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Distribution: Participants

BITUMEN ROUND ROBIN 1-2009

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 Neste Oil/Naantali Refinery Neste Oil/Research and Technology SGS TKK
Norway	Lemminkäinen Norge AS Petrotest SINTEF Skanska Asfalt AS
Sweden	Asfalt och Stenkontroll AB/Hok NCC Roads/ Biskopstorp NCC Roads/ Göteborg NCC Roads/ Sundsvall NCC Roads/ Södra Sandby Nynas/Göteborg Refinery Nynas/Nynäshamn Nynas/Nynäshamn Refinery

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	PEAB/Göteborg
	PEAB/Helsingborg
	PEAB/ Stockholm
	PEAB/Västerås
	Skanska Sverige/Nord
	Skanska Sverige/Syd
	Skanska Sverige/Väst
	Svevia/ Jönköping
	Svevia/ Norrköping
	Svevia/ Umeå
	Svevia/ Örebro
	VTI
	Väglaboratoriet i Norr AB
UK	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 both the samples.
- o Softening point in both the samples
- o Flash point, COC in both the samples.
- o RTFOT, viscosity ratio at 60 °C in the sample B70.

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	54	58	54	55	57	61	58	51	57	55
Viscosity 60°C	Pas	202	207	221	198	203	200	223	212	236	207
Viscosity 135°C	mm ² /s	348	341	370	339	342	355	334	368	438 *	347
Breaking point, Fraass	°C	-12	-10	-13	-15				-17	-14	-14
Softening point	°C	48,2	50,0	50,2	49,6	50,0	49,6	49,6	50,8	50,0	48,8
Flash point, COC	°C		320	324	340	324			322	330	
RTFOT											
-change of mass	m-%	0,05	0,05	0,04	0,03	0,09	0,01	0,00	0,02	0,07	
-penetration 25°C	1/10mm	32	36	32	33		33	34	35	34	
retained penetration	%	59,3	62	59	60,0		54	58,6	68,6	60	
-softening point	°C	55,2	56,2	56,8	56		57,7	56,6	57,6	57,0	
change in softening point	°C	7,0	6,2	6,6	6,4		8,1	7,0	6,8	7,0	
-viscosity 60°C	Pas	713	773	791	646		769	814		905	
viscosity ratio at 60°C		3,5	3,73	3,6	3,3		3,8	3,6		3,8	

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	51	59	54	63	57	58	57	56	57,5	59
Viscosity 60°C	Pas		215	204		206	202	201,7			
Viscosity 135°C	mm ² /s	392	343	343		343	351	362,5			
Breaking point, Fraass	°C			-11		-18	-18				
Softening point	°C	49,7	49,4	49,8	50,6	49,0	49,4	49,6	49,5	50,2	49,4
Flash point, COC	°C		318	338		324	317	330			
RTFOT											
-change of mass	m-%	0,0426		0,09	-0,02	0,04	0,04	0,064			
-penetration 25°C	1/10mm	25 *		35	35	34	35	34			
retained penetration	%	49 **		65	56,6	60	60	59,6			
-softening point	°C	56,6		57,4	58,6	55,6	56,0	56,7			
change in softening point	°C	6,9		7,6	8,0	6,6	6,6	7,1			
-viscosity 60°C	Pas			877		744	692	797,7			
viscosity ratio at 60°C				4,3		3,6	3,4	4,0			

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	58	59	59	54	61	60	50	57	61	58
Viscosity 60°C	Pas		194								
Viscosity 135°C	mm ² /s			348	367					342	
Breaking point, Fraass	°C										
Softening point	°C	50,0	49,9	49,2	51,8	49,6	50,0	50,6	50,0	49,4	50,0
Flash point, COC	°C										
RTFOT											
-change of mass	m-%										
-penetration 25°C	1/10mm										
retained penetration	%										
-softening point	°C										
change in softening point	°C										
-viscosity 60°C	Pas										
viscosity ratio at 60°C											

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm	57	51	53	55	50	55	55	44,1	54,3	68
Viscosity 60°C	Pas										
Viscosity 135°C	mm ² /s										
Breaking point, Fraass	°C										
Softening point	°C	49,2	50,1	51,4	49,2	50,0	51,6	50,6	48,8		51,5
Flash point, COC	°C										
RTFOT											
-change of mass	m-%										
-penetration 25°C	1/10mm										
retained penetration	%										
-softening point	°C										
change in softening point	°C										
-viscosity 60°C	Pas										
viscosity ratio at 60°C											

*) Outlier

**) The result is omitted from the evaluation because of the RTFOT penetration 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	169	183	164	175	179	188	180	157	180	173
Viscosity 60°C	Pas	39	39,1	41	36,7	38,5	39	40,5	38,1	42,9	39
Viscosity 135°C	mm ² /s	173	171	181	168,8	165	181	167	178	209 *	171
Breaking point,Fraass	°C	-19	-19	-15	-22				-22	-14	-17
Softening point	°C	38,2	40,2	40,0	39,4	39,0	40,5	39,2	40,4	39,0	38,4
Flash point, COC	°C		320	308	338	328			326	302	
RTFOT											
-change of mass	m-%	0,03	0,01	0,02	0,19 *		0,00	0,00	0,02	0,03	
-penetration 25°C	1/10mm	80	91	82	78		89	86		84	
retained penetration	%	47,3	50	50	44,6		47	47,8		47	
-softening point	°C	44,8	45,8	46,2	46		46,4	45,8	45,4	45,8	
change in softening point	°C	6,6	5,6	6,2	6,6		5,9	6,6	5,0	6,8	
-viscosity 60°C	Pas	103	99,0	105	98,1		101	106		118	
viscosity ratio at 60°C		2,6	2,53	2,6	2,7		2,6	2,6		2,8	

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	177	176	155	193	189	184	190	184	170,2	184
Viscosity 60°C	Pas		41,6	40		38,5	39,0	39,1			
Viscosity 135°C	mm ² /s	182	171	175		169	171	178,6			
Breaking point,Fraass	°C			-20		-21	-21				
Softening point	°C	38,8	39,6	40,3	40,4	38,6	38,8	38,8	39,7	40,6	39,8
Flash point, COC	°C		326	338		330	320				
RTFOT											
-change of mass	m-%	0,017		0,04	-0,05	-0,01	-0,01	-0,008			
-penetration 25°C	1/10mm	72		81	88	89	84	85			
retained penetration	%	41		52	46	47	46	44,7			
-softening point	°C	44,7		46,5	49,4 *	45,0	45,8	45,8			
change in softening point	°C	5,9		6,2	9 **	6,4	7,0	7,0			
-viscosity 60°C	Pas			105		96,0	101	110,8			
viscosity ratio at 60°C				2,6		2,5	2,6	2,86			

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	180	184	182	183	177	178	162	174	180	179
Viscosity 60°C	Pas		37,3								
Viscosity 135°C	mm ² /s			169	187					170	
Breaking point,Fraass	°C										
Softening point	°C	39,0	40,2	41,2	42,4	38,8	39,6	39,8	39,2	38,8	39,4
Flash point, COC	°C										
RTFOT											
-change of mass	m-%										
-penetration 25°C	1/10mm										
retained penetration	%										
-softening point	°C										
change in softening point	°C										
-viscosity 60°C	Pas										
viscosity ratio at 60°C											

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm	177	173	162	158	162	164	170	155	164,3	195
Viscosity 60°C	Pas										
Viscosity 135°C	mm ² /s										
Breaking point,Fraass	°C										
Softening point	°C	38,4	40,0	41,0	39,4	39,4	40,4	40,2	38,0		40,5
Flash point, COC	°C										
RTFOT											
-change of mass	m-%										
-penetration 25°C	1/10mm										
retained penetration	%										
-softening point	°C										
change in softening point	°C										
-viscosity 60°C	Pas										
viscosity ratio at 60°C											

*) Outlier

**) The result is omitted from the evaluation because of the RTFOT softening point 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	40	56	4,1	24	EN 1426	6 % from average	3	12	no
Viscosity 60 °C	Pas	16	208	10,8	42,0	EN 12596	10 % from average	20,8	30,7	no
Viscosity 135 °C	mm ² /s	18	352	14,6	58,0	EN 12595	6 % from average	21,1	41,4	no *
Breaking point FRAASS	°C	10	-14	2,8	8,0	EN 12593	6	6	8	no
Softening point	°C	39	49,9	0,8	3,6	EN 1427	2,0	2,0	2,2	no
Flash point COC	°C	11	326	7,6	23	EN ISO 2592	17	17	22	no
RTFOT										
change of mass	m-%	15	0,04	0,031	0,11	EN 12607-1	0,20 ¹⁾	0,20	0,09	(yes)
penetration 25 °C	1/10mm	13	34	1,2	4	EN 12607-1	not given			*
retained penetration	%	13	60	3,6	15	EN 12607-1	10	10	10	yes *
softening point	°C	14	57,0	0,9	3,4	EN 12607-1	not given			
change in softening point	°C	14	7,0	0,6	1,9	EN 12607-1	4,0	4,0	1,6	yes
viscosity 60 °C	Pas	11	775	76,1	259	EN 12607-1	not given			
viscosity ratio at 60 °C		11	3,7	0,3	1,0	EN 12607-1	20 % from average	0,7	0,8	no
Bitumen B200										
Penetration 25 °C	1/10mm	40	175	10,5	40	EN 1426	6 % from average	11	30	no
Viscosity 60 °C	Pas	16	39,3	1,6	6,2	EN 12596	10 % from average	3,9	4,4	no
Viscosity 135 °C	mm ² /s	18	174	6,1	22	EN 12595	6 % from average	10,4	17,2	no *
Breaking point FRAASS	°C	10	-19	2,8	8,0	EN 12593	6	6	8	no
Softening point	°C	39	39,6	0,9	4,4	EN 1427	2,0	2,0	2,6	no
Flash point COC	°C	10	324	11,7	36	EN ISO 2592	17	17	33	no
RTFOT										
change of mass	m-%	13	0,01	0,02	0,09	EN 12607-1	0,20 ¹⁾	0,20	0,07	(yes) *
penetration 25 °C	1/10mm	13	84	5,2	19	EN 12607-1	not given			
retained penetration	%	13	47	2,8	11	EN 12607-1	10	10	8	yes
softening point	°C	13	45,7	0,6	1,8	EN 12607-1	not given			*
change in softening point	°C	13	6,3	0,6	2,0	EN 12607-1	2,0	2,0	1,6	yes *
viscosity 60 °C	Pas	11	104	6,3	22	EN 12607-1	not given			
viscosity ratio at 60 °C		11	2,6	0,1	0,4	EN 12607-1	20 % from average	0,5	0,3	yes

¹⁾ for 0,3 % < value < 0,80 %

*) Outliers are excluded in the evaluation.