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Distribution: Participants

BITUMEN ROUND ROBIN 1-2014

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 Oil.

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

Belgium	Nynas/Antwerp
Denmark	NCC Roads Kemi
Estonia	Nynas/Tallinn Teede Tehnokeskus
Finland	Aalto University Destia Icopal Katepal Lemminkäinen/Laboratory NCC Roads Neste Oil/Naantali Refinery Neste Oil/Research and Technology Nordic Waterproofing SGS Inspection Service
Norway	Feiring Asfalt Lemminkäinen/ Fjellhamar Lemminkäinen/Greåker Petrotest Statens vegvesen Veidekke Industri Veiteknisk institutt
Sweden	Lemminkäinen/Sundsvall NCC Roads/ Biskopstorp NCC Roads/ Hisings Kärra

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	NCC Roads/ Sundsvall
	NCC Roads/ Södra Sandby
	NCC Roads/Umeå
	NCC Roads/Upplands Väsby
	NCC Roads/Västerås
	Nynas/Goteborg Refinery
	Nynas/Nynashamn Refinery
	Nynas/Nynashamn Quality Control
	PEAB Asfalt/Boden
	PEAB Asfalt/Hisings Backa
	PEAB Asfalt/Helsingborg
	PEAB Asfalt/Stockholm
	Skanska Sverige/Gunnilse
	Skanska Sverige/Malmö
	Skanska Sverige/Norrköping
	Skanska Sverige/Vällsta, Upplands Väsby
	Svevia/Jönköping
	Svevia/Umeå
	Svevia/Örebro
	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.

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 the sample B50.
- Kinematic viscosity in both the samples.
- Breaking point, Fraass in the sample B200.
- Softening point in the sample B200.
- Flash point, COC in both the samples.
- RTFOT, viscosity ratio at 60°C in both the samples.

NB. 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.

According to the dynamic viscosity method the result should be given with the accuracy of three significant figures. (EN 12596)

According to the kinematic viscosity method the result should be given with the accuracy of three significant figures. (EN 12595)

According to the RTFOT method the change of mass should be given with the accuracy of 0,01 m-%. (EN 12607-1)

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	

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Table 2 Bitumen B50

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	50	58	51	51	56	50	51	53	50	50
Viscosity 60°C	Pas	332	218,7 *	320	335	336,0	324,115	323		320,3	294,1
Viscosity 135°C	mm ² /s	410	426,7	401	409	436,0	413,361	395			395,8
Breaking point,Fraass	°C	-14		-16	-14	-13	-13	-14			-12
Softening point	°C	51,2	50,7	51,8	52,2	51,4	51,8	51,2	51,4	51,6	51,8
Flash point, COC	°C				312	335	326	326			322
RTFOT											
-change of mass	m-%	0,07		0,07	0,09	0,10	0,05	0,14			0,01
-penetration 25°C	1/10mm	29		31	30	37	29	33			31
retained penetration	%	58,0		61	59	66,1	58	65			62
-softening point	°C	59,2		59,2	59,0	59,4	60,0	58,8			
change in softening point	°C	8,0		7,4	6,8	8	8,2	7,6			7,7
-viscosity 60°C	Pas	1357		1291	1329	1494,0					
viscosity ratio at 60°C		4,09		4,0	4,0	4,4					
Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	55	52	51	52	54	53	53	53	50	53
Viscosity 60°C	Pas		328	306		323	323	326	339	444 *	318
Viscosity 135°C	mm ² /s			444		403	419	408	394	447	400
Breaking point,Fraass	°C	-14,5	-15	-15		-21 *	-16	-12	-15		
Softening point	°C	51,5	51,8	52,2	51,4	51,6	51,4	51,4	51,0	52,2	51,5
Flash point, COC	°C		314	342	321	322	317	318			
RTFOT											
-change of mass	m-%	-0,04		0,17		0,09	0,06	0,05	0,08	0,089	0,07
-penetration 25°C	1/10mm	35		30		32	32	31	30	30	34
retained penetration	%	63,64		59		59	60	58	56,9	60,0	64
-softening point	°C	60,5		59,8		59,2	59,0	59,2	59,2	61,8	59,1
change in softening point	°C	9,0		8,0		7,6	7,6	7,8	8,3	9,6	7,6
-viscosity 60°C	Pas			1220		1221	1490	1336	1437		1115
viscosity ratio at 60°C				4,0		3,8	4,6	4,10	4,2		3,5
Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	47	52	49,4	52	48	50	50	47	48	52
Viscosity 135°C	mm ² /s	414	407								
Softening point	°C	52,0	50,4	52,0	51,4	53,0	50,6	51,2	51,6	50,8	50,8
Breaking point,Fraass	°C				-13						
Flash point, COC	°C										
Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm		52	53	52	50	51	52	52	54	45
Softening point	°C	60,0 *	50,8		54,8 *	51,7	52,2	51,8	51,4	52,4	53,6
Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25°C	1/10mm	50,3	50	44	49	51		50			
Softening point	°C	55,2 *	51,4	51,4	53,2	53,2	51,2				

*) Outlier

Table 3 Bitumen B200

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	168	188	178	180	189	169	176	189	175	174
Viscosity 60°C	Pas	42,3	29,0 *	42	42	45,0 *	41,247	42,2		40,7	41,1
Viscosity 135°C	mm ² /s	170	172,3	171	165	173,0	171,136	163			167,2
Breaking point,Fraass	°C	-20		-18	-14	-19	-16				-18
Softening point	°C	39,4	38,7	39,8	39,2	39,2	39,8	37,4	40,2	39,4	40,4
Flash point, COC	°C				308	334	316	308			326
RTFOT											
-change of mass	m-%	-0,08		-0,07	-0,04	-0,06	-0,15	-0,02			-0,12
-penetration 25°C	1/10mm	71		83	75	81	78	81			77
retained penetration	%	42,3		47	45	42,9	46	46			44
-softening point	°C	47,6		46,6	46,6	46,4	47,8	46,2			
change in softening point	°C	8,2		6,8	7,4	5,6	8,0	8,8			7,3
-viscosity 60°C	Pas	162		123	122	135,0					
viscosity ratio at 60°C		3,8		2,9	2,9	3					

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	180	180	178	176	194	182	181	184	170	180
Viscosity 60°C	Pas		41,2	40,4		41	41,5	41,4	43	56,6 *	39
Viscosity 135°C	mm ² /s			180		166	178	165	166	180	165
Breaking point,Fraass	°C	-19	-22	-21		-26	-25	-18	-22		
Softening point	°C	39,8	39,0	40	39,0	39,4	39,0	39,4	39,2	39,6	39,1
Flash point, COC	°C		304	320	308	306	313	306			
RTFOT											
-change of mass	m-%	-0,05				-0,06	-0,09	-0,05	-0,11	-0,041	-0,04
-penetration 25°C	1/10mm	81		82		86	77	86	76	71	83
retained penetration	%	45,00		46		44	42	48	41,5	41,8	46
-softening point	°C	46,5		48,8		46	46,8	45,6	46,6	48,0	45,5
change in softening point	°C	6,7		8,8		6,6	7,8	6,2	7,4	8,4	6,4
-viscosity 60°C	Pas			148		120	145	120	141		125
viscosity ratio at 60°C				3,7		2,9	3,5	2,90	3,3		

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	170	186		177	176	176	158	151	168	173
Viscosity 135°C	mm ² /s	170	169								
Softening point	°C	40,0	37,8		39,2	41,0	38,0	39,0	40,0	39,2	39,4
Breaking point,Fraass	°C				-22						
Flash point, COC	°C										

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm		178	183	197	163	179	172	179	186	155
Softening point	°C	39,8	38,8		42,4	40,3	38,6	40,8	39,4	40,8	41,2

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25°C	1/10mm	175,4	181	161	173	177		166			
Softening point	°C	42	39,4	41,2	41,4	41,4	38,6				

*) Outlier

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	45	51	2,58	14	6% from average	3	7	no
Viscosity 60 °C	Pas	15	323	11,50	44,9	10% from average	32,3	32,5	no *
Viscosity 135 °C	mm ² /s	17	413	16,49	53,0	6% from average	24,8	46,7	no
Breaking point, Fraass	°C	14	-14	1,31	4,0	6	6	4	yes *
Softening point	°C	42	51,6	0,70	3,2	2,0	2,0	2,0	yes *
Flash point COC	°C	11	323	8,90	30	17	17	25	no
RTFOT									
change of mass	m- %	15	0,07	0,05	0,21	0.20 ¹⁾	0,20	0,14	yes
penetration 25 °C	1/10mm	15	32	2,29	8	not given			
retained penetration	%	15	61	2,86	9	10	10	8	yes
softening point	°C	14	59,5	0,80	3,0	not given			
change in softening point	°C	15	7,9	0,67	2,8	4,0	4,0	1,9	yes
viscosity 60 °C	Pas	10	1329	123,12	379	not given			
viscosity ratio at 60 °C		10	4,1	0,30	1,1	20% from average	0,8	0,9	no
Bitumen B200									
Penetration 25 °C	1/10mm	44	176	9,54	46	6% from average	11	27	no
Viscosity 60 °C	Pas	14	41,4	0,97	4,0	10% from average	4,1	2,8	yes *
Viscosity 135 °C	mm ² /s	17	170	5,28	17,0	6% from average	10,2	14,9	no
Breaking point, Fraass	°C	14	-20	3,28	12	6	6	9	no
Softening point	°C	44	39,7	1,06	5,0	2,0	2,0	3,0	no
Flash point, COC	°C	11	314	9,59	30	17	17	27	no
RTFOT									
change of mass	m- %	14	-0,07	0,04	0,13	0.20 ¹⁾	0,20	0,10	yes
penetration 25 °C	1/10mm	15	79	4,72	15	not given			
retained penetration	%	15	45	2,04	7	10	10	6	yes
softening point	°C	14	46,8	0,94	3,3	not given			
change in softening point	°C	15	7,4	0,97	3,2	4,0	4,0	2,8	yes
viscosity 60 °C	Pas	10	134	14,49	42,0	not given			
viscosity ratio at 60 °C		9	3,2	0,37	0,9	20% from average	0,6	1,1	no

¹⁾ for 0, 3% <value <0,80%

^{*)} Outliers are excluded in the evaluation.