

Author: Auli Kettunen, Neste
Approved by: Helena Remes

29 May 2017

Distribution: Participants

BITUMEN ROUND ROBIN 1-2017

Background

This paving grade bitumen round robin test is performed annually. The participating laboratories are primarily from the Nordic countries representing asphalt contractors, roofing felt factories, research laboratories and bitumen suppliers. The practical arrangements and the reporting have been managed by Neste.

Test materials

The following paving grade bitumens were tested.

Bitumen B50

Bitumen B200

Test methods

The test methods used are given in table 1.

Participating Laboratories

Estonia	Lemminkäinen/Tallinn
	Nynas/Tallinn
	Teede Tehnokeskus
Finland	Aalto University
	Katepal
	Lemminkäinen/Laboratory
	Mitta
	NCC Industry
	Neste /Naantali Refinery
	Neste /Research and Technology
	Nordic Waterproofing
	SGS Inspection Service
Germany	Nynas / Harburg
Norway	Lemminkäinen/Central Laboratory
	Lemminkäinen/Eikefet
	Lemminkäinen/Greåker
	Lemminkäinen/Tønsberg
	Lemminkäinen/ Tromsø
	Petrotest
	Statens vegvesen
	Veidekke Industri
Sweden	Veiteknisk institutt
	Lemminkäinen/Sundsvall
	NCC Industry/ Biskopstorp

Auli Kettunen

29 May 2017

NCC Industry/ Hisings Kärra
NCC Industry/ Södra Sandby
NCC Industry/Umeå
NCC Industry/Upplands Väsby
NCC Industry/Västerås
Nynas/Goteborg Refinery
Nynas/Nynashamn Refinery
Nynas/Nynashamn Quality Control
PEAB Asfalt/Boden
PEAB Asfalt/ Helsingborg
PEAB Asfalt/ Hisings Backa
PEAB Asfalt/Stockholm
Skanska Sverige/Gunnilse
Skanska Sverige/Malmö
Skanska Sverige/Norrköping
Skanska Sverige/Vällsta, Upplands Väsby
Svevia/Jönköping
Svevia/Umeå
Svevia/Örebro
TeMaKon
VTI
UK Nynas/Eastham Quality Control
Nynas/Research and Development
SGS

Results

The primary results from the participating laboratories are given in tables 2 and 3.

Evaluation

Grubb's test has been applied to find out possible outliers in the data set. The test has been used for the highest and the lowest values in each data set as follows.

$$G_{\min} = (x_{\text{ave}} - x_{\min})/s \quad \text{and} \quad G_{\max} = (x_{\max} - x_{\text{ave}})/s$$

If the statistic factor (G) is greater than its 1 % critical value the item is called a statistical outlier and it is indicated by an asterisk in tables 2 and 3. The outliers are omitted in the further evaluation of the data set.

Evaluation of the results has been made against the reproducibility given in the appropriate test methods, R_{stand} . Reproducibility based on the results is calculated according to the formula

$$R_{\text{calc}} = 2,8 * s$$

s = multilaboratory standard deviation from this round robin without outliers.

Auli Kettunen

29 May 2017

Discussion of the results

The results received from the following methods do not fulfil the precision criteria.

- Penetration in both the samples.
- Dynamic viscosity in both the samples.
- Kinematic viscosity in both the samples.
- Breaking point, Fraass in both samples.
- Softening point in both the samples.
- Flash point, COC in both samples.
- RTFOT, viscosity ratio at 60°C in both the samples.

NB. According to the penetration method the result should be rounded to the nearest whole number. (EN 1426)

According to the softening point method the result should be given with the accuracy of 0,2°C when the softening point is less than 80°C. (EN 1427)

According to the RTFOT method the retained penetration should be rounded to the nearest whole number. (EN 12607-1)

Auli Kettunen

29 May 2017

Table 1 Test methods

Property	Standard method
Penetration 25°C	EN 1426
Viscosity 60°C	EN 12596
Viscosity 135°C	EN 12595
Breaking point, Fraass	EN 12593
Softening point	EN 1427
Flash point, COC	EN ISO 2592
RTFOT	EN 12607-1
change of mass	
penetration 25°C	EN 1426
retained penetration	
softening point	EN 1427
change in softening point	
viscosity 60°C	EN 12596
viscosity ratio at 60°C	

Auli Kettunen

29 May 2017

Table 2 Bitumen B50

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	61	58	59	64	62	60	62	63	61	59
Viscosity 60°C	Pas	216	202	211	202	208	219	214	206	241	
Viscosity 135°C	mm ² /s	314	329	331	329	337	343	330	326	336	
Breaking point, Fraass	°C	-12	-15	-12	-22	-16	-14	-14	-21		-19
Softening point	°C	49,2	58,4 *	50,2	49,4	48,2	49,4	48,8	48,8	48,8	49,0
Flash point, COC	°C	310	322		306	314	324	236 *			
RTFOT											
-change of mass	m-%	0,03	0,09	0,02	0,11	0,03	0,07	0,07		0,07	
-penetration 25°C	1/10mm	34		33	37	35	33	37		35	
retained penetration	%	56	54,3	55	58	56	55	60		57	
-softening point	°C	57,4		59,0	56,8	57,0	57,6	56,4		55,7	
change in softening point	°C	8,2	8,4	9,0	7,4	8,8	8,2	7,6		6,9	
-viscosity 60°C	Pas	944			812	989	915	684		855	
viscosity ratio at 60°C		3,0			4,0	4,8	4,2	3,2		3,5	

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	62	60	60	59	62	54	60	58	54	59
Viscosity 60°C	Pas		226	219	207				203		255
Viscosity 135°C	mm ² /s		345		328	339	326	337			370 *
Breaking point, Fraass	°C	-17,2	-14	-15							
Softening point	°C	48,9	48,6	49,8	48,5	48,4	50,4	47,6	49,2	49,8	49,6
Flash point, COC	°C	318		318	300	312					
RTFOT											
-change of mass	m-%	0,01	0,02								
-penetration 25°C	1/10mm	39	34								
retained penetration	%	63	56,7								
-softening point	°C	57,6	55,6								
change in softening point	°C	8,6	7,0								
-viscosity 60°C	Pas										
viscosity ratio at 60°C											

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	60	59	59	58	57	62	64	61	58	59
Softening point	°C	48,8	49,9	48,4	52		50,0	48,2	47,4	49,2	50,4
Breaking point, Fraass	°C						-16				
Flash point, COC	°C	322					340				

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm		58	60	65	58	58,2	63	62,7	60	59,9
Softening point	°C	48,6	50,8	49,2		48,8	49,8	48,6	49,4	51,4	48,8

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25°C	1/10mm	59	55	58	64	54,5	55	60	58		
Softening point	°C	50,6	49,8	47,8	49,6		52	53,6			

*) Outlier

Auli Kettunen

29 May 2017

Table 3 Bitumen B200

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	173		165	178	175	163	173	170	180	167
Viscosity 60°C	Pas	45,8	41	44,8	43,7	44,2	46	47,1	43,1	52	
Viscosity 135°C	mm ² /s	169	175	169	172	168	178	167	170	175	
Breaking point, Fraass	°C	-19	-20	-15	-23	-23	-20	-21	-20		-24
Softening point	°C	40,8		41,4	39,6	38,2	39,8	39,8	39,4	39,2	39,6
Flash point, COC	°C	290	317		305	298	318	228 *			
RTFOT											
-change of mass	m-%	0,03	-0,04	-0,10	0,09	-0,04	0,01	0,03		-0,10	
-penetration 25°C	1/10mm	78		70	79	73	77	79		81	
retained penetration	%	45	44,2	42	44	42	47	46		45	
-softening point	°C	47,0		49,0	47,6	47,2	47,0	46,2		46,8	
change in softening point	°C	6,2	6,8	7,6	8,0	9,0	7,2	6,4		7,6	
-viscosity 60°C	Pas				144	158	137	125		147	
viscosity ratio at 60°C					3,3	3,6	3,0	2,7		2,8	

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	165	184	169	163	165	156	167	164	155	160
Viscosity 60°C	Pas		45,0	44,7	42,1				43,7		54
Viscosity 135°C	mm ² /s		172		171	174	170	171			180
Breaking point, Fraass	°C	-20,1	-20	-23							
Softening point	°C	41,1	40,2	40,0	39,6	38,6	41,2	39,0	39,8	40,4	39,2
Flash point, COC	°C	300		305	294	294					
RTFOT											
-change of mass	m-%	0,05	-0,09								
-penetration 25°C	1/10mm	74	73								
retained penetration	%	45	42,0								
-softening point	°C	47,4	46,6								
change in softening point	°C	6,3	6,4								
-viscosity 60°C	Pas		149								
viscosity ratio at 60°C			3,3								

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	167	175	156	174	166	168	171	164	162	182
Softening point	°C	39,0	40,1	39,0	42,6		41,4	39,0	37,0	40,4	39,4
Breaking point, Fraass	°C						-22				
Flash point, COC	°C	303					332				

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm		162	172	167	164	168	171	189	161	167,9
Softening point	°C	39,2	41,6	40,0		40,2	41,6	39,2	40,4	41,6	38,9

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25°C	1/10mm	164	155	157	162	143,8	163	183	191		
Softening point	°C	40,4	40,4	38,4	40,2		42	44,8 *			

*) Outlier

Auli Kettunen

29 May 2017

Table 4 Evaluation of the results

		n	Average	Standard deviation s	Maximum difference of the results	Reproducibility R in standard	R stand	R calc	Acceptance
Bitumen B50									
Penetration 25 °C	1/10mm	47	60	2,63	11	6% from average	4	8	no
Viscosity 60 °C	Pas	14	216	15,44	53,0	10% from average	21,6	43,7	no
Viscosity 135 °C	mm ² /s	14	332	8,04	31,0	6% from average	19,9	22,7	no *
Breaking point, Fraass	°C	13	-16	3,13	10,0	6	6	9	no
Softening point	°C	43	49,4	1,22	6,2	2,0	2,0	3,5	no *
Flash point COC	°C	11	317	10,60	40,0	17	17	30	no *
RTFOT									
change of mass	m- %	10	0,05	0,03	0,10	0.20 ¹⁾	0,20	0,10	yes
penetration 25 °C	1/10mm	9	35	2,05	6	not given			
retained penetration	%	10	57	2,65	9	10	10	8	yes
softening point	°C	9	57,0	1,06	3,4	not given			
change in softening point	°C	10	8,0	0,74	2,1	4,0	4,0	2,1	yes
viscosity 60 °C	Pas	6	867	109,3	305	not given			
viscosity ratio at 60 °C		6	3,8	0,68	1,8	20% from average	0,8	1,9	no
Bitumen B200									
Penetration 25 °C	1/10mm	46	168	9,24	47	6% from average	10	26	no
Viscosity 60 °C	Pas	14	45,5	3,56	13,0	10% from average	4,6	10,1	no
Viscosity 135 °C	mm ² /s	15	172	3,69	13,0	6% from average	10,3	10,5	no
Breaking point, Fraass	°C	13	-21	2,35	9	6	6	7	no
Softening point	°C	42	40,0	1,13	5,6	2,0	2,0	3,2	no *
Flash point, COC	°C	11	305	12,60	42	17	17	36	no *
RTFOT									
change of mass	m- %	10	-0,02	0,07	0,19	0.20 ¹⁾	0,20	0,19	yes
penetration 25 °C	1/10mm	9	76	3,64	11	not given			
retained penetration	%	10	44	1,75	5	10	10	5	yes
softening point	°C	9	47,2	0,79	2,8	not given			
change in softening point	°C	10	7,2	0,91	2,8	4,0	4,0	2,6	yes
viscosity 60 °C	Pas	6	143	11,29	33,0	not given			
viscosity ratio at 60 °C		6	3,1	0,34	0,9	20% from average	0,6	1,0	no

¹⁾ for 0, 3% <value <0,80%

*) Outliers are excluded in the evaluation.