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26 May 2015

Distribution: Participants

## **BITUMEN ROUND ROBIN 1-2015**

### **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 B50

Bitumen B200

### **Test methods**

The test methods used are given in table 1.

### **Participating Laboratories**

|         |                                   |
|---------|-----------------------------------|
| Denmark | NCC Roads Kemi                    |
| Estonia | Lemminkäinen/Tallinn              |
|         | Nynas/Tallinn                     |
|         | Teede Tehnokeskus                 |
| 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 Asfalt                    |
|         | Lemminkäinen/ Fjellhamar          |
|         | Lemminkäinen/Greåker              |
|         | Lemminkäinen/Olsvik               |
|         | Lemminkäinen/Sem                  |
|         | Petrotest                         |
|         | Statens vegvesen                  |
|         | Veidekke Industri                 |
|         | Veiteknisk institutt              |
| Sweden  | Lemminkäinen/Sundsvall            |

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|    |                                         |
|----|-----------------------------------------|
|    | NCC Roads/ Biskopstorp                  |
|    | NCC Roads/ Hisings Kärra                |
|    | 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/ Helsingborg                |
|    | PEAB Asfalt/ Hisings Backa              |
|    | 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/Eastham Quality Control           |
|    | 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,  $R_{\text{stand}}$ . 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.
- Softening point in both the samples.
- Flash point, COC in the sample B200.
- RTFOT, viscosity ratio at 60°C in the sample B50.

**NB.** According to the penetration method the result should be rounded to the nearest whole number. (EN 1426)

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. (EN 1427)

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)

According to the RTFOT method the retained penetration should be rounded to the nearest whole number. (EN 12607-1)

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**Table 1      Test methods**

| <b>Property</b>           | <b>Standard method</b> |
|---------------------------|------------------------|
| 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 B50**

| Laboratory                |                    | 1     | 2    | 3     | 4    | 5    | 6       | 7    | 8    | 9      | 10    |
|---------------------------|--------------------|-------|------|-------|------|------|---------|------|------|--------|-------|
| Penetration 25°C          | 1/10mm             | 48    | 52   | 52    | 54   | 55   | 52      | 54   | 52   | 52     | 53    |
| Viscosity 60°C            | Pas                | 307   | 286  | 264   | 303  |      | 271,734 | 264  |      |        | 239,3 |
| Viscosity 135°C           | mm <sup>2</sup> /s | 396   | 392  | 378   | 368  | 374  | 369,372 | 359  | 332  |        | 333,1 |
| Breaking point, Fraass    | °C                 | -15   |      | -13   | -12  |      | -11     | -16  |      |        | -13   |
| Softening point           | °C                 | 50,4  | 49,0 | 51,4  | 51,0 | 50,4 | 52,6    | 50,8 | 51,6 | 51,6   | 51,4  |
| Flash point, COC          | °C                 |       |      | 322   | 330  |      | 318     | 322  |      |        |       |
| RTFOT                     |                    |       |      |       |      |      |         |      |      |        |       |
| -change of mass           | m-%                | 0,07  |      | 0,08  | 0,09 |      | 0,05    | 0,09 |      |        | 0,02  |
| -penetration 25°C         | 1/10mm             | 29    |      | 32    | 34   |      | 29      | 34   |      |        | 30    |
| retained penetration      | %                  | 60,4  |      | 62    | 63   |      | 56      | 63   |      |        | 57    |
| -softening point          | °C                 | 58,0  |      | 59,0  | 58,4 |      | 60,6    | 58,0 |      |        | 59,2  |
| change in softening point | °C                 | 7,6   |      | 7,6   | 7,4  |      | 8,0     | 7,2  |      |        | 7,8   |
| -viscosity 60°C           | Pas                | 1086  |      | 1137  | 1160 |      |         | 1232 |      |        |       |
| viscosity ratio at 60°C   |                    | 3,5   |      | 4,3   | 3,8  |      |         | 4,7  |      |        |       |
| Laboratory                |                    | 11    | 12   | 13    | 14   | 15   | 16      | 17   | 18   | 19     | 20    |
| Penetration 25°C          | 1/10mm             | 54    | 53   | 56    | 53   | 53   | 54      | 52   | 54   | 53     | 55    |
| Viscosity 60°C            | Pas                |       | 279  | 265   |      | 261  | 262     | 273  | 270  |        | 248   |
| Viscosity 135°C           | mm <sup>2</sup> /s |       |      |       |      | 362  | 401     | 365  | 364  | 401    | 368   |
| Breaking point, Fraass    | °C                 | -15,5 | -14  | -16   |      | -18  | -16     | -11  | -13  |        |       |
| Softening point           | °C                 | 50,9  | 50,2 | 50,8  | 50,4 | 50,8 | 50,8    | 50,4 | 50,4 | 51,6   | 51,2  |
| Flash point, COC          | °C                 |       | 316  | 330   | 323  | 318  | 316     | 322  |      |        |       |
| RTFOT                     |                    |       |      |       |      |      |         |      |      |        |       |
| -change of mass           | m-%                | 0,08  |      | 0,12  |      | 0,08 | 0,03    | 0,12 |      | 0,0087 | 0,09  |
| -penetration 25°C         | 1/10mm             | 31    |      | 32    |      | 32   | 33      | 31   |      | 31     | 32    |
| retained penetration      | %                  | 57,41 |      | 57    |      | 60   | 61      | 60   |      | 58,5   | 58    |
| -softening point          | °C                 | 60,2  |      | 58,0  |      | 58,8 | 59,4    | 59,4 |      | 60,2   | 58,4  |
| change in softening point | °C                 | 9,3   |      | 7,2   |      | 8    | 8,6     | 9,0  |      | 8,6    | 7,2   |
| -viscosity 60°C           | Pas                |       |      |       |      | 1130 | 1313    | 1427 |      |        | 980   |
| viscosity ratio at 60°C   |                    |       |      |       |      | 4,3  | 5,0     | 5,2  |      |        | 4,0   |
| Laboratory                |                    | 21    | 22   | 23    | 24   | 25   | 26      | 27   | 28   | 29     | 30    |
| Penetration 25°C          | 1/10mm             | 49    | 55   | 48,84 | 53   | 53   | 51      | 57   | 53   | 50     | 52    |
| Viscosity 135°C           | mm <sup>2</sup> /s |       |      |       |      |      |         |      |      |        |       |
| Softening point           | °C                 | 51,8  | 51,8 | 51,15 | 50,8 | 51,4 | 50,4    | 50,2 | 52,6 | 54,6   | 50,8  |
| Breaking point, Fraass    | °C                 |       |      |       | -15  |      |         |      |      |        |       |
| Flash point, COC          | °C                 |       |      |       |      |      |         |      |      |        |       |
| Laboratory                |                    | 31    | 32   | 33    | 34   | 35   | 36      | 37   | 38   | 39     | 40    |
| Penetration 25°C          | 1/10mm             |       | 56   | 53    |      | 50   | 52      | 51   | 55   | 45     | 46    |
| Softening point           | °C                 | 50,4  | 50,0 | 49    |      | 52,2 | 50,2    | 51,6 | 50,2 |        | 53    |
| Laboratory                |                    | 41    | 42   | 43    | 44   | 45   | 46      | 47   | 48   | 49     | 50    |
| Penetration 25°C          | 1/10mm             | 52    | 54   | 53    | 53   | 47   | 42,8    | 53   | 43   | 44     |       |
| Softening point           | °C                 | 53,8  | 48,9 | 51,4  | 52,6 | 51,8 | 51,6    | 48,8 |      |        |       |

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**Table 3 Bitumen B200**

| Laboratory                |                    | 1     | 2    | 3      | 4    | 5    | 6       | 7    | 8    | 9     | 10    |
|---------------------------|--------------------|-------|------|--------|------|------|---------|------|------|-------|-------|
| Penetration 25°C          | 1/10mm             | 167   | 168  | 171    | 171  | 183  | 180     | 184  | 176  | 172   | 172   |
| Viscosity 60°C            | Pas                | 42,1  | 42   | 41     | 42,5 |      | 41,011  | 40,8 |      | 40,7  | 36,3  |
| Viscosity 135°C           | mm <sup>2</sup> /s | 171   | 174  | 168    | 159  | 168  | 169,514 | 155  | 167  |       | 149,5 |
| Breaking point, Fraass    | °C                 | -19   |      | -19    | -21  |      | -17     | -19  |      |       | -22   |
| Softening point           | °C                 | 39,6  | 39   | 40,0   | 39,0 | 38,4 | 41,2    | 38,8 | 39,4 | 39,4  | 40,6  |
| Flash point, COC          | °C                 |       |      | 324    |      |      | 318     | 314  |      |       |       |
| RTFOT                     |                    |       |      |        |      |      |         |      |      |       |       |
| -change of mass           | m-%                | 0,10  |      | 0,07   | 0,06 |      | 0,03    | 0,1  |      |       | 0,06  |
| -penetration 25°C         | 1/10mm             | 82    |      | 81     | 80   |      | 82      | 83   |      |       | 81    |
| retained penetration      | %                  | 49,1  |      | 47     | 47   |      | 46      | 45   |      |       | 47    |
| -softening point          | °C                 | 45,6  |      | 47,2   | 45,4 |      | 48,8    | 45,2 |      |       | 47,2  |
| change in softening point | °C                 | 6,0   |      | 7,2    | 6,4  |      | 7,6     | 6,4  |      |       | 6,7   |
| -viscosity 60°C           | Pas                | 114   |      | 121    | 114  |      |         | 105  |      |       |       |
| viscosity ratio at 60°C   |                    | 2,7   |      | 3,0    | 2,7  |      |         | 2,6  |      |       |       |
| Laboratory                |                    | 11    | 12   | 13     | 14   | 15   | 16      | 17   | 18   | 19    | 20    |
| Penetration 25°C          | 1/10mm             | 177   | 175  | 183    | 176  | 190  | 186     | 180  | 177  | 175   | 176   |
| Viscosity 60°C            | Pas                |       | 40,8 | 39,4   |      | 39,9 | 40,0    | 41,3 | 41   |       | 39    |
| Viscosity 135°C           | mm <sup>2</sup> /s |       |      |        |      | 165  | 177     | 164  | 167  | 171   | 169   |
| Breaking point, Fraass    | °C                 | -21,8 | -19  | -23    |      | -24  | -20     | -19  | -24  |       |       |
| Softening point           | °C                 | 40    | 39,0 | 40,2   | 39,0 | 39,4 | 38,8    | 39,6 | 39,2 | 40,2  | 39,6  |
| Flash point, COC          | °C                 |       | 304  | 320    | 306  | 316  | 311     | 308  |      |       |       |
| RTFOT                     |                    |       |      |        |      |      |         |      |      |       |       |
| -change of mass           | m-%                | 0,01  |      | 0,18   |      | 0,08 | -0,01   | 0,07 |      | 0,083 | 0,10  |
| -penetration 25°C         | 1/10mm             | 75    |      | 95     |      | 87   | 81      | 78   |      | 77    | 88    |
| retained penetration      | %                  | 42,37 |      | 52     |      | 46   | 44      | 43   |      | 44,0  | 50    |
| -softening point          | °C                 | 47,4  |      | 45,4   |      | 46,2 | 46,2    | 46,8 |      | 47,6  | 46,4  |
| change in softening point | °C                 | 7,4   |      | 5,2    |      | 6,8  | 7,4     | 7,2  |      | 7,4   | 6,8   |
| -viscosity 60°C           | Pas                |       |      |        |      | 114  | 129     | 133  |      |       | 108   |
| viscosity ratio at 60°C   |                    |       |      |        |      | 2,9  | 3,2     | 3,20 |      |       | 2,8   |
| Laboratory                |                    | 21    | 22   | 23     | 24   | 25   | 26      | 27   | 28   | 29    | 30    |
| Penetration 25°C          | 1/10mm             | 170   | 183  | 157,45 | 177  | 175  | 178     | 182  | 173  | 177   | 171   |
| Viscosity 135°C           | mm <sup>2</sup> /s |       |      |        |      |      |         |      |      |       |       |
| Softening point           | °C                 | 41,0  | 40,6 | 39,2   | 39,4 | 41,0 | 39,2    | 39,2 | 40,6 | 39,0  | 39,6  |
| Breaking point, Fraass    | °C                 |       |      |        | -22  |      |         |      |      |       |       |
| Flash point, COC          | °C                 |       |      |        |      |      |         |      |      |       |       |
| Laboratory                |                    | 31    | 32   | 33     | 34   | 35   | 36      | 37   | 38   | 39    | 40    |
| Penetration 25°C          | 1/10mm             |       | 175  | 150    |      | 159  | 186     | 172  | 181  | 142   | 148   |
| Softening point           | °C                 | 39,6  | 39,0 | 40     |      | 41,9 | 38,2    | 41,2 | 39,2 |       | 42    |
| Laboratory                |                    | 41    | 42   | 43     | 44   | 45   | 46      | 47   | 48   | 49    | 50    |
| Penetration 25°C          | 1/10mm             | 181   | 185  | 170    | 176  | 171  | 147,4   | 176  | 164  | 165   |       |
| Softening point           | °C                 | 42    | 37,5 | 40,4   | 40,8 | 40,6 | 38,3    | 37,8 |      |       |       |

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**Table 4 Evaluation of the results**

|                           |                    | n  | Average | Standard deviation<br>s | Maximum difference<br>of the results | Reproducibility R in<br>standard | R<br>stand | R<br>calc | Acceptance |
|---------------------------|--------------------|----|---------|-------------------------|--------------------------------------|----------------------------------|------------|-----------|------------|
| <b>Bitumen B50</b>        |                    |    |         |                         |                                      |                                  |            |           |            |
| Penetration 25 °C         | 1/10mm             | 47 | 52      | 3,33                    | 14                                   | 6% from average                  | 3          | 9         | no         |
| Viscosity 60 °C           | Pas                | 14 | 271     | 18,53                   | 67,7                                 | 10% from average                 | 27,1       | 52,4      | no         |
| Viscosity 135 °C          | mm <sup>2</sup> /s | 15 | 371     | 21,06                   | 69,0                                 | 6% from average                  | 22,2       | 59,6      | no         |
| Breaking point, Fraass    | °C                 | 14 | -14     | 2,09                    | 7,0                                  | 6                                | 6          | 6         | yes        |
| Softening point           | °C                 | 45 | 51,1    | 1,18                    | 5,8                                  | 2,0                              | 2,0        | 3,3       | no         |
| Flash point COC           | °C                 | 10 | 322     | 5,08                    | 14                                   | 17                               | 17         | 14        | yes        |
| RTFOT                     |                    |    |         |                         |                                      |                                  |            |           |            |
| change of mass            | m- %               | 13 | 0,07    | 0,04                    | 0,11                                 | 0.20 <sup>1)</sup>               | 0,20       | 0,10      | yes        |
| penetration 25 °C         | 1/10mm             | 13 | 32      | 1,61                    | 5                                    | not given                        |            |           |            |
| retained penetration      | %                  | 13 | 59      | 2,36                    | 7                                    | 10                               | 10         | 7         | yes        |
| softening point           | °C                 | 13 | 59,0    | 0,89                    | 2,6                                  | not given                        |            |           |            |
| change in softening point | °C                 | 13 | 8,0     | 0,71                    | 2,1                                  | 4,0                              | 4,0        | 2,0       | yes        |
| viscosity 60 °C           | Pas                | 8  | 1183    | 138,83                  | 447                                  | not given                        |            |           |            |
| viscosity ratio at 60 °C  |                    | 8  | 4,4     | 0,59                    | 1,7                                  | 20% from average                 | 0,9        | 1,7       | no         |
| <b>Bitumen B200</b>       |                    |    |         |                         |                                      |                                  |            |           |            |
| Penetration 25 °C         | 1/10mm             | 47 | 173     | 10,53                   | 48                                   | 6% from average                  | 10         | 30        | no         |
| Viscosity 60 °C           | Pas                | 15 | 40,5    | 1,52                    | 6,2                                  | 10% from average                 | 4,1        | 4,3       | no         |
| Viscosity 135 °C          | mm <sup>2</sup> /s | 15 | 166     | 7,13                    | 27,5                                 | 6% from average                  | 10,0       | 20,2      | no         |
| Breaking point, Fraass    | °C                 | 14 | -21     | 2,16                    | 7                                    | 6                                | 6          | 6         | yes        |
| Softening point           | °C                 | 45 | 39,7    | 1,06                    | 4,5                                  | 2,0                              | 2,0        | 3,0       | no         |
| Flash point, COC          | °C                 | 9  | 313     | 6,73                    | 20                                   | 17                               | 17         | 19        | no         |
| RTFOT                     |                    |    |         |                         |                                      |                                  |            |           |            |
| change of mass            | m- %               | 13 | 0,07    | 0,05                    | 0,19                                 | 0.20 <sup>1)</sup>               | 0,20       | 0,13      | yes        |
| penetration 25 °C         | 1/10mm             | 13 | 82      | 5,22                    | 20                                   | not given                        |            |           |            |
| retained penetration      | %                  | 13 | 46      | 2,80                    | 10                                   | 10                               | 10         | 8         | yes        |
| softening point           | °C                 | 13 | 46,6    | 1,06                    | 3,6                                  | not given                        |            |           |            |
| change in softening point | °C                 | 13 | 6,8     | 0,68                    | 2,4                                  | 4,0                              | 4,0        | 1,9       | yes        |
| viscosity 60 °C           | Pas                | 8  | 117     | 9,77                    | 28,0                                 | not given                        |            |           |            |
| viscosity ratio at 60 °C  |                    | 8  | 2,9     | 0,23                    | 0,6                                  | 20% from average                 | 0,6        | 0,6       | yes        |

<sup>1)</sup> for 0, 3% <value <0,80%