

Author: Auli Kettunen, Neste Oil
Approved by: Timo Blomberg

30 May 2006

Distribution: Participants

BITUMEN ROUND ROBIN 1-2006

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 B150

Test methods

The test methods used are given in table 1.

Participating Laboratories

Belgium	Nynas/Antwerp
China	PNAC laboratory
Denmark	NCC Kemi
Estonia	Technical Center of Estonian Roads
Finland	Icopal
	Katepal
	Lemminkäinen/Factory
	Lemminkäinen/Laboratory
	Neste Oil/Development and Laboratories
	Neste Oil/Naantali Refinery
	Neste Oil/Porvoo Refinery
	TKK
	Valtatie
	VTT
	SINTEF
	Petrotest
	KTH, Vägteknik
Norway	Nynäs/Göteborg
	Nynäs/Nynäshamn
	PEAB
	Skanska Sverige/Syd
	Skanska Sverige/Väst
	VTI
Sweden	Väglaboratoriet

Auli Kettunen

30 May 2006

UK Vägverket/ Jönköping
Vägverket/Umeå
Vägverket/Örebro
Nynas/Dundee
Nynas/Eastham

Results

The primary results from the participating laboratories are given in tables 2 and 3.

Evaluation

Evaluation of the results against the reproducibility of the test methods, according to the ASTM C 670 method, is given in table 4. The average and the maximum difference of the results in each of the test methods are calculated. The maximum difference is compared with the reproducibility of the method, which is calculated based on the R of the method (for two laboratories) and the number of laboratories. The calculation is made according to the following formula.

$$R_n = K_n * (R_2/2,8)$$

R_2 = Reproducibility for two laboratories, given in the method

R_n = Reproducibility for n laboratories

K_n = Constant factor for n laboratories, ASTM C670

n = Number of laboratories

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.
- Softening point in the sample B70
- Breaking point Fraass in both the samples.
- RTFOT, change of mass in the sample B150.

Auli Kettunen

30 May 2006

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	

Auli Kettunen

30 May 2006

Table 2 Bitumen B70

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	76	72	66	72	69	72	72	68	75	75
Viscosity 60°C	Pas	147	143		142	142	142	134	143	139	139
Viscosity 135°C	mm ² /s	299	302		300	284	282	292	288	286	268
Breaking point, Fraass	°C				-10	-17		-16,5			-8
Softening point	°C	47,2	47,6	49,4	46,8	46,4	47,2	46,8	48,0	47,4	47,7
Flash point, COC	°C		318		320	314	316	326			307
RTFOT											
-change of mass	m-%		0,07	0,071	0,11	0,10		0,08	-0,01	0,08	0,08
-penetration 25°C	1/10mm		42	34,8	38	40		42	41	40	38
retained penetration	%		58	52,73	53	58,0		58	60	53,3	51
-softening point	°C		53,6	55,4	53,7	53,6		53,6	54,5	54,9	55
change in softening point	°C		6,0	6,0	6,9	7,2		6,8	6,5	7,5	7,3

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	71	72	76	75	72	73	72	76	74	68
Viscosity 60°C	Pas	143						146	143,5	145	
Viscosity 135°C	mm ² /s	309		288				328	284	307	320
Breaking point, Fraass	°C	-14,5							-23	-23	
Softening point	°C	46,4	46,9	49,8	47,0	46,8		45,8	47,4	46,8	
Flash point, COC	°C						310		321	318	
RTFOT											
-change of mass	m-%	0,102						0,07	0,16	0,19	
-penetration 25°C	1/10mm	36						41	42	43	
retained penetration	%	50,7						56,9	55	58	
-softening point	°C	54,0						52,8	54,0	53,8	
change in softening point	°C	7,6						7,0	6,6	7,0	

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	71	51	70	70	72	73	72	69	73	
Viscosity 60°C	Pas					148,7	153			146	
Viscosity 135°C	mm ² /s				295	270,5	283		331	305	
Breaking point, Fraass	°C									-19	
Softening point	°C	46,8	46,4	47,6	46,4	48,2	47,1	47,2	48,1	48,2	
Flash point, COC	°C					>300	332			325	
RTFOT											
-change of mass	m-%					-0,05	0,17		0,0811	0,08	
-penetration 25°C	1/10mm					42	44		36	45	
retained penetration	%					58	60,27		52	62	
-softening point	°C					54,2	52,9		54,3	54,2	
change in softening point	°C					6,0	5,8		6,2	6,0	

Auli Kettunen

30 May 2006

Table 3 Bitumen B150

Laboratory		1	2	3	4	5	6	7	8	9	10
Penetration 25°C	1/10mm	159	153	159	151	138	156	150	145	159	145
Viscosity 60°C	Pas	46	44,7		46	45,0	45,4	45	46	47	45,1
Viscosity 135°C	mm ² /s	176	182		171	169	170	171	168	164	158
Breaking point, Fraass	°C				-14	-18		-19,5			-15
Softening point	°C	40,9	41,2	42,4	39,6	40,2	40,2	40,6	41,2	40,6	40,7
Flash point, COC	°C		313		312	318	318	322			302
RTFOT											
-change of mass	m-%		0,06	0,143	0,09	0,06		-0,09	0,04	0,09	0,09
-penetration 25°C	1/10mm		74	71	68	71		76	70	74	67
retained penetration	%		48	44,90	45	51,4		51	48	46,5	46
-softening point	°C		47,7	47,8	47	46,0		46,6	46,2	47,7	48,9
change in softening point	°C		6,5	5,4	7,4	5,8		6,0	5,0	7,1	8,2

Laboratory		11	12	13	14	15	16	17	18	19	20
Penetration 25°C	1/10mm	154	152	168	160	158	148	153	159	149	155
Viscosity 60°C	Pas	45,2						46	46,4	46,6	
Viscosity 135°C	mm ² /s	184		173				184	169	179	176
Breaking point, Fraass	°C	-15							-24	-20	
Softening point	°C	40,0	40,4	42,4	39,8	40,2		38,8	40,8	40,2	
Flash point, COC	°C						304		312	314	
RTFOT											
-change of mass	m-%	0,086						0,03	0,10	0,05	
-penetration 25°C	1/10mm	67						76	77	75	
retained penetration	%	43,5						49,7	48	50	
-softening point	°C	46,4						45,6	47,6	47,0	
change in softening point	°C	6,4						6,8	6,8	6,8	

Laboratory		21	22	23	24	25	26	27	28	29	30
Penetration 25°C	1/10mm	151	121	153	142	157	148	159	152	173	
Viscosity 60°C	Pas					48,57	45,5				
Viscosity 135°C	mm ² /s				176	170,5	161		196		
Breaking point, Fraass	°C										
Softening point	°C	39,8	40,2	41,2	40,4	40,5	40,3	40,2	39,8	41,2	
Flash point, COC	°C						296			322	
RTFOT											
-change of mass	m-%					-0,07	0,32		0,074		
-penetration 25°C	1/10mm					76	76		70		
retained penetration	%					48	51,35		46		
-softening point	°C					48	47		46,7		
change in softening point	°C					7,5	6,7		6,9		

Auli Kettunen

30 May 2006

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)

Auli Kettunen

30 May 2006

Table 4 Evaluation of the results

		Average	Maximum difference of the results	Allowed maximum difference of the method (number of laboratories)	Acceptance
Bitumen B70					
Penetration 25°C	1/10mm	71	25	10,9 % from av. = 8 (29)	no
Viscosity 60°C	Pas	144	19	17,1 % from av. = 24,7 (16)	yes
Viscosity 135°C	mm ² /s	296	63	10,7 % from av. = 32 (20)	no
Breaking point, Fraass	°C	-16	15	9 (8)	no
Softening point	°C	47,3	4,0	3,6 (27)	no
Flash point, COC	°C	319	25	28 (11)	yes
RTFOT					
change of mass	m-%	0,09	0,24	0,34 ¹⁾ (16)	yes
penetration 25°C	1/10mm	40	10,2	no repeatability given (16)	
retained penetration	%	56	11	17 (16)	yes
softening point	°C	54,0	2,6	no repeatability given (16)	
change in softening point	°C	6,7	1,8	6,8 (16)	yes
Bitumen B150					
Penetration 25°C	1/10mm	153	52	10,9 % from av. = 17 (29)	no
Viscosity 60°C	Pas	45,9	3,9	17,1 % from av. = 7,9 (15)	yes
Viscosity 135°C	mm ² /s	174	38	10,7 % from av. = 19 (19)	no
Breaking point, Fraass	°C	-18	10	9 (7)	no
Softening point	°C	40,5	3,6	3,6 (27)	yes
Flash point, COC	°C	312	26	28 (11)	yes
RTFOT					
change of mass	m-%	0,07	0,41	0,34 ¹⁾ (15)	no
penetration 25°C	1/10mm	73	10	no repeatability given (15)	
retained penetration	%	48	8	17 (15)	yes
softening point	°C	47,1	3,3	no repeatability given (15)	
change in softening point	°C	6,6	3,2	6,8 (15)	yes

¹⁾ The reproducibility is specified between 0,3 m-% and 0,8 m-%, (% absolute)