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Distribution: Participants

BITUMEN ROUND ROBIN 1-2008

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 B70

Bitumen B200

Test methods

The test methods used are given in table 1.

Participating Laboratories

Belgium	Nynas/Antwerp
Denmark	NCC Kemi Danish Road Institute
Estonia	Technical Center of Estonian Roads
Finland	Icopal Katepal Lemminkäinen/Factory Lemminkäinen/Laboratory NCC Roads Neste Oil/Naantali Refinery Neste Oil/Research and Technology SGS TKK
Norway	Lemminkäinen Norge AS Petrotest SINTEF
Sweden	KTH, Vägteknik Nynas/Göteborg Refinery Nynas/Nynäshamn Nynas/Nynäshamn Refinery PEAB/Göteborg PEAB/Helsingborg PEAB/Kumla PEAB/Stockholm

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	Skanska Sverige/Nord
	Skanska Sverige/Syd
	Skanska Sverige/Väst
	VTI
	Väglaboratoriet
	Vägverket/Jönköping
	Vägverket/Umeå
	Vägverket/Örebro
UK	Eastham Refinery
	Nynas/Dundee
	Nynas/Eastham
	Nynas/Research and Development

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.

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Discussion of the results

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

- o Penetration in both the samples.
- o Dynamic viscosity in both the samples.
- o Kinematic viscosity in both the samples.
- o Breaking point, Fraass in the sample B200.
- o Softening point in both the samples
- o Flash point, COC in both the samples.
- o RTFOT, retained penetration in the sample B200.
- o RTFOT, change in softening point in the sample B200.
- o RTFOT, viscosity ratio at 60° C in both the samples.

Deviations in the test results have become larger compared to the last year's round robin. The laboratories having the largest differences from the average should take actions to check their test procedures.

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)

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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 B70

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	65,8	62	64	65	62	80 *	62,3	64	59	52
Viscosity 60°C	Pas					183		200,3			
Viscosity 135°C	mm2/s		342		343	334		341,27			
Breaking point, Fraass	°C					-19		-16			
Softening point	°C	49,4	49,2	49,0	49,2	48,4	49,6	47,4	49,0	50,0	48,1
Flash point, COC	°C				320	320		326			
RTFOT											
-change of mass	m-%				0,09	-0,04	0,03	0,0174			
-penetration 25°C	1/10mm				42	33	33	33,9			
retained penetration	%				65	53,2	41 *	54,7			
-softening point	°C				53	56,6	58,8	56,2			
change in softening point	°C				3,8	8,2	9,2	8,9			
-viscosity 60°C	Pas					722		896,25			
viscosity ratio at 60°C						3,9		4,45			

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	57	61	64	64	67	64	66	61	62	66
Viscosity 60°C	Pas	204	210	192	189	185	195	196			191
Viscosity 135°C	mm2/s	367	424 *	360	338	338	358	348		342	348
Breaking point, Fraass	°C	-16	-15	-18	-15	-15					-12
Softening point	°C	48,4	49,6	48,4	48,6	48,8	49,4	49,2	49,0	48,4	49,1
Flash point, COC	°C		336	319	294	332					317
RTFOT											
-change of mass	m-%	0,01	0,005	-0,02	0,00	0,01	-0,04	-0,01			-0,07
-penetration 25°C	1/10mm	35	34	38	37	41	37	36			41
retained penetration	%	61,4	55,74	60	57,8	61	58	54,5			62
-softening point	°C	55,2	57,2	56,2	55,6	55,6	56,2	56,1			55,3
change in softening point	°C	6,8	7,6	7,8	7,0	6,8	6,8	6,9			6,2
-viscosity 60°C	Pas	709	901	684	682	545	578	798			626
viscosity ratio at 60°C		3,48	4,3	3,6	3,6	2,95	3,0	4,1			3,3

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	60	64	67	64	70	62	68	63	62	61
Viscosity 60°C	Pas				188	185	170	204		209	
Viscosity 135°C	mm2/s	317,77		360		352	348	357		372	
Breaking point, Fraass	°C					-12	-18			-13	
Softening point	°C	51,4	48,8	50,8	50,2	49,0	48,2	48,9	50,2	49,4	50,0
Flash point, COC	°C		312			306				340	
RTFOT											
-change of mass	m-%					0,07				0	
-penetration 25°C	1/10mm					41				38	
retained penetration	%					59				61	
-softening point	°C					56,0				56,2	
change in softening point	°C					7,0				6,8	
-viscosity 60°C	Pas					672					
viscosity ratio at 60°C						3,63					

Laboratory		31	32	33	34	35	36				
Penetration 25°C	1/10mm	60	59	59	57	63	62				
Viscosity 60°C	Pas					187	215,12				
Viscosity 135°C	mm2/s				381	360	355,7				
Breaking point, Fraass	°C					-16					
Softening point	°C	50,2	50,4	49,6	49,3	49,4	49,9				
Flash point, COC	°C					324	324				
RTFOT											
-change of mass	m-%				-0,018	-0,01	-0,02				
-penetration 25°C	1/10mm				30	36	36				
retained penetration	%				53	57	58				
-softening point	°C				56,4	55,4	58,1				
change in softening point	°C				7,1	6	8,2				
-viscosity 60°C	Pas						802				
viscosity ratio at 60°C							3,73				

*) Outlier

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Table 3 Bitumen B200

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	183	172	184	185	166	165	173	181	162	163
Viscosity 60°C	Pas					36,5		40,48			
Viscosity 135°C	mm ² /s		169		212 *	168		168			
Breaking point, Fraass	°C					-21		-18			
Softening point	°C	40,0	38,6	39,2	38,2	39,2	40,6	38,2	38,8	40,0	38,2
Flash point, COC	°C				316	332		256 *			
RTFOT											
-change of mass	m-%				0,06	0,02	0,02	0,013			
-penetration 25°C	1/10mm				101	81	72,6	83,3			
retained penetration	%				55	48,8	44	49			
-softening point	°C				44,2	46,2	46,8	43,8			
change in softening point	°C				6,0	7,0	6,2	5,6			
-viscosity 60°C	Pas					105		95,9			
viscosity ratio at 60°C						2,9		2,37			

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	165	172	176	182	176	188	181	184	177	181
Viscosity 60°C	Pas	52 *	41,9	38,2	37,8	37	39	39			38,8
Viscosity 135°C	mm ² /s	173	185	181	164	168	166	169		168	171
Breaking point, Fraass	°C	-18	-14	-19	-19	-19,5					-17
Softening point	°C	37,4	39,0	39,2	38,8	39,4	40,1	39,2	39,7	39,8	38,4
Flash point, COC	°C		336	319	>320	328					318
RTFOT											
-change of mass	m-%	0,00	0,018	0,00	0,02	0,02	-0,01	0,01			-0,015
-penetration 25°C	1/10mm	87	84	88	87	96	90	82			100
retained penetration	%	52,7	48,84	50	48	55	48	45,3			55
-softening point	°C	44,4	43,4	46,0	45,0	45,4	45,5	46,3			43,5
change in softening point	°C	7,0	4,4	6,8	6,2	6,0	5,4	7,1			5,1
-viscosity 60°C	Pas	101		97,7	92,5	100	93	108			102
viscosity ratio at 60°C		1,94		2,6	2,4	2,7	2,4	2,8			2,6

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	176	178	196	181	185	174	194	180	170	158
Viscosity 60°C	Pas				35,7	38,8	38,2	41		40	
Viscosity 135°C	mm ² /s	158,19		175		173	196	175		169	
Breaking point, Fraass	°C					-18	-20			-21	
Softening point	°C	41,8	40,0	41,8	40,0	40,2	38,0	40,8	40,2	39,6	41,2
Flash point, COC	°C		322			316				344	
RTFOT											
-change of mass	m-%					0,03				0,02	
-penetration 25°C	1/10mm					86				85	
retained penetration	%					46				50	
-softening point	°C					46,2				45,5	
change in softening point	°C					6,0				5,9	
-viscosity 60°C	Pas					102					
viscosity ratio at 60°C						2,64					

Laboratory		31	32	33	34	35	36				
Penetration 25°C	1/10mm	189	162	160	176	178	160				
Viscosity 60°C	Pas					37,8	37,55				
Viscosity 135°C	mm ² /s				178	173	189,8				
Breaking point, Fraass	°C					-24					
Softening point	°C	40,2	41,2	38,8	38,6	39,8	39				
Flash point, COC	°C					334	338				
RTFOT											
-change of mass	m-%				0,0162	0,01	0,04				
-penetration 25°C	1/10mm				72	87	76				
retained penetration	%				41	49	48				
-softening point	°C				45,9	45,6	46,6				
change in softening point	°C				7,3	5,8	7,6				
-viscosity 60°C	Pas						124,7				
viscosity ratio at 60°C							3,32				

*) Outlier

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Table 4 Evaluation of the results

		n	Average	Standard deviation	Maximum difference of the results	Standard method	Reproducibility R in standard	Rstand	Rcalc	Acceptance
Bitumen B70										
Penetration 25 °C	1/10mm	35	63	3,5	18	EN 1426	6 % from average	4	10	no *
Viscosity 60 °C	Pas	17	194	11,6	45	EN 12596	10 % from average	19,4	32,8	no
Viscosity 135 °C	mm ² /s	21	351	14,2	63	EN 12595	6 % from average	21,0	40,1	no *
Breaking point FRAASS	°C	12	-15	2,3	7	EN 12593	6	6	6	yes
Softening point	°C	36	49,3	0,8	4,0	EN 1427	2,0	2,0	2,3	no
Flash point COC	°C	13	321	12,3	46	EN ISO 2592	17	17	35	no
RTFOT										
change of mass	m-%	17	0,00	0,0	0,16	EN 12607-1	0,20 ¹⁾	0,20	0,11	(yes)
penetration 25 °C	1/10mm	17	37	3,3	12	EN 12607-1	not given			
retained penetration	%	16	58	3,4	12	EN 12607-1	10	10	10	yes *
softening point	°C	17	56,0	1,2	5,8	EN 12607-1	not given			
change in softening point	°C	17	7,1	1,2	5,4	EN 12607-1	4,0	4,0	3,5	yes
viscosity 60 °C	Pas	12	718	113	356	EN 12607-1	not given			
viscosity ratio at 60 °C		12	3,7	0,5	1,5	EN 12607-1	20 % from average	0,7	1,3	no
Bitumen B200										
Penetration 25 °C	1/10mm	36	176	9,8	38	EN 1426	6 % from average	11	28	no
Viscosity 60 °C	Pas	16	38,6	1,6	6,2	EN 12596	10 % from average	3,9	4,7	no *
Viscosity 135 °C	mm ² /s	21	173	8,8	38	EN 12595	6 % from average	10,4	24,9	no *
Breaking point FRAASS	°C	12	-19	2,5	10	EN 12593	6	6	7	no
Softening point	°C	36	39,5	1,1	4,4	EN 1427	2,0	2,0	3,0	no
Flash point COC	°C	11	328	9,9	28	EN ISO 2592	17	17	28	no *
RTFOT										
change of mass	m-%	17	0,02	0,02	0,08	EN 12607-1	0,20 ¹⁾	0,20	0,05	(yes)
penetration 25 °C	1/10mm	17	86	8,2	29	EN 12607-1	not given			
retained penetration	%	17	49	3,9	14	EN 12607-1	10	10	11	no
softening point	°C	17	45,3	1,1	3,4	EN 12607-1	not given			
change in softening point	°C	17	6,2	0,8	3,2	EN 12607-1	2,0	2,0	2,4	no
viscosity 60 °C	Pas	11	102	8,9	32	EN 12607-1	not given			
viscosity ratio at 60 °C		11	2,6	0,4	1,4	EN 12607-1	20 % from average	0,5	1,0	no

¹⁾ for 0,3 % < value < 0,80 %

*) Outliers are excluded in the evaluation.