CEN TC336/WG2 Round Robin on bituminous emulsions

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ABSTRACT

In Europe, the implementation of CE marking has emphasized the need for simple, robust and reliable test methods. In the case of bituminous emulsions, these requirements are not always met and the WG2 working group of the CEN TC336 standardization committee (bituminous binders) is devoting a large part of his work to improve this situation. One of the on-going actions aims at replacing the present standard for determination of water content (EN 1428 - azeotropic distillation) by a much simpler, faster and solvent-free method using a drying balance. The second action aims at replacing the reference fillers (Sikaisol and Forshammer) presently used for the determination of breaking behavior (EN 13075-1) by a filler offering better guarantees of future availability. A third action aims at improving the emulsion recovery and stabilization procedures described in EN 13074-1&2. After a series of investigations and preliminary testing among its members, WG2 was able to propose operating conditions for the drying balance and could identify a new filler (Caolin Q92) for EN 13075-1. A better insight into the factors affecting the outcome of EN 13074-1&2 could also be gained and led to new proposals for more strict operating conditions. To validate these proposals and define precision data (repeatability/reproducibility), WG2 decided then to launch an extensive Round Robin test program. Conducted on three different emulsions, the program has gathered 46 laboratories from 14 countries. The paper discusses its outcome (precision data for the drying balance method, precision data and conversion factors to Forshammer filler for EN 13075-1, improvements and further progress to be made for EN 13074-1&2). It also underlines the importance (and need) of pre-normative research to support the work of standardization committees. Acknowledgements are made to CEREMA, AYTON Products and COLAS Austria for the preparation and dispatching of test samples, to CEREMA for the statistical analysis and special thanks go to the SFERB emulsion producer's associations for their financial support.

Keywords: Emulsions, Quality assurance, Standardisation, Testing

1. INTRODUCTION

In Europe, the implementation of CE marking has emphasized the need for simple, robust and reliable test methods. In the case of bituminous emulsions, these requirements are not always met and the WG2 working group of the CEN TC336 standardization committee (bituminous binders) is devoting a large part of his work to improve this situation. One of the on-going aims is replacing the present standard for the determination of water content (EN 1428 - azeotropic distillation) by a much simpler, faster and solvent-free method using a drying balance. There is also a need to find alternative reference fillers for the determination of emulsion breaking behavior since the Sikaisol filler requested by the EN 13075-1:2009 test standard is no longer produced and running out of stock. A third aim is improving the emulsion recovery and stabilization procedures described in EN 13074-1&2. After a series of investigations and preliminary testing among its members, WG2 was able to issue proposals on these three topics but those needed to be further validated and complemented with precision data (repeatability and reproducibility values). WG2 decided thus to launch an extensive Round Robin test program which took place in the period going from April to August 2014 and gathered up to 46 laboratories from 14 countries.

2. THE INVESTIGATED TEST METHODS

2.1 Water content of bituminous emulsions using a drying balance

A drying balance, or moisture analyzer, is basically a precision balance in which the product sample can be heated and maintained at a certain temperature to evaporate the moisture it contains. The evolution of sample weight is monitored over time, the objective being to reach constant weight equilibrium i.e. the stage where all the water has been evaporated. Critical parameters are the temperature (which impacts the speed of the evaporation process) and the so-called "stopping criterion" (usually defined in mg/s) which defines the end of the test. A stopping criterion set at 1 mg/50 s means that the test is stopped when the weight does not change by more than 1 mg in 50 s. As a matter of fact, such a test is much simpler, safer and, above all, much faster (average duration of about ½ hour) than the traditional EN 1428 azeotropic distillation or EN 1431 residual binder by distillation methods. It is thus ideally suited for production control operations. In practice, there are however some hurdles. Some emulsions used in paving applications are still fluxed with mineral (generally of petroleum origin) or vegetal flux oils. At the temperatures applied in the drying balance, mineral fluxes may partly evaporate together with the water. The presence of such volatile components does also complicate the definition of an adequate stopping criterion since the test may go too far if the criterion is set at too low values. This problem is not encountered with vegetal fluxes which are not volatile at the applied temperatures but may nevertheless generate fumes if heated too strongly. There is thus an optimum to be found for the test temperature (the higher the temperature, the faster the test but also the more chances for evaporating non-water components). The same is true for sample size (the smaller the sample, the faster the test but also the lower the precision). After some preliminary testing at their respective laboratories, the WG2 experts came to following proposals with regard to test conditions:

- Size of emulsion sample : (4 ± 0.5) g
- Test temperature : 110°C for mineral fluxed emulsions with up to 1,5% flux and emulsions with more than 5% of vegetal flux;
  up to 130°C for vegetal fluxed emulsions with less than 5% of flux;
  up to 150°C for non-fluxed emulsions.
- Stopping criterion : to be set at a value closest to 1 mg/50 s (not all equipment gives full freedom for the setting of this parameter)

Emulsions which are fluxed with more than 1.5% of a mineral flux do therefore fall "out of the scope" of the method. This does not mean that the method cannot be used but it needs to be checked against the reference EN 1428 or EN1431 methods and, if necessary, a correction factor has to be established to account for loss of flux during the test.

2.2 Revision of EN 13075-1:2009 – Determination of breaking behavior – mineral filler method

In this method, a reference filler is added at a uniform rate to (100 ± 1) g of stirred cationic bitumen emulsion until the emulsion is completely broken. The corresponding mass of filler (in grams) multiplied by 100 and divided by the amount of emulsion (in grams) is the breaking value.

In the presently applicable 2009 version of EN 13075-1, the reference filler to be used is the "Sikaisol" silicious filler. However, the obtained breaking value has to be converted into an equivalent "Forshammer" (another, previously specified, reference filler) breaking value (which is still referred to by the EN 13808:2013 bituminous emulsion specification). Due to the announced shortage and withdrawal of the Sikaisol filler, WG2 had to identify possible alternative products, which resulted in the selection of a Spanish filler "Caolin Q92".

For the revised version of EN 13075-1, WG2 decided therefore to maintain three possible fillers:

- Forshammer, since specification classes in EN 13808:2013 are still based on this filler
- Caolin Q92 as the alternative to Sikaisol
- Sikaisol, for continued use till exhaustion of the present stock

The revised standard had however to provide the appropriate conversion factors from Caolin Q92 and Sikaisol breaking values to Forshammer breaking values. In addition, precision data were to be established since reproducibility figures are not available in EN 13075-1:2009.

2.3 Revision of EN 13074-1& 2 – Recovery and stabilization of binders from bituminous emulsion or cut-back or fluxed bituminous binders

The objective of these two methods is to get a residual binder for further testing. In EN 13074-1:2011, a thin layer of product is spread onto a suitable sheet of material and conditioned for 24h at ambient temperature in the laboratory, before being transferred into a ventilated oven for 24 h at 50 °C. In the case of bituminous emulsions, this step is thought to essentially evaporate the water but, in the case of fluxed emulsions (volatile fluxes) it may also evaporate part of the flux oil. In EN 13074-2:2011, the binder recovered after EN 13074-1 is kept in the ventilated oven for another 24 h period at 85°C, leading to the so-called "stabilized" binder. These conditioning methods are repeatedly blamed for their poor precision, as shown for instance by the highly scattered penetration and softening point measurements on recovered and stabilized binders. This has triggered yet another request for an anticipated revision of these methods. The task is however made difficult by the fact that many operating parameters may influence the end result and that their impact may differ depending on the formulation of the tested emulsion. Also here the presence and the nature of flux oils play a key role.

3. THE ROUND ROBIN TEST PROGRAM

3.1 Objectives and products to be tested

The main goals of the program can be listed as follows:

- Measurement of water content by drying balance, with the objective of establishing precision data and confirming the domain of applicability of the method (3 types of emulsions).
- Measurement of breaking value (EN 13075-1) with different fillers, with the double objective of establishing conversion factors and precision data.
- Performance of recovery and stabilization tests (EN 13074-1&2) under tightened operating conditions (see table 1), with the objective of gathering input on used test conditions and their possible impact on the characteristics (penetration and softening point) of the stabilized binder.

To cover both the scope of the draft standard on water content by drying balance and a wide enough range of breaking index values, three types of emulsion formulations have been targeted by WG2.

Emulsion A Relatively "fast breaking" surface dressing emulsion based on pure bitumen, with about 1.5% of mineral flux.

Emulsion B Surface dressing emulsion based on pure bitumen fluxed with 4 to 5% of a vegetal flux.

Emulsion C "Medium-setting" slurry-seal emulsion based on un-fluxed pure bitumen.

3.2 Test program

The final test program with the instructions given to the participants is summarized in Table 1.

Table 1: Summary of test program

	Emulsion A 2 samples per laboratory	**Emulsion B** 2 samples per laboratory	**Emulsion C** 2 samples per laboratory
Characteristics			
Bitumen grade	160/220	70/100	160/220
Bitumen content (%)	65	67	60
Flux type	mineral	vegetal	no flux
Flux content (%)	1,5	4 - 5	-
Breaking behaviour	rapid setting (surface dressing)	rapid setting (surface dressing)	slow setting (slurry-seal)

Instructions	**Drying balance**		
Sample size	(4 ± 0,5) g	(4 ± 0,5) g	(4 ± 0,5) g
Test temperature	110°C	130°C	150°C
Stopping criterion	closest to 1 mg/50 s	closest to 1 mg/50 s	closest to 1 mg/50 s
Filter paper	Should pereferably be a glassfibre fabric. If a cellulose fabric is used, it has to be dried before use.		
Number of test repetitions	2 (1 for each sample)	2 (1 for each sample)	2 (1 for each sample)

Instructions	**Breaking value**
Fillers to be tested	Forshammer - Sikaisol - Caolin Q92
Number of test repetitions	2 x 2 (2 repeats for each sample)
Specific items to be reported	Procedure to be used : semi-automatic or manual

Instructions	**Recovery and stabilisation**
Results to be generated	Loss of mass after 24h, 48h and 72h Softening and penetration after recovery + stabilization
Number of test repetitions	2 (1 determination for each sample)
Specific Requirements	Ventilated oven = with possibility to introduce fresh air. Ventilation orifice should be set open at 100%. Ventilation rate to be set at usual value.
	Minimum distance between two plates on the same shelve, between a plate and the walls of the oven to be 3 cm, preferably 5 cm.
	Minimum distance between shelves and between shelves and the bottom or top of the oven to be 7 cm, preferably 10 cm.
	Oven has to be loaded with the maximum possible number of plates (while satisfying the above requirements). This means that some plates may be left empty.
	Use of metallic plates with a minimum thickness of 2 mm (to ensure rigidity)
	Maximum internal height of plate edge = 20 mm
	No use of anti-stripping coatings such as silicone paper or fabric, baking parchment, ….
	Binder after stabilisation has to be recovered with a spatula. Homogenisation and moulding of test samples for R&B and penetration measurements shall be done immediately after stabilisation, while following the recommendations given in EN 13074-2, § 6.3.

3.2 Sampling and organization

Three laboratories have offered to collect and dispatch the necessary emulsion samples (Emulsion A by CEREMA St-Brieuc in France, emulsion B by COLAS Austria and emulsion C by AYTON Products in UK). To eliminate possible sources of error and for convenience reasons, the ordering and further dispatching of filler samples (depending on the needs expressed by each participating laboratory) has also been taken over by the Cerema St-Brieuc for the Caolin and Sikaisol filler and by AYTON Products for the Forshammer filler.

End of April 2014, TC336/WG2 sent out a call for participation to all TC336 member organizations, in the form of a document explaining the purpose and content of the planned Round Robin exercise, together with the relevant test standards, additional instructions and pre-defined data reporting sheets (Excel files) [1]. With up to 46 participating laboratories (see table 2), the response turned out to be far beyond expectations.

The sending of the samples took place almost simultaneously on the 16th and 17th of June and laboratories were given instructions to proceed with testing (at least for drying balance and breaking value) within maximum two weeks after reception of the samples.

Table 2: Participants per country

Country / Number of participating laboratories					
Austria	3	France	6	Netherlands	2
Belgium	2	Germany	3	Spain	10
Croatia	4	Ireland	3	Sweden	4
Czech Republic	1	Italy	1	UK	4
Denmark	2	Lithuania	1		
Total : 46					

3.3 Available results

All participating laboratories were not able to perform the full test program, mainly because some of the required equipment was not available, or because of their overall workload. For most of the contemplated test methods, the number of participating laboratories was however well in excess of 20, which guarantees the validity of the Round Robin. A synopsis of the number of available results per test method is given in Table 3.

Table 3: Number of available results

Drying Balance	Breaking value			Recovery & Stabilization		
	Forshammar	Caolin	Sikaisol	Emulsion A	Emulsion B	Emulsion C
27	**39**	**41**	**39**	**23**	**20**	**26**
	In two cases, emulsion B has not been tested In one case, only emulsion C has been tested			In one case, weight loss has not been recorded		

4. TEST RESULTS

4.1 Drying balance

The synopsis of the obtained results is gathered in Table 4. The measured average values for the water content of the three different emulsions is well in line with their theoretical value. Although this cannot be taken as a general proof (only one specific type of mineral and vegetal flux have been investigated), it nevertheless suggests that the temperature limits which have been defined in the project test standard [2] are adequate. Repeatability values are quite close, irrespective of the emulsion-type and slightly better than the value stated in EN 1428 (1 % mass fraction in absolute value). Reproducibility values are higher and more differentiated, the worst result being obtained, as expected, for emulsion A containing 1.5% of a volatile flux oil. The corresponding value of 1.8 % mass fraction in absolute value stays however lower than those stated in EN 1428 (2% for non-fluxed emulsions and 3% for fluxed emulsions).

Table 4: Drying balance – Repeatability and Reproducibility values

Product	**Emulsion A**	**Emulsion B**	**Emulsion C**
Bitumen/Flux	160/220 - 1,5% mineral flux	70/100 - 4-5 % vegetal flux	160/220 - no flux
Theoretical water content (%)	35	33	40
Test temperature (°C)	110	130	150
Overall mean value (mass %)	**34,9**	**32,9**	**40,2**
Distribution	log-normal	log-normal	log-normal
Nbr. of labs after elimination of the outliers or due to missing results	26	24	22
Repeatability - r (mass %)	**0,6**	**0,4**	**0,5**
Reproducibility - R (mass %)	**1,8**	**1,6**	**1,2**
Values adopted in project prEN 16849 [2]	**Repeatability** (mass %) : **0,6** **Reproducibility** (mass %) : **1,8**		

A deeper analysis of the individual results has been made so as to identify possible systematic impacts of one or the other operating parameters. No systematic trends could be evidenced with regard to:

- The used apparatus. Eight different brands, for a total of 18 different models, have been used by the participants. Although a definitive statement cannot be made (some models were only used by a single laboratory), no obvious differences in response could be detected.
- Variations in the mass of the test sample. All laboratories used the prescribed amount of (4 ± 0.5) g for the test sample and the possible differences (less than 0.5 g) between two test samples did not have any particular impact on the test results.
- Date of testing. Due to transportation times and laboratory constraints, samples could not always be tested as quickly as foreseen. Most of the testing was done in the first 20 days after sampling, but for some samples the delay stretched over to 30 days and, for a few, to much more. Nevertheless, we could not evidence any obvious influence of the delay before testing on the variability of results.
- Stopping criterion. A majority of laboratories was able to apply the prescribed stopping criterion of 1 mg/50 s. But other values have also been used. Figure 1 shows the results obtained in the case of Emulsion A, the stopping criterion values being ranged in ascending order from left to right. Also here, there is no obvious influence of this parameter on the variability of the results.

Finally, an interesting operating parameter to be discussed is the use of an absorbing filter paper. The draft test standard prescribes the use of such an absorbing filter fabric which is to be placed on top of the emulsion sample (which may also be placed in-between 2 such fabrics). The purpose of this filter is to ensure a more homogeneous repartition and an even drying of the emulsion sample. The nature of the fabric has however its importance since cellulose fibers, in comparison to glass fibers, are prone to absorb humidity which, if not dried properly before testing, could affect the result. Most of the laboratories used glass fibers. Figure 2 shows the results obtained with emulsion A. The results obtained with cellulose filter papers tend to belong to the extremes (high or low end of water content distribution). The same trend has been observed for the other two emulsions and has motivated TC336/WG2 to impose the use of a glass fiber fabric in its final proposed draft for the test method [2].

Figure 1: Drying balance – Impact of stopping criterion

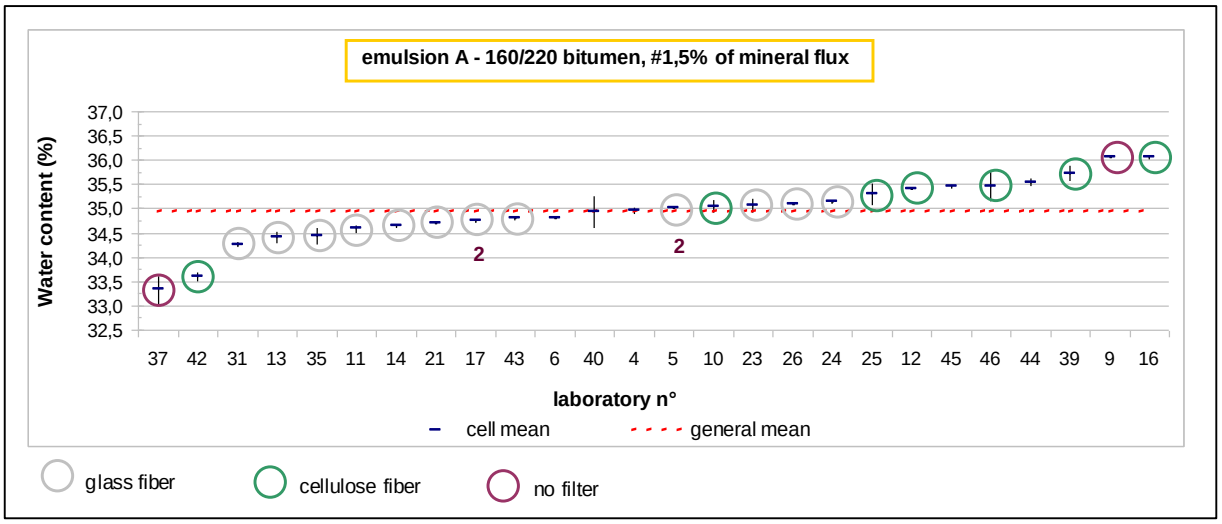

Figure 2: Drying balance – Impact of filter paper

4.2 Breaking behavior – mineral filler method

Average breaking value results for the different emulsions and fillers and the associated repeatability and reproducibility figures are gathered in Table 5.

Repeatability data, expressed as a percentage of the mean value, vary from 5% to 10%, which is in-line with the repeatability value (10%) stated in the 2009 edition of EN 13075-1. Reproducibility values, which range from 20% to 40% of the mean value, are not particularly good and reflect what is generally observed (and complained about) concerning the mineral filler method. It is to be reminded that, so far, reproducibility values had not been established for this method. A closer look leads to following remarks:

- There is no clear impact of the emulsion type on the precision data which seem to depend on both the filler type and the tested emulsion.
- The Sikaisol filler tends to give a better precision, both for repeatability and reproducibility, which is quite frustrating since this product will no longer be available!
- Precision data obtained for the Forshammer and the Caolin Q92 filler are more or less similar.

From these observations, TC336/WG2 decided to adopt repeatability and reproducibility values of 10% and 40% respectively of the mean measured value, for the proposed revised version of EN 13075-1. Since there is no clear advantage of Forshammer versus Caolin Q92 in terms of precision, all three fillers are given the same status of "reference filler" (which may be used in case of dispute), the Sikaisol filler being maintained in the standard so as to allow its continued use till exhaustion of existing stocks.

Table 5: Breaking value – Repeatability and Reproducibility values

Product	**Emulsion A**			**Emulsion B**			**Emulsion C**		
Bitumen/Flux	160/220 - 1,5% mineral flux			70/100 - 4-5 % vegetal flux			160/220 - no flux		
Theoretical water content (%)	35			33			40		
Test temperature (°C)	110			130			150		
	Forshammer	Caolin Q92	Sikaisol	Forshammer	Caolin Q92	Sikaisol	Forshammer	Caolin Q92	Sikaisol
Overall mean value	**99**	**89**	**77**	**99**	**85**	**76**	**163**	**145**	**126**
Ratio $BV_{Forsh.}$ / BV_{filler}	**1**	**1,11**	**1,29**	**1**	**1,16**	**1,3**	**1**	**1,12**	**1,29**
Distribution	log-normal	log-normal	log-normal	log-normal	log-normal	log-normal	log-normal	log-normal	log-normal
Nbr. of labs after elimination of the outliers or due to missing results	36	36	35	30	31	29	36	36	35
Repeatability	**10**	**9**	**6**	**9**	**4**	**5**	**10**	**11**	**6**
Reproducibility	**43**	**39**	**26**	**29**	**35**	**18**	**63**	**59**	**28**
Repeatability (% of mean)	**10**	**10**	**8**	**9**	**5**	**7**	**6**	**8**	**5**
Reproducibility (% of mean)	**43**	**44**	**34**	**29**	**41**	**24**	**38**	**41**	**22**
Values proposed for the revised version of EN 13075-1 [3]	**Ratio $BV_{Forsh.}$ / $BV_{Caolin\ Q92}$: 1,2** **Ratio $BV_{Forsh.}$ / $BV_{Sikaisol}$: 1,3** **Repeatability** (% of mean) : **10** **Reproducibility** (% of mean) : **40**								

In EN 13075-1, the determination of the breaking value may be conducted through a manual or semi-automatic procedure, which could have an influence on the end result. In the frame of the Round Robin exercise, both procedures were represented almost equally, which allows comparisons to be made. This is done in Figure 3. Results tend to be systematically higher with the semi-automatic procedure, especially with the Caolin Q92 filler (difference of about 10%). With regard to precision data, no systematic trend in favor of one or the other method could be evidenced. Considering the overall precision level of the method, it has finally not been found necessary to make a distinction between the two procedures in the frame of the revised EN 13075-1 [3].

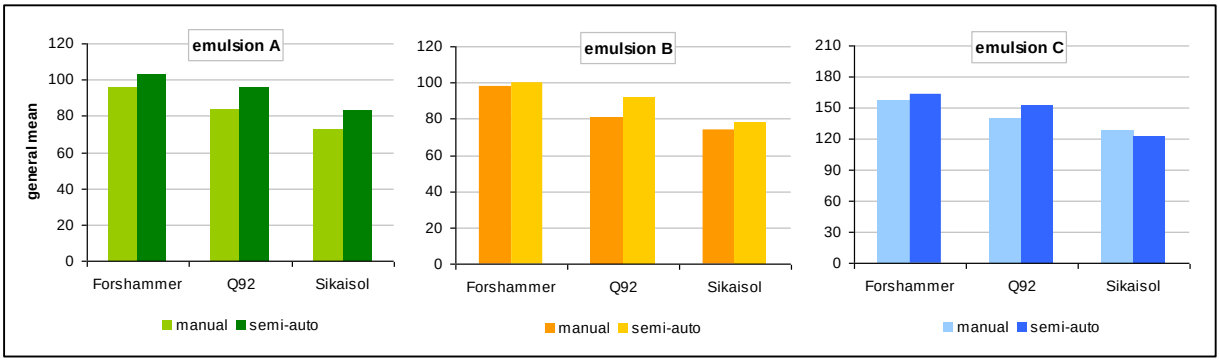

Figure 3: Breaking value – Impact of test procedure (manual or semi-automatic)

As explained in § 2.2, the test standard must specify how to convert breaking values obtained with either the Sikaisol or the Caolin Q92 filler into equivalent "Forshammer values". The relationship between these different breaking values as obtained in the TC336/WG2 Round Robin is shown in Figure 4. As a matter of fact, due to the poor reproducibility, the representative data points are quite scattered, which does not give a great statistical significance to the tentative correlation lines (low R^2 values). TC336/WG2 had however to make a decision, which led to the conversion factors given in Table 5, which call for two additional comments:

- The conversion factor of 1.3 for $BV_{Forsh.}/BV_{Sikaisol}$ differs from the present value of 1.4 in EN 13075-1:2009 but had to be retained since the new data set is far more important than the previous one.
- Based on Spanish background experience with this filler, a value of 1.2 has finally been adopted for $BV_{Forsh.}/BV_{Caolin\ Q92}$ rather than the 1.1 value suggested by the regression analysis.

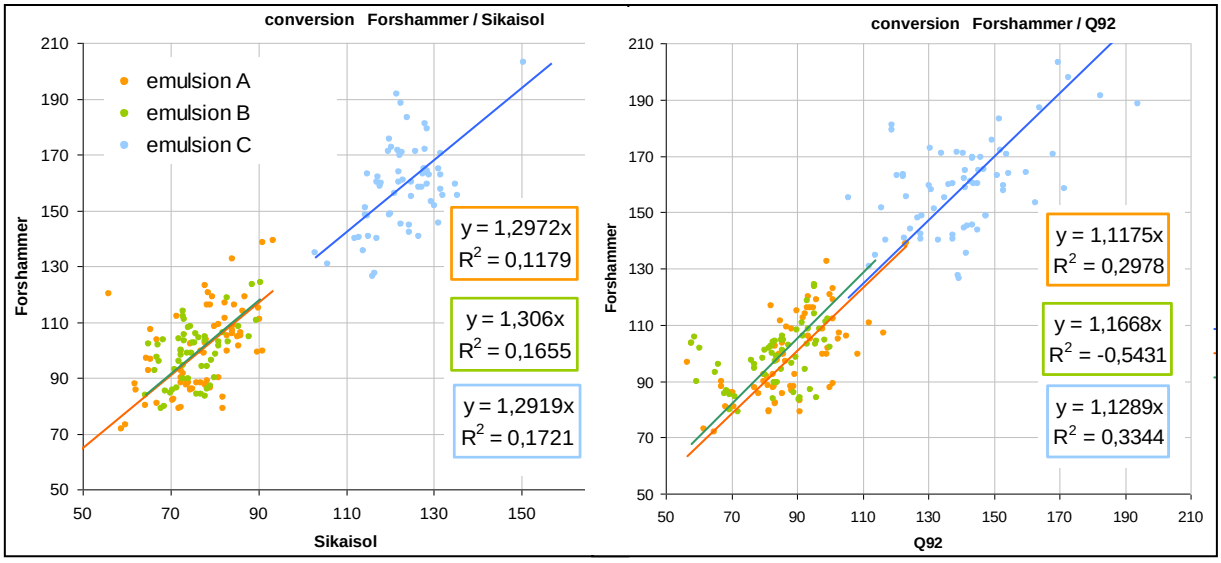

Figure 4: Breaking value – Determination of conversion factors

4.3 Recovery and stabilization

Average penetration and softening point results for the three tested emulsions and the associated repeatability and reproducibility figures are gathered in Table 6. Despite the tightening of some of the operating parameters (see Table 1), precision is still not satisfactory. When comparing to the values given in EN 1426, repeatability values are multiplied by a factor 2 whereas reproducibility is worse by a factor 6. For the softening point (EN 1427), both repeatability and reproducibility values are multiplied by two. Three comments can be made:

- The precision values which have been obtained reflect not only the scatter due to the recovery and stabilization procedures but also the scatter which is specific to the penetration and softening point tests.
- Recent experience (for instance in the frame of the Round Robin tests conducted each year in France) show that reproducibility for the penetration test is generally worse (up to 20% instead of 6%) than what is assumed in EN 1426.
- The measured precision values do not differ from one emulsion to another.

Table 6: Penetration and Softening Point after EN 13074-1&2 – Precision data

Product Bitumen/Flux	**Emulsion A** 160/220 - 1,5% mineral flux		**Emulsion B** 70/100 - 4-5 % vegetal flux		**Emulsion C** 160/220 - no flux	
	Penetration at 25°C (mm/10) EN 1426:2007	**Softening Point** (°C) EN 1427:2007	**Penetration at 25°C** (mm/10) EN 1426:2007	**Softening Point** (°C) EN 1427:2007	**Penetration at 25°C** (mm/10) EN 1426:2007	**Softening Point** (°C) EN 1427:2007
Overall mean value	**147**	**41,1**	**114**	**44,6**	**121**	**42,8**
Distribution	log-normal	log-normal	log-normal	log-normal	log-normal	log-normal
Nbr. of labs after elimination of the outliers or due to missing results	19	21	18	17	24	24
Repeatability	**11**	**1,6**	**10**	**1,7**	**9**	**1,9**
Reproducibility	**52**	**4,2**	**44**	**4,5**	**38**	**4,6**
Repeatability (% of mean)	**8**	-	**9**	-	**7**	-
Reproducibility (% of mean)	**36**	-	**38**	-	**32**	-

Reference values			
Penetration at 25°C (EN 1426:2007)	**Repeatability** (% of mean) :	**4**
	Reproducibility (% of mean) :	**6**
Softening Point (EN 1427:2007)	**Repeatability** (°C) :	**1**
	Reproducibility (°C) :	**2**

How to explain this large scatter?

Although they may have a significant influence, the effective ventilation characteristics of the used ovens are often not well known to the users (this was again reflected by the questionnaire to the participants). Nevertheless, the weight losses reported after the EN 13074-1 (2 days) and EN 13074-2 (3 days) procedures proved to be quite homogeneous and close to the theoretical values (which are already obtained after 2 days and, except for emulsion C, even after the first day of storage at ambient temperature). Ventilation artefacts were therefore probably not the major contributor to the observed scatter in results (Figure 5).

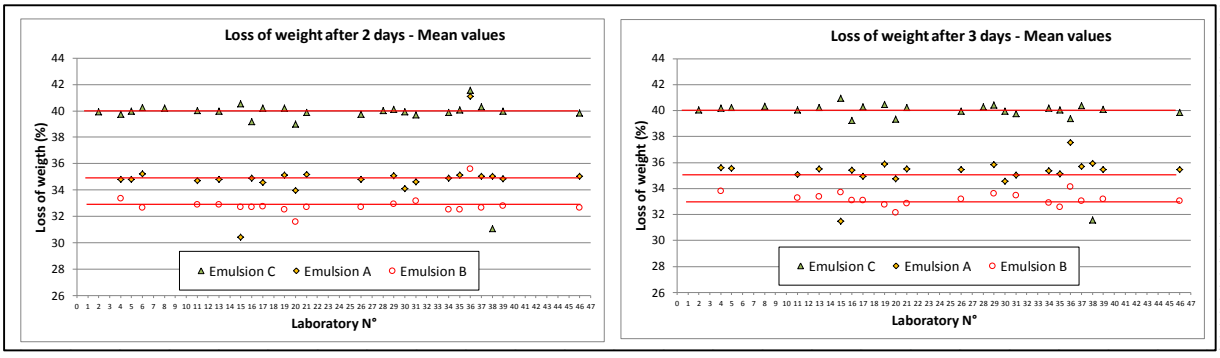

Figure 5: Recovery and stabilization – Weight loss

The restrictions which had been imposed by TC336/WG2 to limit possible testing artefacts (minimum thickness of plates to ensure rigidity, no use of anti-stick fabric to avoid irregular binder films, maximum internal edge height to favor evaporation, positioning of test plates inside the oven, …) have generally been well observed (although with some exceptions). Nevertheless, it is quite astonishing how diverse the different operating conditions turned out to be (see Table 7 and Figure 6).

Table 7: Synopsis of used operating parameters

	Variability of operating parameters				
Internal volume (l)	**40 to 100**	**100 to 170**	**200 to 250**	**400 to 1080**	
Nbr. Labs.	3	10	6	5	
Plate size (cm²)	**R - 300 to 2100**	**S - 320 to 1600**	**C - 160 to 710**	(R = rectangular, S = square, C = circular)	
Nbr. Labs.	15	9	2		
Internal height of plate edge (mm)	**≤ 5**	**6 to 10**	**11 to 15**	**16 to 20**	**> 20**
Nbr. Labs.	5	8	6	5	1
Ratio (surface plate / internal height of edge) (cm²/cm)	**80 ≤ r < 500**	**500 ≤ r < 1000**	**1000 ≤ r < 2000**	**r ≥ 2000**	
Nbr. Labs.	8	10	4	3	
% of shelf surface occupied by plates	**10 ≤ s < 20**	**20 ≤ s < 40**	**40 ≤ s < 60**	**60 ≤ s < 80**	**s ≥ 80**
Nbr. Labs.	1	8	11	2	1

Specific requirements of RRT	YES	NO
Ventilated oven ?	19	7
Minimum thickness of 2 mm	19	4
Minimum surface of 400 cm²	24	3
Internal height of plate edge ≤ 20 mm	24	1
No use of anti-stick fabric	23	3

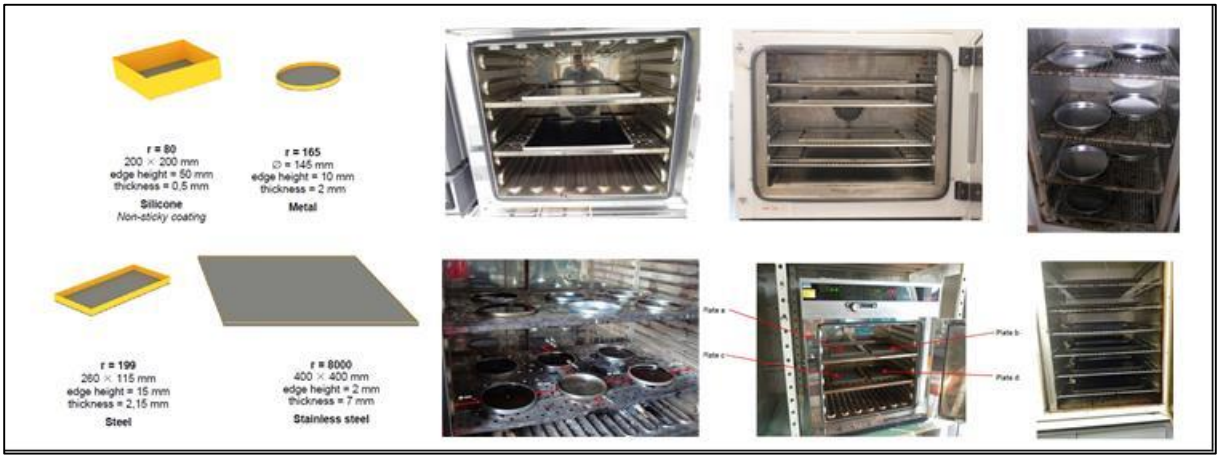

Figure 6: Recovery and stabilization – Example test plate and oven configurations

EN 13074-2 also recommends the stabilized binder to be reheated at a temperature in-between expected Softening Point + 80°C and expected Softening Point + 100°C, and this for the minimum time necessary to prepare the test samples. Also here, this resulted in quite different temperature and heating time values, as shown in Figure 7.

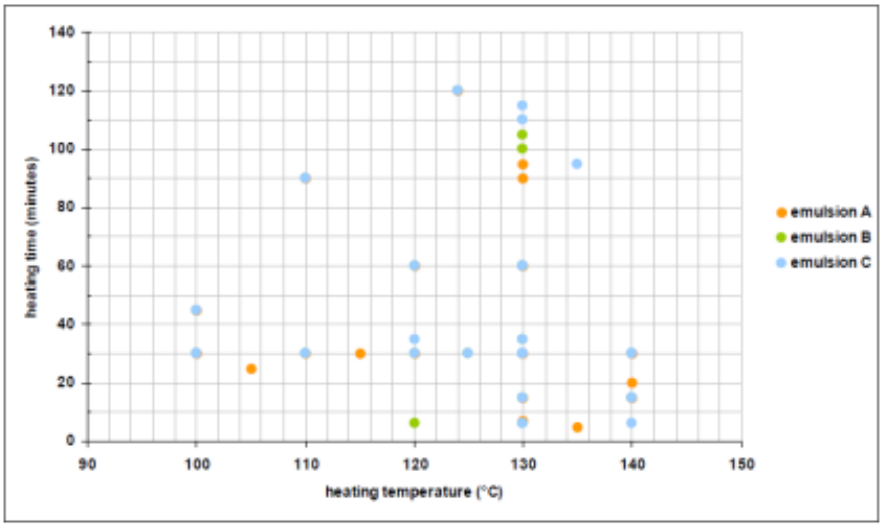

Figure 7: EN 13074-2 – Reheating times and temperatures

Considering all these variations in operating conditions, the precision data shown in Table 6 are not surprising anymore. Unfortunately, and certainly due to the large number of parameters which offered so many possible combinations (no two laboratories operating in exactly the same way), no significant influence of one or the

other could be evidenced. This means that the precision data in Table 6 probably reflect the best which can be expected from the present EN 13074-1&2 without a major change of the operating procedure.

5. CONCLUSION

It is with great satisfaction that TC336/WG2 welcomed the large number of participants to its extensive Round Robin program, which could furthermore be completed in a relatively short time frame. Even if only three different types of emulsions have been tested, it may be stated that the domain of application and operating conditions originally envisaged for the drying balance method seem to be adequate and that precision values are acceptable. This has encouraged TC336/WG2 to propose the draft procedure to CEN Enquiry [2]. The same applies to EN 13075-1 for which conversion factors could be established for the 3 different fillers. One may not be satisfied with the poor precision data which are mainly to be ascribed to the method itself, in which the appreciation of the operator plays a major role. Since EN 13075-1 is a major component of product quality control in the frame of CE marking, TC336/WG2 had however no other choice than to take these results as they are and to implement them in the new proposed draft [3]. The practical consequence is that the breaking value has to be acknowledged as being essentially of qualitative nature and this has also to be recognized by defining relatively large specification ranges in product standards (which has been done in the 2013 version of EN 13808). There is probably little to be expected from further improvements of the method as such, so that future work should better be oriented towards the search for alternative methods. Concerning the EN 13074-1&2 recovery and stabilization methods, TC336/WG2 is confronted with a dilemma. Restricting drastically the flexibility permitted by the present method in terms of oven characteristics and plate dimensions, plate positioning in the oven, sample recovery conditions, …. should certainly help in reducing the observed scatter but will also induce additional costs. Some tightening of operating conditions should nevertheless be possible and TC336/WG2 will have to make proposals in this respect. Unfortunately, since no dominant influencing factor(s) could be identified, these proposals will have to be made (and accepted) on a "common sense" basis. At this stage, it must also be emphasized that however accurate the procedure, the end result will also be conditioned by its strict observance by the user. The next step, if the inevitable scatter induced by the successive recovery + stabilization + final test procedures is not found to be acceptable would then be to look again for other alternatives (e.g. rheology testing of the residual binder without having to reheat it in bulk). It is however to be underlined that developing better or alternative methods is not really the original mission, nor really possible, for standardization working groups which function on a voluntary basis. Such standardization needs the support of pre-normative research and stakeholders should be made aware of this need.

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