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Distribution: Participants

BITUMEN ROUND ROBIN 1-2012

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 Road Directorate
Estonia	Nynas/ Tallinn Technical Center of Estonian Roads
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 Asphalt Lemminkäinen Petrotest Statens vegvesen Veidekke Asphalt
Sweden	NCC Roads/ Biskopstorp NCC Roads/ Hisings Kärra NCC Roads/ Södra Sandby NCC Roads/Umeå

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	NCC Roads/ Upplands Väsby
	NCC Roads/Västerås
	Nynas/Goteborg Refinery
	Nynas/Nynashamn Refinery
	Nynas/Nynashamn Quality Control
	PEAB Asfalt/Boden
	PEAB Asfalt/Hisings Kärä
	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/Dundee
	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.

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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 the sample B70
- Breaking point, Fraass in the sample B70.
- Softening point in the sample B200.
- Flash point, COC in both the samples.
- RTFOT, viscosity ratio at 60 °C in the sample B70.

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	59	67	67	66	65	66	69	65	66	63
Viscosity 60 °C	Pas	183	184	185	174	179,3		185	217,9	179	162,1
Viscosity 135 °C	mm ² /s	350	374	356	334	360,3		344	353,8		355,9
Breaking point, Fraass	°C	-15	-13	-15	-14	-19		-15			-14
Softening point	°C	48,6	48,0	48,0	48,8	52,5 *	49,8	48,0	48,4	48,6	49,2
Flash point, COC	°C		317	328	352	348		318	308		
RTFOT											
-change of mass	m- %	-0,12	-0,06	-0,09	-0,10	-0,11	-0,26	-0,01	-0,09		-0,14
-penetration 25°C	1/10mm	35	39	37	35	36	37	40	36		35
retained penetration	%	66,1	58	55	53	55,4	56,1	58	55,4		56
-softening point	°C	55,4	55,0	54,8	55	55,9	56,0	54,4	55,4		
change in softening point	°C	6,8	7,0	6,8	6,3	3,4 *	6,2	6,4	7		6,8
-viscosity 60°C	Pas	560	651	628	554	674,6		564	690,1		
viscosity ratio at 60 °C		3,1	3,54	3,4	3,2	3,8		3,0	3,16		

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25 °C	1/10mm	74	65	68	64	68	67	72	68	66	70
Viscosity 60 °C	Pas			227	183	177	177	194	187	172	196
Viscosity 135 °C	mm ² /s		342	287 *	356	348	381	365	356	365	346
Breaking point, Fraass	°C				-14	-20	-17		-16	-12	
Softening point	°C	48,8	48,2	48,0	47,8	48,4	48,2	48,2	48,6	48,6	48,8
Flash point, COC	°C	323	312			313	318				
RTFOT											
-change of mass	m- %	-0,04		-0,14	0,04	-0,13	-0,10		-0,15	-0,085	-0,03
-penetration 25°C	1/10mm	38		41	35	40	38		38	37	35
retained penetration	%	51		60	55	59	57		55,9	56,1	50
-softening point	°C	56,2		53,8	54,8	55,8	55,6		56,0	56,2	56,7
change in softening point	°C	7,4		5,8	7,0	7,4	7,4		7,4	7,8	7,9
-viscosity 60°C	Pas			516	691	571	673		690	650	733
viscosity ratio at 60 °C				2,3	3,8	3,2	3,8		3,7	3,8	3,7

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25 °C	1/10mm	63	69	69	66	57	65	63	67	60	64
Viscosity 135 °C	mm ² /s	354	349	(380cPs)							
Softening point	°C	48,0	46,4	49,4	48,3	49,8	48,8	48,2	48,8	48,0	48,0

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25 °C	1/10mm		70	63	66	60,7	63	68	72	70	65
Softening point	°C	48,0	48,4			50,6	49,2	48,0	48,2	48,2	48,6

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25 °C	1/10mm	64	65	60	66	67					
Softening point	°C	48,6		48,8	47,2	49,3					

* 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	151	167	160	164	163	168	161	166	164	162
Viscosity 60 °C	Pas	47	44,7	45	45,6	44,6		45,6	50,7	43,6	41,2
Viscosity 135 °C	mm2/s	187	182	182	181	357*		178	185,9		185,5
Breaking point, Fraass	°C	-22	-19	-21	-22	-19		-19			-20
Softening point	°C	40,2	40,0	39,2	39,8	40,5	41,7	39,4	40,4	40,4	41,2
Flash point, COC	°C		318	328	356	319		310	274		
RTFOT											
-change of mass	m- %	0,03	0,07	0,07	0,07	0,12	0,09	0,09	0,06		0,09
-penetration 25°C	1/10mm	82	85	84	76	86	86	89	81		80
retained penetration	%	54,3	51	53	46	52,8	51,1	55	48,8		49
-softening point	°C	46,2	45,8	46,0	46,4	46,1	47,4	46,0	46,4		
change in softening point	°C	6,0	5,8	6,8	6,6	5,6	5,7	6,6	6		5,7
-viscosity 60°C	Pas	107	110	120	113	109,8		106	136,4		
viscosity ratio at 60 °C		2,3	2,46	2,7	2,5	2,5		2,3	2,69		

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25 °C	1/10mm	205 *	160	166	156	172	165	180	167	155	168
Viscosity 60 °C	Pas			42,7	45	44,1	44,1	47,2	47	46,0	46
Viscosity 135 °C	mm2/s		185	192	184	180	190	189	185	228 *	183
Breaking point, Fraass	°C				-23	-20	-20		-20	-14	
Softening point	°C	41	40,4	40,2	39,0	40,4	39,4	40,7	40,4	41,0	39,6
Flash point, COC	°C	312	318			317	316				
RTFOT											
-change of mass	m- %	-0,01		0,01	-0,09	0,04	0,04		0,04	0,077	0,04
-penetration 25°C	1/10mm	93		86	81	88	80		82	79	84
retained penetration	%	45		52	52	51	48		49,1	51,0	50
-softening point	°C	46,6		45,2	46,0	46	46,6		46,2	47	45,3
change in softening point	°C	5,6		5,0	7,0	5,6	7,2		5,8	6	5,7
-viscosity 60°C	Pas			115	121	108	123		122	116	105
viscosity ratio at 60 °C				2,7	2,7	2,4	2,8		2,6	2,5	2,3

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25 °C	1/10mm	163	170	181	161	151	161	156	167	151	163
Viscosity 135 °C	mm2/s	184	179	(200cPs)							
Softening point	°C	39,8	39,6	42,4	39,6	41,6	41,0	39,6	40,6	39,8	40,2

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25 °C	1/10mm		171	155	177	152,3	150	167	170	166	163
Softening point	°C	39,8	40,0			42,7	41,6	39,6	39,6	39,6	40,2

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25 °C	1/10mm	166	164	149	160	171					
Softening point	°C	39,8		40,2	39,7	40,4					

* Outlier

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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 B70									
Penetration 25 °C	1/10mm	44	66	3,49	17	6 % from average	4	10	no
Viscosity 60 °C	Pas	17	186	15,92	64,9	10 % from average	18,6	45,1	no
Viscosity 135 °C	mm ² /s	18	355	11,36	47,0	6 % from average	21,3	32,1	no *
Breaking point, Fraass	°C	12	-15	2,35	8	6	6	7	no
Softening point	°C	41	48,5	0,71	4,2	2,0	2,0	2,0	yes *
Flash point COC	°C	10	324	14,97	44	17	17	42	no
RTFOT									
change of mass	m- %	17	-0,10	0,07	0,30	0.20 ¹⁾	0,20	0,19	yes
penetration 25 °C	1/10mm	17	37	1,98	6	not given			
retained penetration	%	17	56	3,60	16	10	10	10	yes
softening point	°C	16	55,4	0,76	2,9	not given			
change in softening point	°C	16	7,0	0,58	2,1	4,0	4,0	1,7	yes *
viscosity 60 °C	Pas	14	632	66,61	217	not given			
viscosity ratio at 60 °C		14	3,4	0,43	1,5	20 % from average	0,7	1,2	no
Bitumen B200									
Penetration 25 °C	1/10mm	43	163	7,63	32	6 % from average	10	22	no *
Viscosity 60 °C	Pas	17	45,3	2,09	9,5	10 % from average	4,5	5,9	no
Viscosity 135 °C	mm ² /s	17	184	3,84	14,0	6 % from average	11,1	10,9	yes *
Breaking point, Fraass	°C	12	-20	2,27	9	6	6	6	yes
Softening point	°C	42	40,3	0,82	3,7	2,0	2,0	2,3	no
Flash point, COC	°C	10	317	19,92	82	17	17	56	no
RTFOT									
change of mass	m- %	17	0,05	0,05	0,21	0.20 ¹⁾	0,20	0,14	yes
penetration 25 °C	1/10mm	17	84	4,18	17	not given			
retained penetration	%	17	51	2,69	10	10	10	8	yes
softening point	°C	16	46,2	0,55	2,2	not given			
change in softening point	°C	17	6,0	0,59	2,2	2,0	2,0	1,7	yes
viscosity 60 °C	Pas	14	115	8,69	31,4	not given			
viscosity ratio at 60 °C		14	2,5	0,17	0,5	20 % from average	0,5	0,5	yes

¹⁾ for 0, 3% <value <0,80%

*) Outlier is excluded in the evaluation.