prepare them, ensure controlled manufacturing of proof specimens (replicas), demonstrate homogeneity, deliver to the participants in inter-comparison experiments. Action of support laboratories is led according to a Quality Assurance Plan and performance procedures with a content specified by the executive cell, and subject to approval by the specialized group. They must be holders of a COFRAC accreditation or a LABOROUTE approval or ISO 9000 certification.

3 - Test campaigns

The test methods concerned are standardized methods in the road construction field: coatings, materials treated with hydraulic binders or non treated, aggregates. Tests on bitumens are excluded (Bureau de Normalisation des Pétroles) for which other provisions have already been implemented.

The group gives priority to standardized experiments, for which repeatability ‘r’ and reproducibility ‘R’ are known, which are the most frequently used as a value specified, and which may give rise to comparison within a contractual frame.

EAPIC has defined the ‘campaign’ as being a type of test or a family of experiments. Campaigns are divided into ‘sessions’. Each session is classified as an ‘operation’. Hence the ‘Binder content – Sieve size analysis’ campaign was subject to 3 sessions in 2002, 2003 and 2007 and EAPIC performed 6 operations (3 ‘Binder content’ sessions, one ‘PCG’ session, one ‘LA-MDE’ session, one ‘Blue-flow test’ session).

Selection of participants
A call to applicants is issued through professional unions, the technical network and the standardization commissions involved. The participating laboratory commits to meet the deadlines set, and apply the standard or operating method specified.

Unlike procedures implemented for organizing precision experiments, for which representativity of laboratories is required, the candidates are accepted without selection draw (except exceptional case).

If the participating laboratories are too numerous against the constraints of an experiment (size of stocks, performance time,…) the experiment can be performed in two or several sessions. Performance in several sessions spreads the work load and offers more flexibility to participating laboratories. Conversely, if too few laboratories are included to a session, the latter may be cancelled.

Method for manufacturing objects subject to testing

Support laboratories prepare the objects to be tested by applying procedures approved by the specialized group. Quantities must consider the number of laboratories planned, the nature of the test, number of replicas and conservatory materials to be used as reference after interlaboratory experiment. The main difficulty is to guarantee sample homogeneity, and also their stability over time.

Precautions are applied as upstream as possible for supplying and manufacturing the samples.

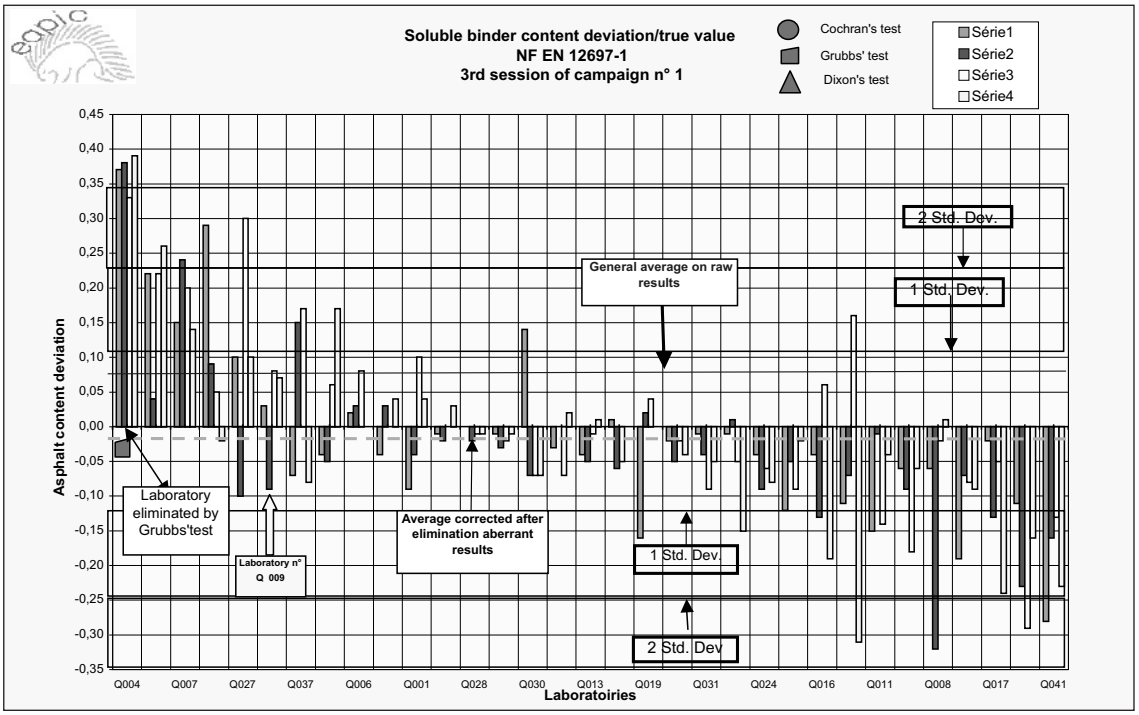
Homogeneity is checked by samples and analyses on the stock made, as per the principles of appendix B of standard ISO 13528.

Data analysis

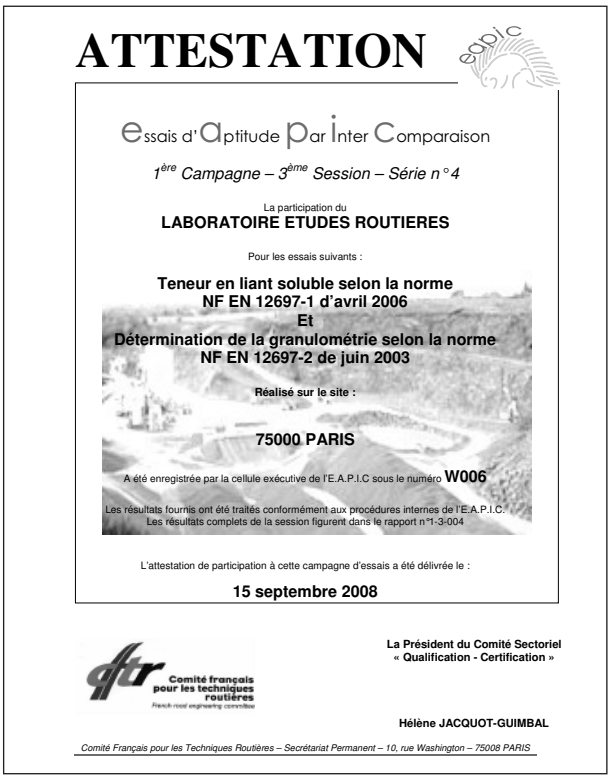
Data analysis is performed by the executive cell, by applying documented procedures based on the NF ISO 5725 standard. Data are coded to ensure confidentiality. The participating laboratories are informed of the ‘true’ values, when they are known, their result is integrated to a general bar chart showing all results or differences between real values and the average values obtained.

As defined by the NF ISO 5725 standard, the statistical tests of COCHRAN's test (dispersion values [intra-laboratory] aberrant on the 1% threshold) and GRUBBS's test (average values [inter-laboratory] aberrant on the 1% threshold) are applied to the test results. DIXON's test detects aberrant individual test values. Standard deviation and repeatability limit, as well as standard deviation and reproducibility limit, are calculated for the parameter considered.

Example of customized histogram



Example of certificate



ATTESTATION

Essais d'Aptitude par Inter Comparaison

1ère Campagne – 3ème Session – Série n°4

La participation du

LABORATOIRE ETUDES ROUTIERES

Pour les essais suivants :

Teneur en liant soluble selon la norme NF EN 12697-1 d'avril 2006
Et
Détermination de la granulométrie selon la norme NF EN 12697-2 de juin 2003

Réalisé sur le site :

75000 PARIS

A été enregistrée par la cellule exécutive de l'E.A.P.I.C sous le numéro **W006**

Les résultats fournis ont été traités conformément aux procédures internes de l'E.A.P.I.C.
Les résultats complets de la session figurent dans le rapport n°1-3-004

L'attestation de participation à cette campagne d'essais a été délivrée le :

15 septembre 2008

Comité français pour les techniques routières

La Président du Comité Sectoriel « Qualification - Certification »

Hélène JACQUOT-GUIMBAL

Comité Français pour les Techniques Routières – Secrétariat Permanent – 10, rue Washington – 75008 PARIS

All these results are communicated to the participants, together with a customized participation certificate mentioning the confidential code of the laboratory, to be situated in the bar charts.

Campaigns led

Since 2002, EAPIC has led 6 different operations, in 4 campaigns, one of which included 3 sessions.

The list of operations is given in table 1.

Table 1 – List of EAPIC operations led

Campaign	Opération Nr	Tests	Test standard	Session	Number of participants	Période of performance
1	1	Content in soluble binder Sieve size analysis	EN 12697-1 P 18-560	EAPIC 1.1	31	September 2002 to April 2003
	2		EN 12697-1 P 15-560	EAPIC 1.2	28	September 2003 to June 2004
	5		EN 12697-1 EN 12697-2	EAPIC 1.3	41	September 2007 to April 2008
2	3	Maximum density of bituminous mixtures Gyratory shear press compaction	EN 12697-5 EN 12697-31	EAPIC 2.1	28	November 2004 to June 2005
3	4	Los Angeles test Micro-Deval test	EN 1097-1 EN 1097-2	EAPIC 3.1	39	April 2007 to October 2007
4	6	Methylen blue test Fine aggregate density Fine aggregate flow test	EN 939-9 EN 1097-6 EN 933-6	EAPIC 4.1	65 (2 simultaneous sessions)	October 2008 to May 2009

Example of results, deviation, repeatability 'r' and reproducibility 'R' (change over 3 sessions)

The campaign on determining soluble binder content and sieve size analysis was performed in 3 sessions. The results of these 3 sessions are given in tables 2 to 4 for example ; they show the change over these sessions.

Note: The units in test results for binder contents and passing at different sieve size are expressed in percentages. The average difference against the true value mentioned in the tables is the difference in absolute value between these percentages, and not a relative difference, as could be deducted from the percentage expression alone. This is why the units in the tables are expressed in 'absolute %'.

Table 2 – Campaign 1 – Content in soluble binder as per EN 12697-1

	Number of non-rejected laboratories	Difference between average and true absolute % value	repeatability 'r' Absolute %	Reproducibility 'R' Absolute %
EAPIC 1.1	30	- 0,15	0,21	0,35
EAPIC 1.2	23	- 0,125	0,18	0,22
EAPIC 1.3	35	- 0,03	0,23	0,31

Table 3 - Campaign 1 – Sieve size analysis EN 12697-2 screened at 0.063 mm

	Number of non-rejected laboratories	Difference between average and true absolute % value	repeatability 'r' Absolute %	Reproducibility 'R' Absolute %
EAPIC 1.1	29	0,1	0,9	1,0
EAPIC 1.2	28	0,5	1,3	1,5
EAPIC 1.3	40	- 0,4	1,2	1,6

Table 4 - Campaign 1 – Sieve size analysis EN 12697-2 screened at 2.0 mm

	Number of non-rejected laboratories	Difference between average and true absolute % value	repeatability 'r' Absolute %	Reproducibility 'R' Absolute %
EAPIC 1.1	31	0,0	1,2	2,4
EAPIC 1.2	26	0,2	1,3	1,8
EAPIC 1.3	39	- 0,5	1,3	3,0

Table 5 - Campaign 1 - Sieve size analysis EN 12697-2 screened at 6.0 mm

	Number of non-rejected laboratories	Difference between average and true absolute % value	repeatability 'r' Absolute %	Reproducibility 'R' Absolute %
EAPIC 1.1	31	1,6	3,0	4,3
EAPIC 1.2	27	1,0	2,5	3,9
EAPIC 1.3	39	- 0,6	3,1	5,2

Comments

When determining binder content, we had observed over the first two sessions that the difference between the 'true' value (components weighed) and the value measured by the analysis led (in absolute value) to an average difference of 0.15 % or 0.125 %. During the last session, this difference was considerably reduced, close to zero. No obvious explanatory factor appears, the aggregates have the same origin, an identical replica preparation method, performed by the same support laboratory. The origin of the 'difference' observed during the first experiments is currently unexplained, and we must chose carefully the nature of materials and change in methods of the next sessions on the subject.

The last results show that the introduction of a systematic corrective value in analysis results is not justifiable.

The differences and repeatability and reproducibility values do not show any noticeable change over the 3 sessions.

Method validation

The equivalence between the methods used to determine asphalt content, described explicitly in the standard, such as cold dissolution, and the method using automatic device was demonstrated thanks to the EAPIC results.

Binder content between the automatic device method and the 'classic' cold dissolution method, leads to the same average difference against the 'true value', dispersion during the 3rd session being slightly stronger than in the classic method. In sieve size analysis, a slight difference in fine content of 0.3% was seen, a value which is lower than with the classic method. Uncertainty estimated on the 'real' value of fines content does not allow to conclude that the result of one or the other method is closer.

Table 6 – The campaigns programmed

Year	EAPIC operation Nr.	Identification of the session campaign's ability test	Nature of the test
2009	EAPIC 8	E6.1	Wheel
2009	EAPIC 9	E7.1	Polishing stone PSV value
2010	EAPIC 10	E8.1	Water sensitivity
2010	EAPIC 11	E2.2	Giratory shear compaction
2010	EAPIC 12	E1.4	Binder content, grading

4 - In practice

How to participate ?

➤ Visit the CFTR website: http://www.cftr.asso.fr/ then 'Produits', then 'Qualification-Certification', then 'EAPIC'.

➤ Future sessions are published at the time of call for candidates in the RGRA magazine (Revue Générale des Routes et Aérodromes).

➤ Directly: LREP the executive cell updates a list of 'clients', you may pre-register for the list, to be informed of campaigns being launched, by addressing your requests to:

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email:
jean-luc.delorme@developpement-durable.gouv.fr

Nicole VERCHERE
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LREP - 319, avenue Georges Clemenceau
BP 505 - 77015 MELUN Cedex
Fax : +33 (0)1 60 56 64 01

Cost ?

Cost is determined by the expenses engaged for preparing the objects submitted to the test, for logistics, processing and publication of results, according to the number of participants expected. Work to be performed by the support laboratory is usually preponderant in this equation.

For information, during the campaigns led, participation was about:

- 1500 € excluding VAT for the campaign 'Binder content – Grading analysis',

- 2500 € excluding VAT for the campaign 'Rotating shearing press – Maximum density'

- 990 € excluding VAT for the campaign 'Los Angeles + Micro-Deval'.

Satisfaction surveys

They are performed upon each operation. They bear on the quality of relations with EAPIC, the technical skills, organisation, quality of the report, compliance with lead times.

➤ Legibility of reports is the main comment. The bar charts were customized to better view the laboratory results, an image of the population has been added. A new presentation is being prepared for the next sessions, with a table of differences against the conventional value.

5 - Summary and prospects

The first campaign 'Binder content – Sieve size analysis', divided into 3 sessions, mobilised about one hundred participating laboratories. Campaigns dedicated to aggregates, Los Angeles, Micro-Deval, blue test, sand flow test also drew over a hundred candidates. This shows major expectations by the profession.

The large number of participants per session (at least 30) allows representative statistical use.

EAPIC reports were appropriate tools during audits for accredited or certified laboratories.

EAPIC contributed to the application of European standards and their validation (e.g. Real density on mix).

Glossary:

'r' repeatability: *results obtained on the same object or a similar object in the same measuring conditions: measurement procedure, operators, measurement system, operating conditions, location, during a short period.*

'R' reproducibility: *results obtained on the same object or a similar object in different measurement conditions: locations, operators, measurement procedure, measurement system, operating conditions , but only for a short period of time.*

Campaign: *set of operations within a same type or family of experiments. A campaign is divided into one or several sessions.*

Operation: *operations include launching a session, construction of replicas, collection and processing results.*

Replicas: *individual samples produced by the support laboratory and considered as similar. Each participating laboratory receives several replicas, usually 3 or 4.*

Session: *operation on a given test campaign.*

Grubbs' test: *this statistical test, codified in the ISO 5725 standard, allows detecting an outlying average (1% threshold) among the population of results.*

Cochran's test: *this statistical test, codified in the ISO 5725 standard, allows detecting an aberrant dispersion (1% threshold) among the population of results.*

Dixon's test: *this statistical test allows detecting an aberrant value isolated among the population of results.*

True value: *a 'true' value is a value known with a low uncertainty. For example, granular fractions and quantity of binder in asphalt samples are weighed very precisely, and mixing devices are left stored in containers to avoid losing bitumen particles. Binder content is then considered as a 'true' value to which the values measured can be compared.*

Conventional value: *for certain quantities, true values do not exist, e.g. material density. The average value obtained during the session, after removing outliers values, is considered as being the conventional value.*

This note was written by Jean-Luc Delorme (LREP) with the participation of the EAPIC group

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