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Distribution: Participants

BITUMEN ROUND ROBIN 1-2011

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	Nynas/ Tallinn Technical Center of Estonian Roads
Finland	Aalto Destia Icopal Katepal Lemminkäinen/Laboratory NCC Roads Neste Oil/Naantali Refinery Neste Oil/Research and Technology Nordic Waterproofing SGS
Norway	Feiring Asphalt Lemminkäinen Petrotest SINTEF Statens vegvesen Veidekke Asphalt
Sweden	Asfalt och stenkontroll/Hok Asfalt och stenkontroll/Östad NCC Roads/ Biskopstorp

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	NCC Roads/ Hisings Kärra
	NCC Roads/ Sundsvall
	NCC Roads/ Södra Sandby
	NCC Roads/Umeå
	NCC Roads/Västerås
	Nynas/Nynäshamn Quality Laboratory
	PEAB/Boden
	PEAB/Hisings Kärra
	PEAB/Helsingborg
	PEAB/ Stockholm
	Skanska Sverige/Göteborg
	Skanska Sverige/Malmö
	Skanska Sverige/Norrköping
	Skanska Sverige/Vällsta
	Svevia/ Jönköping
	Svevia/ Umeå
	Svevia/ Örebro
	VTI
UK	Eastham Refinery
	Nynas/Dundee
	Nynas/Eastham
	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.
- Kinematic viscosity in both the samples.
- Breaking point, Fraass in both the samples.
- Softening point in both the samples.
- Flash point, COC in the sample B70.
- 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)

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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	68	74	77	65	67	69	73	76	79	74
Viscosity 60 °C	Pas	134	139	134	136,2	136		133	137	134	
Viscosity 135 °C	mm ² /s	292	291	283	279,8	289		285	282	312	
Breaking point, Fraass	°C	-13	-15	-17,5	-12	-17				-18	-15
Softening point	°C	45,2	47,2	46,8	46,8	46,9	50,8*	46,4	47,0	47,2	47,2
Flash point, COC	°C		320	314	346	349	315				324
RTFOT											
-change of mass	m- %	0,06	0,08	0,06	0,07	0,13	0,08	-0,03	0,05		0,09
-penetration 25°C	1/10mm	37	40	44	39	39	42	40		44	41
retained penetration	%	54,4	54	57	60	56,2	61	55	55,3	55,7	55,41
-softening point	°C	53,2	54,8	53,6	53,8	53,7	55,6	52,7		54,2	54,2
change in softening point	°C	8,0	7,6	6,8	7,0	6,8	4,8	6,3	7,3	7,0	7,0
-viscosity 60°C	Pas	471	506	451	461,3	455		460			
viscosity ratio at 60°C		3,5	3,64	3,4	3,4	3,3		3,5	4,1		

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25 °C	1/10mm	71	73	74	71	75	74	77	76		
Viscosity 60 °C	Pas		160*		138	134	136	139	138,5		
Viscosity 135 °C	mm ² /s	281	281	313	286	281	290	294	288,8		
Breaking point, Fraass	°C	-18			-14	-21	-16				
Softening point	°C	47,4	48,0	47,3	46,8	47,4	47,2	46,0	47,6		
Flash point, COC	°C	319	316			326	319		310		
RTFOT											
-change of mass	m- %	0,19		0,057	-0,111	0,03	0,05		0,07		
-penetration 25°C	1/10mm	35		40	38	42	40		39		
retained penetration	%	49,30		54	54	56	53		49,40		
-softening point	°C	56,3		55,1	54,4	55,0	55,0		55,2		
change in softening point	°C	8,9		7,8	7,6	7,6	7,8		7,6		
-viscosity 60°C	Pas				540	488	552		210,2*		
viscosity ratio at 60°C					3,9	3,6	4,1		1,518*		

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25 °C	1/10mm	72	69	69	72	69	72	69	69	71	74
Viscosity 135 °C	mm ² /s	292	280								
Softening point	°C	48,3	47,2	48,1	47,0	48,0	48,4	48,0	47,0	47,2	47,0

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25 °C	1/10mm		74	70	67	65	65	67	71	76	74
Viscosity 135 °C	mm ² /s										
Softening point	°C	47,0	46,2		50,0	49,0	49,4	47,8	47,8	47,0	47,4

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25 °C	1/10mm	71	72	65	71	68	73	73,1	75	78	
Viscosity 135 °C	mm ² /s										
Softening point	°C	47,6	49,0	49,2	47,6		46,0	47,55	47,2	47,8	

*) 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	175	200	182	184	174	169	183	188	191	186
Viscosity 60 °C	Pas	39	38,1	38	37,8	40,3		38	40	37,2	
Viscosity 135 °C	mm2/s	163	163	165	161,2	172		164	162	174	
Breaking point, Fraass	°C	-19	-18	-20	-23	-21				-21	-17
Softening point	°C	36,2	39,6	38,8	38,6	38,7	39,8	38,9	39,0	38,6	39,0
Flash point, COC	°C		318	314	324	324	316				320
RTFOT											
-change of mass	m- %	0,05	0,04	0,05	0,05	0,09	0,19*	0,03	0,01	0,04	0,07
-penetration 25°C	1/10mm	80	87	87	83	86	92	96		96	82
retained penetration	%	45,7	44	48	45	49,4	54	52	46,3	50	44,09
-softening point	°C	43,6	46,0	45,4	45	44,9	46,8	45,6		45,4	45,8
change in softening point	°C	7,4	6,4	6,6	6,4	6,2	7,0	6,7	6,6	6,8	6,8
-viscosity 60°C	Pas	99	111	105	89,6	97,3		95			
viscosity ratio at 60°C		2,5	2,91	2,8	2,4	2,4		2,5	2,8		

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25 °C	1/10mm	180	182	182	180	188	190	195	172		
Viscosity 60 °C	Pas		41,2		38	37,8	37,9	41,1	38,1		
Viscosity 135 °C	mm2/s	164,5	159	171	164	165	169	167	167,6		
Breaking point, Fraass	°C	-21			-22	-21	-26				
Softening point	°C	39,4	39,0	39,2	38,4	39,4	37,8	38,5	38,2		
Flash point, COC	°C	321	310			325	320		326		
RTFOT											
-change of mass	m- %	0,05		0,0356	-0,082	0,02	0,03		0,06		
-penetration 25°C	1/10mm	84		79	87	89	87		81		
retained penetration	%	46,67		43	48	47	46		47,10		
-softening point	°C	46		45,9	45,8	45,8	46,2		46,2		
change in softening point	°C	6,6		6,7	7,4	6,4	8,4		8		
-viscosity 60°C	Pas				114	104	108		115,2		
viscosity ratio at 60°C					3,0	2,8	2,8		3,023		

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25 °C	1/10mm	180	177	171	184	172	186	168	180	179	189
Viscosity 135 °C	mm2/s	168	161								
Softening point	°C	40,8	38,0	40,0	39,2	39,4	38,8	39,0	39,2	39,2	39,0

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25 °C	1/10mm		190	175	175	165	165	184	176	181	186
Viscosity 135 °C	mm2/s										
Softening point	°C	37,8	38,4		41,6	40,0	40,2	39,0	39,8	40,4	38,4

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25 °C	1/10mm	183	204	165	179	162	184	170,1	194	189	
Viscosity 135 °C	mm2/s										
Softening point	°C	39,6	40,9	41,0	39,8		39,2	39,4	39,2	39,4	

* 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	46	72	3,63	14	6 % from average	4	10	no
Viscosity 60 °C	Pas	13	136	2,13	6	10 % from average	13,6	6,0	yes *
Viscosity 135 °C	mm ² /s	18	289	9,76	33	6 % from average	17,3	27,6	no
Breaking point FRAASS	°C	11	-16	2,59	9,0	6	6	7	no
Softening point	°C	45	47,5	1,04	5,6	2,0	2,0	2,9	no
Flash point COC	°C	11	323	12,72	39	17	17	36	no
RTFOT									
change of mass	m- %	15	0,06	0,07	0,30	0.20 ¹⁾	0,20	0,19	yes
penetration 25 °C	1/10mm	15	40	2,42	9	not given			
retained penetration	%	16	55	3,05	12	10	10	9	yes
softening point	°C	15	54,5	0,96	3,6	not given			
change in softening point	°C	16	7,2	0,89	4,1	4,0	4,0	2,5	yes
viscosity 60 °C	Pas	9	487	37,69	101	not given			*
viscosity ratio at 60 °C		10	3,6	0,29	0,8	20 % from average	0,7	0,8	no*
Bitumen B200									
Penetration 25 °C	1/10mm	46	181	9,30	42	6 % from average	11	26	no
Viscosity 60 °C	Pas	14	38,8	1,33	4,0	10 % from average	3,9	3,8	yes
Viscosity 135 °C	mm ² /s	18	166	4,07	15	6 % from average	9,9	11,5	no
Breaking point, FRAASS	°C	11	-21	2,44	9	6	6	7	no
Softening point	°C	45	39,2	0,94	5,4	2,0	2,0	2,7	no
Flash point, COC	°C	11	320	5,00	16	17	17	14	yes
RTFOT									
change of mass	m- %	16	0,05	0,05	0,27	0.20 ¹⁾	0,20	0,15	yes
penetration 25 °C	1/10mm	15	86	5,25	17	not given			
retained penetration	%	16	47	2,95	11	10	10	8	yes
softening point	°C	15	45,6	0,74	3,2	not given			
change in softening point	°C	16	6,9	0,61	2,2	4,0	4,0	1,7	yes
viscosity 60 °C	Pas	10	104	8,49	26	not given			
viscosity ratio at 60 °C		11	2,7	0,23	0,6	20 % from average	0,5	0,7	no

¹⁾ for 0, 3% <value <0,80%

*) Outlier is excluded in the evaluation.