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27 May 2010

Distribution: Participants

BITUMEN ROUND ROBIN 1-2010

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 Skanska Asfalt AS Statens vegvesen
Sweden	KTH NCC Roads/ Biskopstorp NCC Roads/ Hisings Kärä NCC Roads/ Sundsvall NCC Roads/ Södra Sandby NCC Roads/Umeå

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	Nynas/Göteborg Refinery
	Nynas/Nynäshamn
	Nynas/Nynäshamn Refinery
	PEAB/Boden
	PEAB/Göteborg
	PEAB/Helsingborg
	PEAB/ Stockholm
	Skanska Sverige/Syd
	Skanska Sverige/Väst
	Svevia/ Jönköping
	Svevia/ Umeå
	Svevia/ Örebro
	VTI
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 the sample B70.
- o Softening point in the sample B200.
- o Flash point, COC in the sample B200.
- o 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	

[illegible]

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Table 3 Bitumen B200

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	171	182	176	176	181	181	182	182	166	169
Viscosity 60°C	Pas	40	40,6	39	45	38,8	38	41	38,6	47,1	40
Viscosity 135°C	mm2/s	174	177	170	168,5	166	178	170	177	176	169
Breaking point,Fraass	°C	-19	-22	-20,5	-17				-23	-18	
Softening point	°C	37,4	40,2	39,4	39,4	39,8	39,6	39,4	39,6	39,8	38,4
Flash point, COC	°C		312	328	312	318					
RTFOT											
-change of mass	m-%	-0,08	-0,08	-0,05	-0,07	-0,05	-0,08	-0,12	-0,09	-0,032	0,01
-penetration 25°C	1/10mm	78	87	84	77	87	92	86	86	79	86
retained penetration	%	45,6	48	48	44	48	51	47,3	47,3	47,59	51
-softening point	°C	44,6	45,4	45,8	45,9	45,4	45,7	46,2	46,4	46,6	45,4
change in softening point	°C	7,2	5,2	6,4	6,5	5,6	6,1	6,8	6,8	6,8	7,0
-viscosity 60°C	Pas	109	99,9	106	120,8		95	117		119	115
viscosity ratio at 60°C		2,7	2,46	2,7	2,7		2,5	2,8		2,5	2,9

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm		177	169	161	165	171	180	187	189	178
Viscosity 60°C	Pas			40			44,3	39,2	39,0		
Viscosity 135°C	mm2/s	178		176			168,5	168	179		
Breaking point,Fraass	°C			-19				-23	-22		
Softening point	°C	39,2	40,2	39,6	41,4	41,1	39,5	39,4	39,2	40,5	40,35
Flash point, COC	°C		322	346			266	316	311		
RTFOT											
-change of mass	m-%	0,0956		-0,08			-0,095	-0,10	-0,11		
-penetration 25°C	1/10mm			77			75	83	81		
retained penetration	%			46			43,9	46,1	43		
-softening point	°C	46,2		47,3			46,7	44,8	46,6		
change in softening point	°C	7,0		7,7			7,2	5,4	7,4		
-viscosity 60°C	Pas			128			141,9	108	122		
viscosity ratio at 60°C				3,2			3,20	2,8	3,1		

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	184	179,7	183	158	175	183	178	171	188	180
Viscosity 60°C	Pas			40,6	39,1						
Viscosity 135°C	mm2/s			175		174				168	
Breaking point,Fraass	°C										
Softening point	°C	38,6	40,6	39,2	39,4	39,5	39,8	38,8	40,4	37,4	39,0
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	177	179	166	183	185	179	200	174	173	180
Viscosity 60°C	Pas										
Viscosity 135°C	mm2/s										
Breaking point,Fraass	°C										
Softening point	°C	40,0	40,8	40,2	41,6	39,4	41,0	39,5	38,4		39,8
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											

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Table 4 Evaluation of the results

		n	Aver.	Stand. Dev. s	Max. diff. of the results	Standard method	Reproducibility R in standard	R stand	R calc	Acceptance
Bitumen B70										
Penetration 25 °C	1/10mm	40	65	3,0	12	EN 1426	6 % from average	4	8	no
Viscosity 60 °C	Pas	16	180	9,1	42,4	EN 12596	10 % from average	18,0	25,7	no
Viscosity 135 °C	mm ² /s	18	324	12,3	43,5	EN 12595	6 % from average	19,4	34,8	no
Breaking point FRAASS	°C	10	-17	2,5	6,5	EN 12593	6	6	7	no
Softening point	°C	39	48,8	0,7	3,0	EN 1427	2,0	2,0	2,0	yes
Flash point COC	°C	9	320	5,1	16	EN ISO 2592	17	17	15	yes
RTFOT										
change of mass	m- %	15	0,03	0,04	0,14	EN 12607-1	0.20 ¹⁾	0,20	0,10	yes
penetration 25 °C	1/10mm	15	37	2,6	10	EN 12607-1	not given			
retained penetration	%	15	58	3,0	10	EN 12607-1	10	10	9	yes
softening point	°C	15	56,0	0,7	2,4	EN 12607-1	not given			
change in softening point	°C	15	7,0	0,6	2,0	EN 12607-1	4,0	4,0	1,6	yes
viscosity 60 °C	Pas	12	644	49,5	152	EN 12607-1	not given			
viscosity ratio at 60 °C		12	3,6	0,3	1,1	EN 12607 -1	20 % from average	0,7	0,9	no
Bitumen B200										
Penetration 25 °C	1/10mm	39	177	8,2	42	EN 1426	6 % from average	11	23	no
Viscosity 60 °C	Pas	16	40,6	2,6	9,1	EN 12596	10 % from average	4,1	7,3	no
Viscosity 135 °C	mm ² /s	18	173	4,3	13	EN 12595	6 % from average	10,4	12,2	no
Breaking point, FRAASS	°C	9	-20	2,2	6,0	EN 12593	6	6	6	yes
Softening point	°C	39	39,7	0,9	4,2	EN 1427	2,0	2,0	2,6	no
Flash point, COC	°C	9	315	21,3	80	EN ISO 2592	17	17	60	no
RTFOT										
change of mass	m- %	15	-0,06	0,05	0,22	EN 12607-1	0.20 ¹⁾	0,20	0,15	yes
penetration 25°C	1/10mm	14	83	5,0	17	EN 12607-1	not given			
retained penetration	%	14	47	2,4	8	EN 12607-1	10	10	7	yes
softening point	°C	15	45,9	0,7	2,7	EN 12607-1	not given			
change in softening point	°C	15	6,6	0,7	2,5	EN 12607-1	4,0	4,0	2,1	yes
viscosity 60 °C	Pas	12	115	12,8	46,9	EN 12607-1	not given			
viscosity ratio at 60 °C		12	2,8	0,3	0,7	EN 12607 -1	20 % from average	0,6	0,7	no

¹⁾ for 0,3% <value <0,80%