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Distribution: Participants

BITUMEN ROUND ROBIN 1-2013

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	Danish Road Institute NCC Roads Kemi
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/ Fjellhamar Lemminkäinen/Greåker Petrotest Statens vegvesen Veidekke Industri
Portugal	LNEC/ LEMOC-UOII
Sweden	NCC Roads/ Biskopstorp NCC Roads/ Hisings Kärä

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NCC Roads/ Sundsvall
NCC Roads/ Södra Sandby
NCC Roads/Umeå
NCC Roads/Upplands Väsby
NCC Roads/Västerås
Nynas/Goteborg Refinery
Nynas/Nynashamn Refinery
Nynas/Nynashamn Quality Control
PEAB Asfalt/Boden
PEAB Asfalt/Hisings Kärra
PEAB Asfalt/Helsingborg
PEAB Asfalt/Stockholm
PEAB Asfalt/Sundsvall
Skanska Sverige/Gunnilse
Skanska Sverige/Malmö
Skanska Sverige/Norrköping
Skanska Sverige/Vällsta, Upplands Väsby
Svevia/Jönköping
Svevia/Umeå
Svevia/Örebro
VTI
UK 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 both the samples.
- Breaking point, Fraass in both the samples.
- Flash point, COC in both the samples.
- RTFOT, viscosity ratio at 60°C in the sample B70.
- RTFOT, retained penetration in the sample B200.

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	71	75	74	76	74,0	72	75		73	72
Viscosity 60°C	Pas	139		137	158	138,5	139	141		138,5	136,7
Viscosity 135°C	mm ² /s	305		304	303	315,3	306	295			308,6
Breaking point, Fraass	°C	-15	-16	-15	-14	-15	-14	-16			-17
Softening point	°C	46,8	48,0	47,6	47,0	46,9	48,0	46,0		47,2	47,5
Flash point, COC	°C			330	326	348	330	(>320)			
RTFOT											
-change of mass	m-%	0,07	0,08	0,07	0,07	0,089	0,09	0,13			-0,03 *
-penetration 25°C	1/10mm	41	49	42	41	41	40	44			42
retained penetration	%	57,7	66	57	54	55,4	56,0	58,7			58
-softening point	°C	52,4	52,8	54,4	52,8	53,5	54,4	53,0			
change in softening point	°C	5,6	4,8	6,8	5,8	6,6	6,4	7,0			6,3
-viscosity 60°C	Pas	388		469	415	479,3					
viscosity ratio at 60°C		2,79		3,4	2,6	3,5					

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	80,1	74	81	71	77	74	74	78	73	75
Viscosity 60°C	Pas		146	131	140	138	138	141	140	158	138
Viscosity 135°C	mm ² /s	325,9		309	308	301	325	314	302	352 *	296
Breaking point, Fraass	°C	-12,3	-14	-16	-16	-21	-22		-17	-14	
Softening point	°C	47,2	46,6	45,8	46,4	47,2	46,6	47,0	47,2	47,6	47
Flash point, COC	°C	325	326	342		320	351	318		334	
RTFOT											
-change of mass	m-%	0,06		0,07	0,09	0,09	0,07		0,09	0,1	0,09
-penetration 25°C	1/10mm	50,8		47	44	44	42		43	41	42
retained penetration	%	63,42		58	62	57	57		55,4	56,2	56
-softening point	°C	54,2		53,0	52,2	53	53,6		53,7	54,8	54,1
change in softening point	°C	7,0		7,2	5,8	5,8	7,0		6,5	7,2	7,1
-viscosity 60°C	Pas			454	383	405	452		492,3	499	450
viscosity ratio at 60°C				3,5	2,7	2,9	3,3		3,5	3,2	3,3

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	73	76	75	74	81	76	74	68	67	75
Viscosity 135°C	mm ² /s	311	301				310				
Softening point	°C	47,7	46,2	47,6	47,4	47,0	47,1	47,0	48,2	46,4	46,6
Breaking point, Fraass	°C				-20						
Flash point, COC	°C							331			

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm		77	72	67		71	74	76	77	76
Softening point	°C	47,0	46,8		48,6	47,63	47,8	48,0	46,8	46,2	47,4

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25°C	1/10mm	75	63	62	64,8	72	74	70	73	64,5	
Softening point	°C	48,0		47,8	48,4	46,4	47,6	47,2	46,8	47,2	

* 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	178	180	174	182	176	183	185		180	176
Viscosity 60°C	Pas	49 *		42	45	41,3	41,4	42,4		40,9	40,5
Viscosity 135°C	mm ² /s	176		170	167	174,9	174	170			169,7
Breaking point, Fraass	°C	-18	-21	-19	-19	-19	-17	-20			-22
Softening point	°C	38,6	39,6	39,6	39,2	39	39,6	38,2		38,8	39,8
Flash point, COC	°C			328	306	330	316	314			
RTFOT											
-change of mass	m-%	0,03	0,09	0,06	0,09	0,034	0,04	0,07			0,00
-penetration 25°C	1/10mm	89	105	83	84	87	81	78			78
retained penetration	%	50,0	58	48	46	49,4	44,3	42,2			44
-softening point	°C	45,0	45,2	46,4	45,4	45,7	46,2	44,6			
change in softening point	°C	6,4	5,6	6,8	6,2	6,7	6,6	6,4			6
-viscosity 60°C	Pas	107		117	117	123,4					
viscosity ratio at 60°C		2,2		2,8	2,6	3,0					

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	245,9 *	178	182	169	203	183	185	185	173	187
Viscosity 60°C	Pas		43,2	38,6	42	41,1	41,0	38,8	43	42,2	39
Viscosity 135°C	mm ² /s	180,7		175	173	170	181	176	175	179	165
Breaking point, Fraass	°C	-15,1	-20	-21	-23	-21	-22		-25	-14	
Softening point	°C	39,1	38,8	38,6	38,2	39,4	38,2	39,0	38,8	39,6	39,1
Flash point, COC	°C	302	306	313		315	298	308		331	
RTFOT											
-change of mass	m-%	0,06		0,02	0,09	0,05	-0,02		0,03	0,07	0,01
-penetration 25°C	1/10mm	86,2		96	83	91	79		85	80	84
retained penetration	%	35,05		53	49	45	43		45,9	46,2	45
-softening point	°C	46,2		44,8	44,8	45,2	45,4		45,6	46,8	45,2
change in softening point	°C	7,1		6,2	6,6	5,8	7,2		6,9	7,2	6,1
-viscosity 60°C	Pas			108	118	113	125		130	124	116
viscosity ratio at 60°C				2,8	2,8	2,7	3,0		3,0	2,9	3,0

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	178	196	202	184	186	172	179	179	173	184
Viscosity 135°C	mm ² /s	175	173				178				
Softening point	°C	39,5	38,6	39,7	38,8	38,5	39,2	38,6	39,4	38,8	38,6
Breaking point, Fraass	°C				-22						
Flash point, COC	°C							319			

Laboratory		31	32	33	34	35	36	37	38	39	40
Penetration 25°C	1/10mm		183	168	168		166	186	190	196	187
Softening point	°C	39,2	38,4		41,2	40,0	39,6	38,8	38,6	39,0	40,4

Laboratory		41	42	43	44	45	46	47	48	49	50
Penetration 25°C	1/10mm	186	167	156	165,8	195	178	168	169	172,8	
Softening point	°C	39,0		42 *	40,4	38,5	39,2	38,6	38,4	39,2	

* 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	73	4,24	19	6 % from average	4	12	no
Viscosity 60 °C	Pas	17	141	7,01	27,0	10 % from average	14,1	19,8	no
Viscosity 135 °C	mm ² /s	18	308	8,45	30,9	6 % from average	18,5	23,9	no *
Breaking point, Fraass	°C	17	-16	2,64	10	6	6	8	no
Softening point	°C	46	47,2	0,64	2,8	2,0	2,0	1,8	yes
Flash point COC	°C	12	332	10,40	33	17	17	29	no
RTFOT									
change of mass	m- %	15	0,08	0,02	0,07	0.20 ¹⁾	0,20	0,05	yes *
penetration 25 °C	1/10mm	16	43	3,08	10,8	not given			
retained penetration	%	16	58	3,20	12	10	10	9	yes
softening point	°C	15	53,5	0,79	2,6	not given			
change in softening point	°C	16	6,4	0,70	2,4	2,0	2,0	2,0	yes
viscosity 60 °C	Pas	11	444	40,80	116	not given			
viscosity ratio at 60 °C		11	3,2	0,34	0,9	20 % from average	0,63	1,0	no
Bitumen B200									
Penetration 25 °C	1/10mm	45	180	9,90	47	6 % from average	11	28	no *
Viscosity 60 °C	Pas	16	41,4	1,69	6,4	10 % from average	4,1	4,8	no *
Viscosity 135 °C	mm ² /s	19	174	4,37	16,0	6 % from average	10,4	12,4	no
Breaking point, Fraass	°C	17	-20	2,79	11	6	6	8	no
Softening point	°C	45	39,1	0,63	3,0	2,0	2,0	1,8	yes *
Flash point, COC	°C	13	314	10,55	33	17	17	30	no
RTFOT									
change of mass	m- %	16	0,05	0,03	0,11	0.20 ¹⁾	0,20	0,09	yes
penetration 25 °C	1/10mm	16	86	7,11	27	not given			
retained penetration	%	16	47	5,05	23	10	10	14	no
softening point	°C	15	45,5	0,65	2,2	not given			
change in softening point	°C	16	6,5	0,49	1,6	4,0	4,0	1,4	yes
viscosity 60 °C	Pas	11	118	7,14	23,0	not given			
viscosity ratio at 60 °C		11	2,8	0,24	0,8	20 % from average	0,56	0,7	no

¹⁾ for 0, 3% <value <0,80%

*) Outlier is excluded in the evaluation.