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Internal Report no. 2291

Paper to the BCRA '02 conference:

Testing adhesion between bitumen and aggregate with the Rolling Bottle Test and the Boiling Test

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Summary

Innlegg til BCRA '02, 6th International Conference on the Bearing Capacity of Roads, Railways and Airfields. Lisboa 24.-26. juni 2002.

I det norske samarbeidsprosjektet PROKAS (PROporsjonering og Kontroll av ASfalt) er det gjennomført en ringanalyse på vedheftsmetodene *Koketest* og *Rulleflaskemetoden*.

Det ble brukt bitumen uten og med amintilsetning på to steinmaterialer med dårlig vedheft og to steinmaterialer med god vedheft.

Ringanalysen viser at begge metodene skiller mellom gode og dårlige bindemiddel/ stein kombinasjoner. Blandinger med middels god vedheft hadde ikke god nok reproduserbarhet. Begge prøvingsmetodene viste en positiv effekt av vedheftningsmiddelet.

Rulleflaskemetoden hadde akseptabel presisjon for de fleste bindemiddel/stein kombinasjonene, og kan brukes til produksjonskontroll av råmaterialer. Koketesten hadde tilfredsstillende presisjon kun på dårlige bindemiddel/stein kombinasjoner og kan brukes til å påvise dårlig vedheft.

I vedlegget vises postere som ble presentert på konferansen.

Key words: *asphalt mixtures, adhesion, stripping test, bitumen, amine*

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Testing adhesion between bitumen and aggregate with the Rolling Bottle Test and the Boiling Test

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Keywords: Asphalt, stripping, adhesion test, aggregate, bitumen, adhesion agent

ABSTRACT: Two adhesion test methods were evaluated in a round robin test, the "Rolling Bottle Test" and the "Boiling Test". Bitumen without and with liquid adhesion agent were used together with two stripping prone aggregates and two non-stripping aggregates.

The round robin test showed that both test methods can sort out good and bad aggregate-binder combinations. Combinations with intermediate adhesion had insufficient reproducibility. Both test methods showed a significant effect from the adhesion agent. The Rolling Bottle Test had acceptable precision for most of the aggregate-binder combinations and can be used e.g. in production control on raw materials. The Boiling Test had only acceptable precision for bad aggregate-binder combinations and may be used to identify problem materials.

1. INTRODUCTION

The norwegian PROKAS project was initiated in 1998 with a main objective to develop or adopt improved systems for mix design and quality control of asphalt mixes and pavements. PROKAS is divided in three main areas: Materials, Mix Design and Quality Control.

Due to the rather humid and cool norwegian climate and the use of certain stripping prone aggregates, adhesion agents are often used. There are numerous quarries and a wide variety in aggregate types used in asphalt mixtures. The time and cost do not always allow for comprehensive testing of the water susceptibility of asphalt mixtures. In the daily life the contractor often must rely on the given information on his raw materials.

Adhesion testing of the raw materials adds useful information to the judgement of the water susceptibility of asphalt mixtures. Such knowledge may help saving time and efforts in the laboratory testing on the actual mixtures. Test results that reveals stripping prone aggregates is a warning of possible problems in the field. Actions such as adding adhesion agent should then be considered.

Norwegian laboratories have 15 years experience with the Rolling Bottle Test. The test is proposed as an European Standard (Anonymous 2001). The Boiling Test, which is a slightly modified Texas Boiling Test (Kennedy et al. 1984), is used as a screening test on laboratory made mixtures and on asphalt mix plant samples. In both test methods the degree of bitumen coverage is determined by visual observation.

The precision and the validity of both methods have been questioned and there are only few studies on this issue (Isacsson & Jørgensen 1987, Peltonen 1997).

The Rolling Bottle Test is used to evaluate the adhesion of aggregates and binders for hot asphalt mixtures. Effect and dosage of adhesion agents is also evaluated with this method. Only the coarse fraction of the aggregates is tested (5.6/8.0 mm, 6/10 mm or 8/11 mm).

The Boiling Test is mostly used as an (in-house) control test. It has the advantage that it can be applied on the total aggregate mixture or on individual fractions. For aggregates smaller than 2 mm, the visual estimation of binder coverage can be difficult.

A norwegian round robin test on these two test methods were arranged in 2000-2001. Seven laboratories participated on each method. The aim of the study was to determine whether the test methods had satisfying precision and if limits for “acceptable adhesion” could be specified.

2. TEST METHODS

2.1 The Boiling Test

In this study washed 4/8 mm aggregate was used. The aggregate was heated to the prescribed mixing temperature and mixed with 3.0 % bitumen (by weight of aggregate). When liquid adhesion agent (“amine”) is used, it is carefully added to the heated bitumen 30 ± 5 minutes before mixing with aggregate. Portions of the mixture is spread on metal lids or silicone paper and is stored overnight at ambient temperature before testing. Two 100 g part samples is prepared for the test.

A 1000 mL beaker half filled with distilled water is heated to boiling. The part samples are boiled for ten minutes, and stirred with a glass rod at three minutes intervals. During and after boiling stripped bitumen is skimmed off the surface of the water with a paper towel. After boiling the mixture is allowed to cool in the water. The cooled water is poured off and the mixture is stored on a paper towel overnight. The average readings of two operators on the two part samples is reported.

2.2 The Rolling Bottle Test

In this study washed 5.6/8.0 mm aggregate was used. The aggregate was heated to the prescribed mixing temperature and mixed with 3.4 % bitumen (by weight of mixture). The liquid adhesion agent (“amine”) was carefully added to the heated bitumen 30 ± 5 minutes before mixing with aggregate. Portions of the mixture was spread on metal lids or silicone paper and stored overnight at ambient temperature before testing. The binder coverage shall be 100 % after mixing.

Three part samples, each weighing 150 g, was transferred to their respective bottles in individual particles or small lumps. Cold distilled water was added to the bottle. A glass rod with a rubber tube was inserted in the bottle and is fixed between the bottom and the screw cap. The bottles were placed on a rolling bottle machine (figure 1), and rotated at a speed of 60 rpm at ambient temperature.

After 6 h, 24 h, 48 h and 72 h rolling time, the machine was stopped and the degree of binder coverage in each bottle was estimated. The average readings of two operators on three part samples were reported. A graphical presentation of the results was made to ease the interpretation of the results.

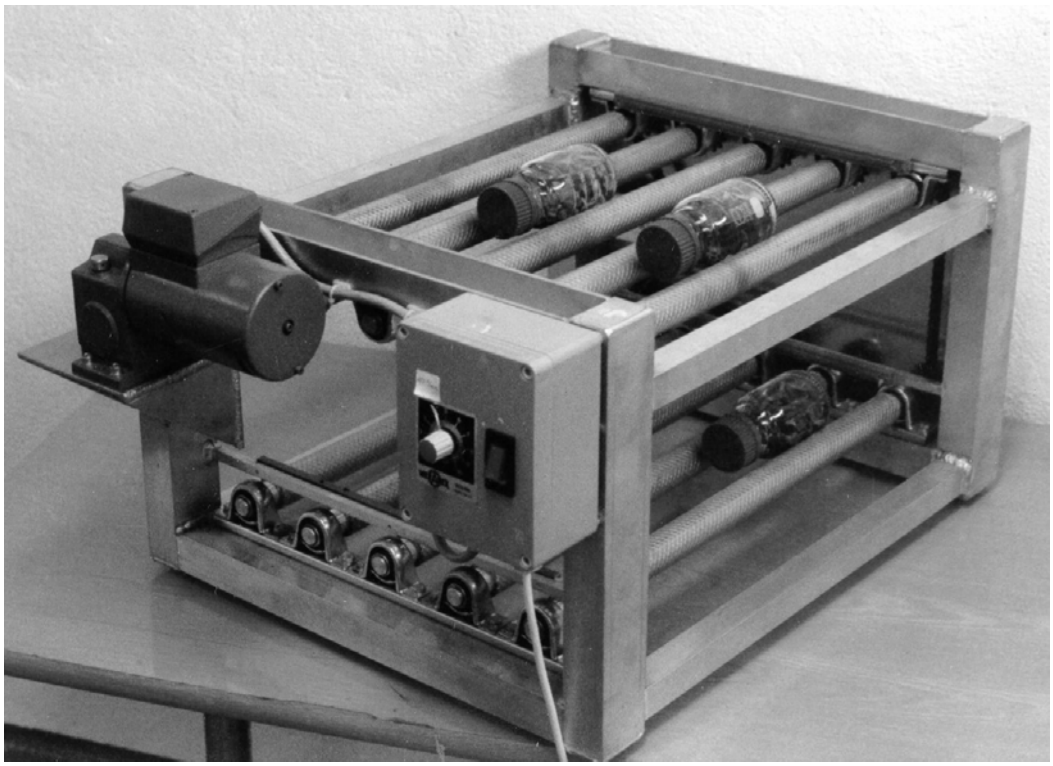

Figure 1. Rolling bottle machine.

3. ROUND ROBIN TEST

Seven laboratories participated in the round robin test on the Boiling Test and the Rolling Bottle Test (Jørgensen 2001). One sample of bitumen 160/220, one sample of “amine” (adhesion agent) and one sample of each of the four aggregates were distributed to the participating laboratories. The adhesion tests were made only on one test sample, i.e. no repeats, due to the time available and the cost for the laboratories. Results from the binder testing are given in table 1. Mineralogy and mechanical properties of the aggregates are given in table 2.

Table 1. Test results on bitumen 160/220

Method	Unit	Results
Penetration at 25 °C	0.1 mm	177
Viscosity at 60 °C	Pa·s	65
Viscosity at 135 °C	mm^2/s	221
Acid number	mgKOH/g	3.8

Table 2. Aggregates used in the round robin test *

Aggregate name	Feiring	Hellvik	Hadeland	Vassfjellet
Mineralogy	Gneiss, medium grain size	Anorthosite, labradorite	Porphyry	Gabbro
Colour of wet aggregate	grey	white, bright	dark auburn	dark grey
Stripping tendency	non-stripping	stripping	stripping	non-stripping
Density, g/cm^3	2.87	2.71	2.60	3.03
Nordic abrasion value	10	7	3.5	8
Polished stone value	53	55	48	50

* data from supplier

The amine used was a mixture of alkylamines and alkylamidoamines/imidazolines. The dosage of amine in bitumen was 0.4 % (m/m).

The operators from the participating laboratories met at the start of the round robin test to discuss how to run the tests and how to estimate the degree of coverage. In the statistical treatment of the test datas, standard formulas were used (Anonymous 1994).

4. RESULTS - BOILING TEST

Table 3 and figure 2 gives an overview of the results on the boiling test.

Table 3. Results from the round robin test on the Boiling Test

	Percent coverage after boiling							
Aggregate	Feiring		Hellvik		Hadeland		Vassfjellet	
Adhesion agent	no	yes	no	yes	no	yes	no	yes
Laboratory no.								
1	39	98	8	97	4	97	80	100
2	10	64	3	38	3	30	38	75
3	19	78	0	34	0	19	45	81
4	18	93	0	90	3	96	68	100
5	10	68	5	20	5	36	43	95
6	0	10	0	13	0	25	73	85
7	0	10	5	15	5	10	65	85
Average, $\bar{x}$	14	60	3	44	3	45	59	89
Std. deviation, s_x	13	36	3	35	2	36	17	10

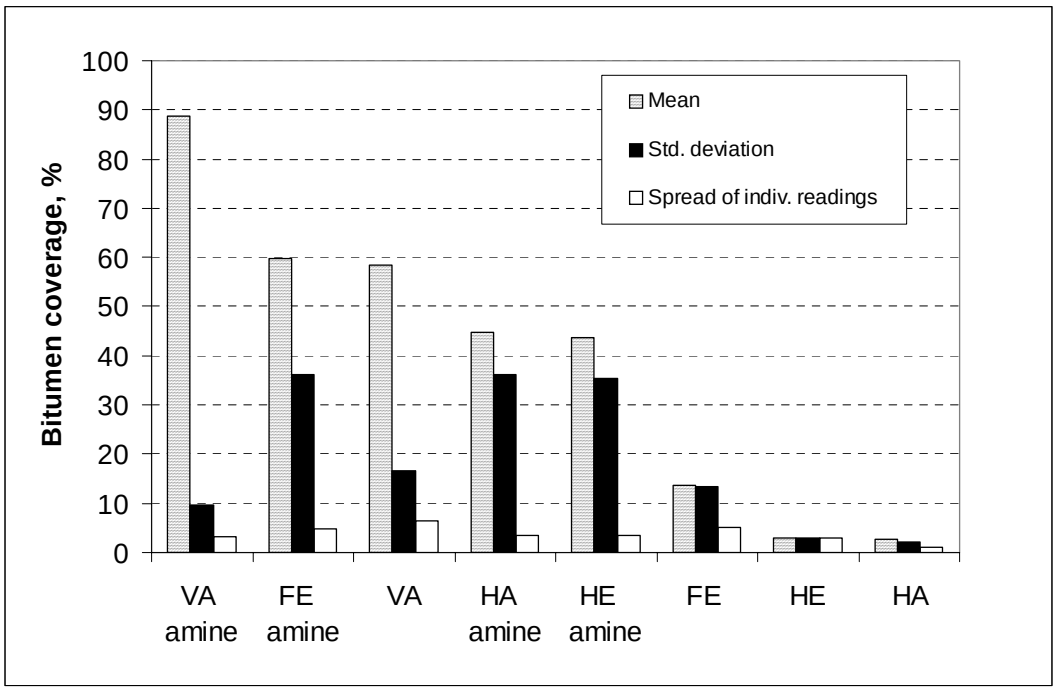

Figure 2. Results from the Boiling Test. (VA = Vassfjellet, FE = Feiring, HE = Hellvik, HA = Hadeland)

5. RESULTS - ROLLING BOTTLE TEST

There were not reported any big problems with aggregate particles forming lumps. Tables 4-7 show the results from the Rolling Bottle Test.

Table 4. Results from the Rolling Bottle Test after 5 h rolling time

	Percent coverage							
Aggregate	Feiring		Hellvik		Hadeland		Vassfjellet	
Adhesion agent	no	yes	no	yes	no	yes	no	yes
Laboratory no.								
1	81	90	61	83	68	89	80	83
2	83	95	35	80	35	93	93	95
3	39	80	22	58	48	93	66	77
4	60	92	51	68	40	81	75	78
5	60	88	23	70	13	85	75	83
6	70	98	53	83	15	83	85	93
7	66	80	29	55	37	83	66	76
Average, $\bar{x}$	66	89	39	71	36	87	77	83
Std. deviation, s_x	15	7	16	11	19	5	10	8

Table 5. Results from the Rolling Bottle Test after 24 h rolling time

	Percent coverage							
Aggregate	Feiring		Hellvik		Hadeland		Vassfjellet	
Adhesion agent	no	yes	no	yes	no	yes	no	yes
Laboratory no.								
1	30	58	5	55	0	64	43	56
2	28	60	5	40	0	53	33	53
3	28	41	3	36	3	54	36	44
4	38	76	5	53	0	58	57	73
5	30	58	3	43	0	68	50	70
6	10	65	0	55	0	48	45	55
7	26	57	0	45	5	47	51	61
Average, $\bar{x}$	27	59	3	47	1	56	45	59
Std. deviation, s_x	8	11	2	8	2	8	9	10

Table 6. Results from the Rolling Bottle Test after 48 h rolling time

	Percent coverage							
Aggregate	Feiring		Hellvik		Hadeland		Vassfjellet	
Adhesion agent	no	yes	no	yes	no	yes	no	yes
Laboratory no.								
1	14	43	*	42	*	46	33	42
2	5	43	*	27	*	40	30	45
3	9	34	*	22	*	38	28	38
4	5	59	*	39	*	21	43	68
5	13	43	*	18	*	38	28	35
6	5	45	*	35	*	25	30	43
7	12	36	*	32	*	32	33	48
Average, $\bar{x}$	9	43	-	31	-	34	32	45
Std. deviation, s_x	4	8	-	9	-	9	5	11

* testing finished

Table 7. Results from the Rolling Bottle Test after 72 h rolling time

	Percent coverage							
Aggregate	Feiring		Hellvik		Hadeland		Vassfjellet	
Adhesion agent	no	yes	no	yes	no	yes	no	yes
Laboratory no.								
1	0	33	*	38	*	38	22	35
2	0	23	*	18	*	23	24	33
3	2	30	*	17	*	30	13	35
4	0	30	*	19	*	5	26	39
5	5	30	*	15	*	28	18	30
6	0	*	*	*	*	10	23	33
7	0	21	*	20	*	18	24	34
Average, $\bar{x}$	1	28	-	21	-	22	21	34
Std. deviation, s_x	2	5	-	8	-	12	4	3

* testing finished

In figure 3 the overall averages for the different aggregates are summarized. The standard deviations for the determinations at different rolling times are shown in figure 4.

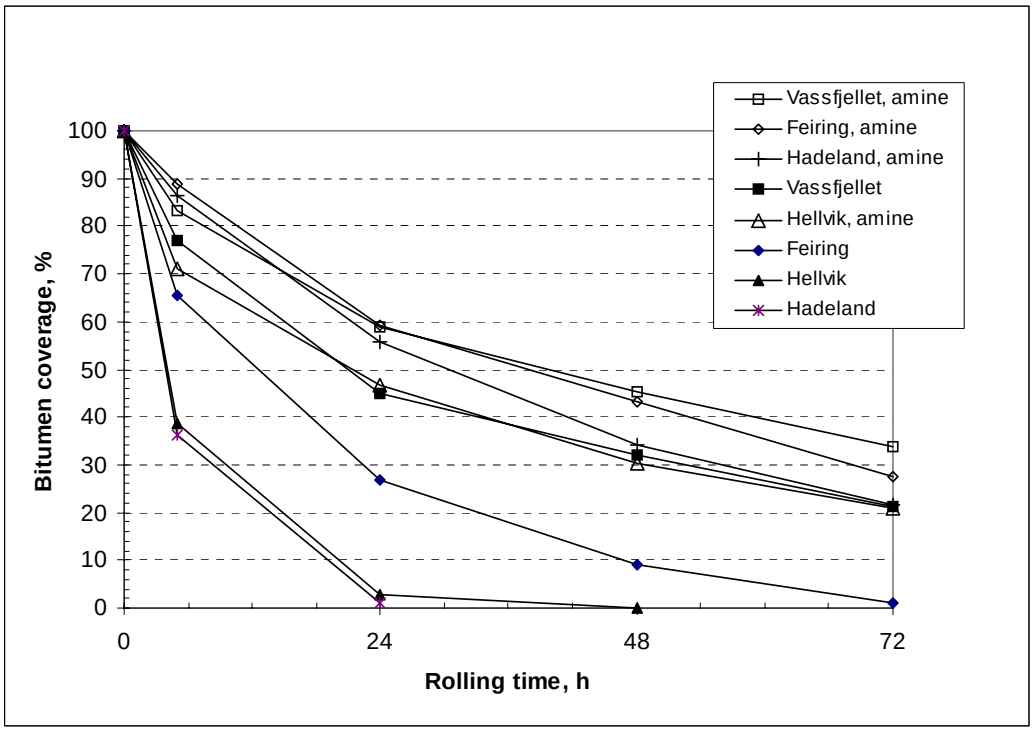

Figure 3. Overall average values for the eight samples in the round robin.

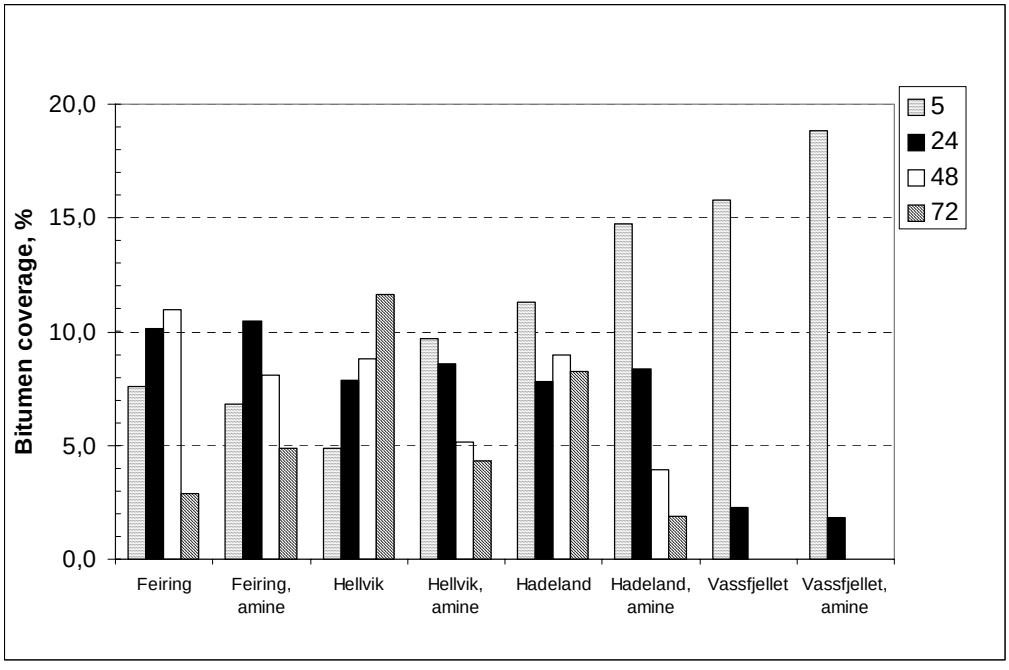

Figure 4. Standard deviations for the eight samples at different rolling times

The determinations after 5 h rolling time are less precise than those of longer rolling times. In the Norwegian practice the 5 h results are seldom used to judge the adhesion. If they are omitted a more realistic estimate of precision can be made. In the plot of averages against standard deviations in figure 5, the 5 h determinations therefore are omitted.

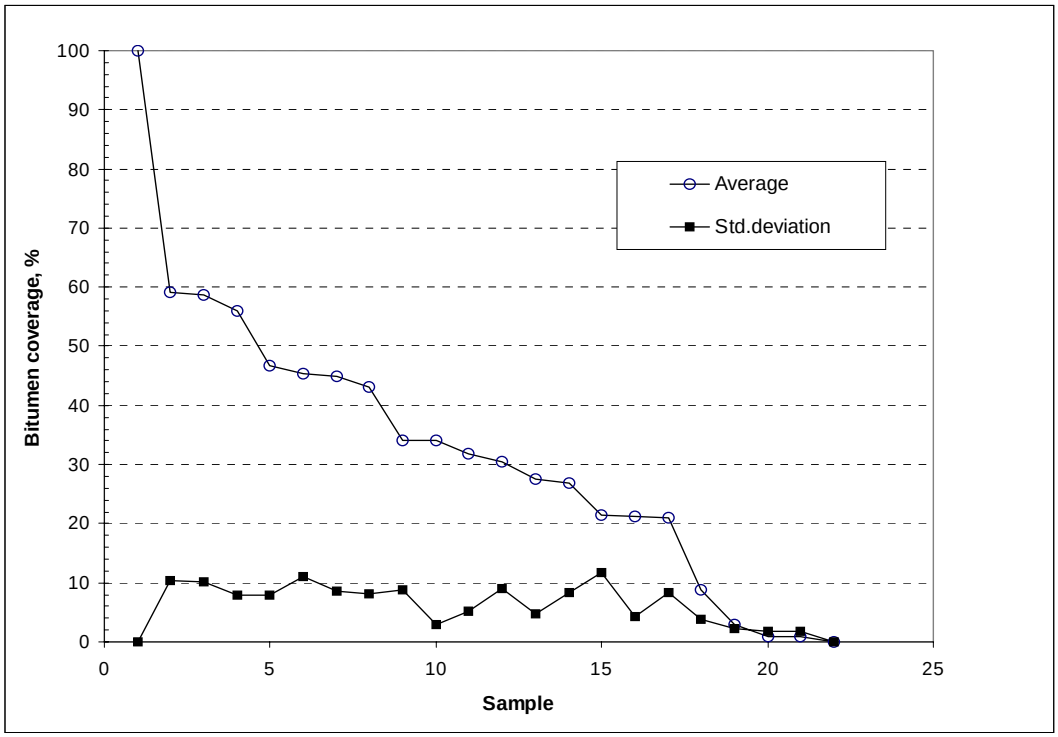

Figure 5. Plot of averages and corresponding standard deviations. The results from 5 h rolling time are omitted

6. CONCLUSIONS

The round robin test was not planned and arranged with the objective to determine the reproducibility and repeatability of the two methods. The standard deviations found are nevertheless indicative for the reproducibility standard deviations of the methods.

Using the same materials and operators in the two test methods, gives a fair opportunity to see which of the two methods that is the best. Both ranked the material combinations almost in the same order and both could point out the two stripping prone aggregates. The effect of adhesion agent was also clearly demonstrated with both tests.

The statistical treatment of the test datas for the Boiling Test concluded that:
- the average standard deviation was 19 %
- at binder coverages less than 20 % and more than 80 % the average standard deviation was 7 %

The recommended use of the Boiling Test is to detect stripping prone aggregates, i,e. a yes/no check. At intermediate coverage values, the precision is poor and the results will be doubtful.

The statistical treatment of the test datas for the Rolling Bottle Test concluded that:
- the average standard deviation was 8 %
- the average standard deviation when the 5 h determinations are omitted was 6 %

The Rolling Bottle Test had best precison for bitumen coverages below 20 % or above 75 %. The results show that certain aggregates gives poorer standard deviation, probably due to difficulties in the visual dermination of binder coverage. In the procedure there is a warning of the risk of underestimating the binder coverage on bright aggregates and overestimating it on dark aggregates.

In the nordic round robin test in 1986 (Isacsson & Jørgensen 1987) the average standard deviation was 15 %. The improved precision in this study probably reflects that the operators are more experienced with the test method. The results in this study suggest a reproducibility of approximatily 15 %. In the drafted european standard prEN 12697-11 the estimate of reproducibilty is 30 %.

The results from the Rolling Bottle Test should allow a division of aggregate-binder mixtures into at least three adhesion classes:
- very poor adhesion
- somewhat poor/acceptable adhesion
- satisfying/good adhesion

In a mix design system such information will give an “early warning” of possible problems and help the designer to consider proper actions in his test program on asphalt mixtures.

Both the Rolling Bottle Test and the Boiling Test may be used in the product control of raw materials.

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Appendix / Vedlegg

Poster presentation at the BCRA ’02

Norwegian Public Roads Administration,
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Testing adhesion between bitumen and aggregate with the Rolling Bottle Test and the Boiling Test

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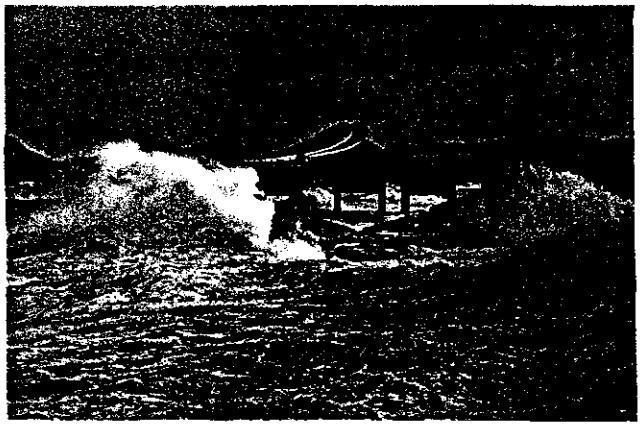


Reasons for testing the asphalt raw materials

Humid and cool climate & use of stripping prone aggregates

- ➔ necessitate use of adhesion agent
- ➔ determine the use and dosage of adhesion agents.

Quality control of aggregates, binders or adhesion agents

Quick and inexpensive methods for the asphalt mix design

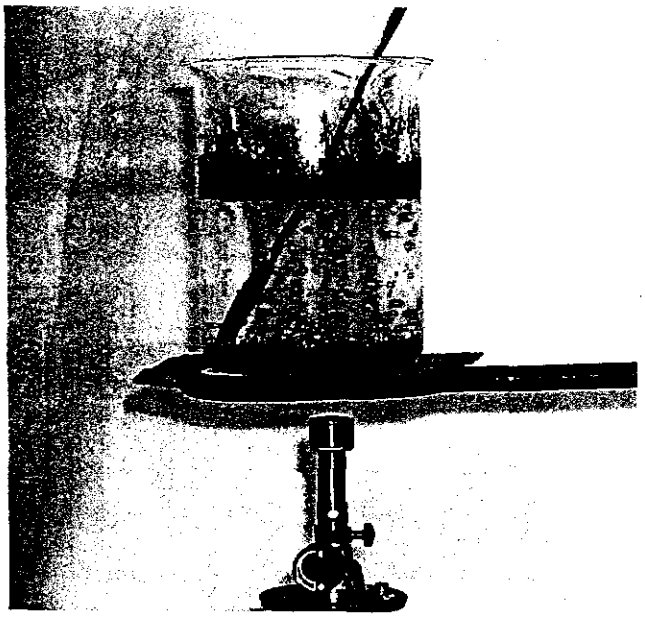
Important to know!

- ▸ the precision of the test methods
- ▸ interpreting the test results – validation by field practice.

Round Robin Test 2001: the Boiling Test & the Rolling Bottle Test

- ▸ 7 laboratories
- ▸ Bitumen 160/220 without and with 0.4 % liquid amine
- ▸ 4 aggregates: 2 non-stripping (FE, VA) and 2 stripping (HA, HE).

The Boiling Test (modified Texas Boiling Test)



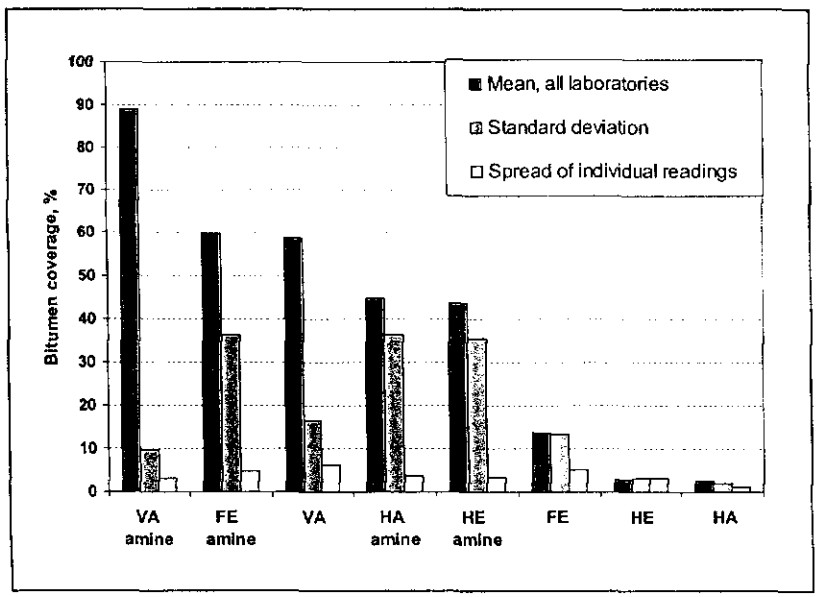
on the total aggregate mixture e.g. 0/11 mm
or on the coarse aggregate e.g. 4/8 mm

100 g mixture is put in boiling water for 10 min and stirred at 3 min intervals

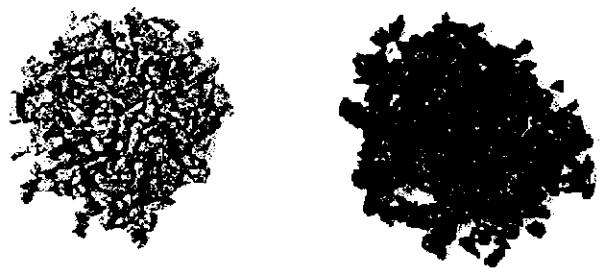
the mixture is cooled in water, decantated and stored on paper cloth overnight

visual assessment of percent binder coverage

Round robin on the Boiling Test



Average values and standard deviations of the round robin.



Material HE (anorthosite) after boiling

Left: without amine Right: with amine

Material HA (porphyry) after boiling

Left: without amine Right: with amine

The Boiling Test – conclusions

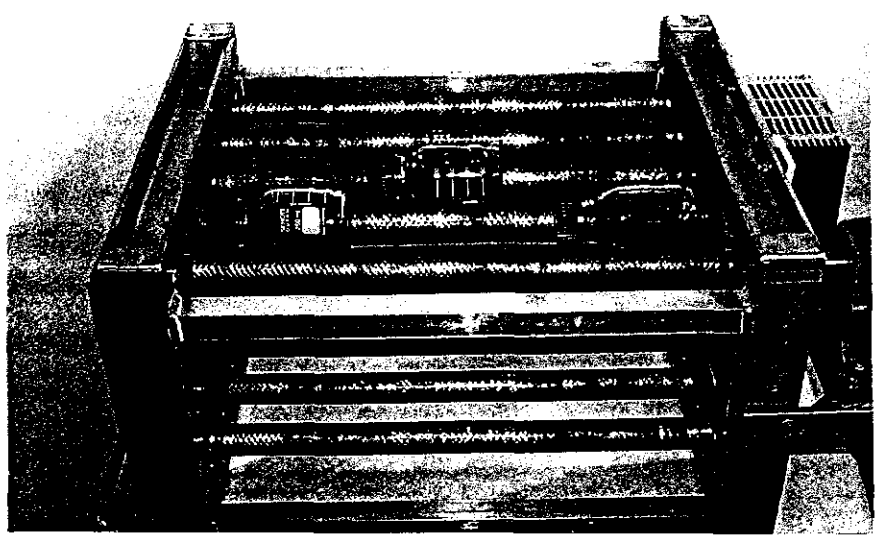
- Average standard deviation: 19 %
- Average standard deviation at binder coverages < 20 % or > 80 %: 7 %
- Can discriminate between stripping and non-stripping aggregates
- Effect of adhesion agent (amine) was demonstrated.

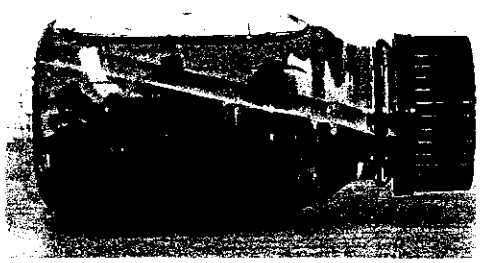
Recommended use:

- Detect stripping aggregates or stripping asphalt mixtures
- Product control of raw materials.

The Rolling Bottle Test (prEN 12697-11)

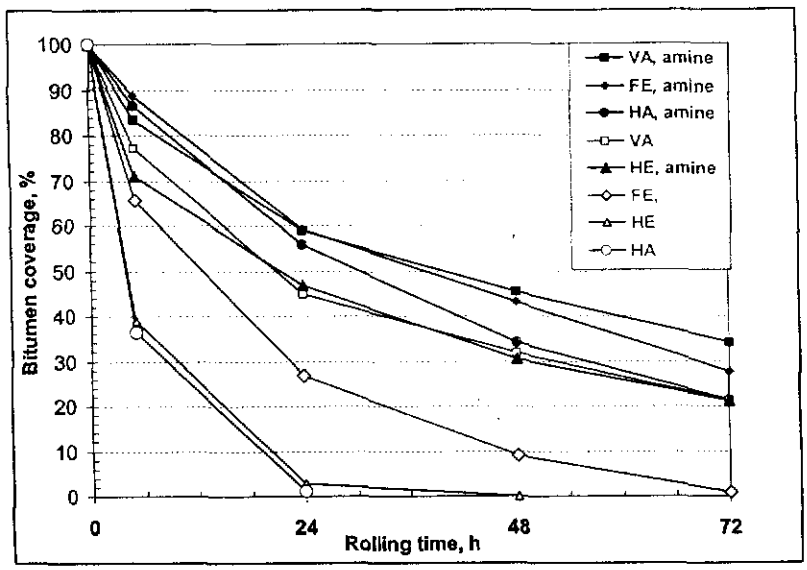
- 5.6/8.0 mm aggregate mixed with 3.4 % bitumen
- 150 g sample in a bottle with a fixed glass rod and distilled water
- bottle rotation speed 60 rpm.



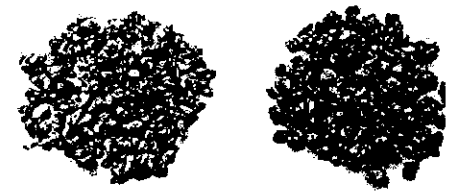


Estimation of binder coverage after 6 h, 24 h, 48 h and 72 h rolling time.

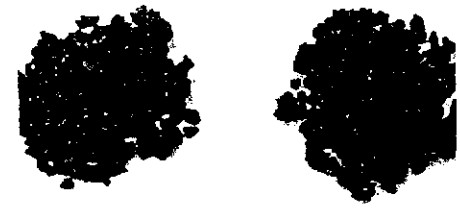
Round robin on the Rolling Bottle Test



Average values of the round robin
HA and HE are stripping prone aggregates.



Material HE (anorthosite)
Left: without amine Right: with amine



Material HA (porphyry)
Left: without amine Right: with amine

The Rolling Bottle Test – conclusions

- Average standard deviation without 5 h readings: 6 %
- Average standard deviation at binder coverages < 20 % or > 75 %: below 6 %
- Three adhesion levels suggested:
  - satisfying/good adhesion
  - between poor and satisfying adhesion
  - very poor adhesion
- Effect of adhesion agent (amine) was demonstrated.

Recommended use:

- Detect stripping binder/aggregate combinations
- Product control of raw materials.