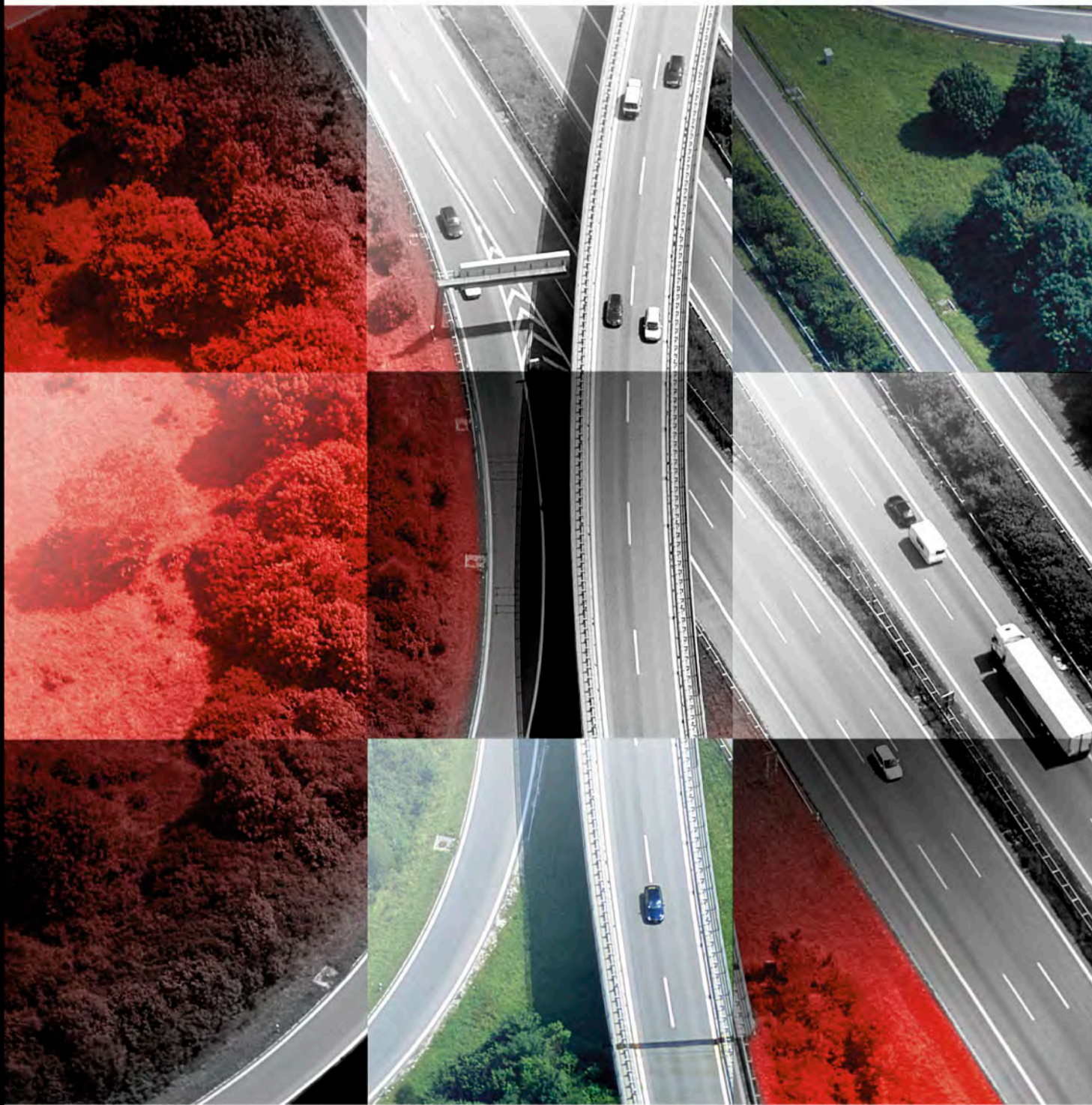
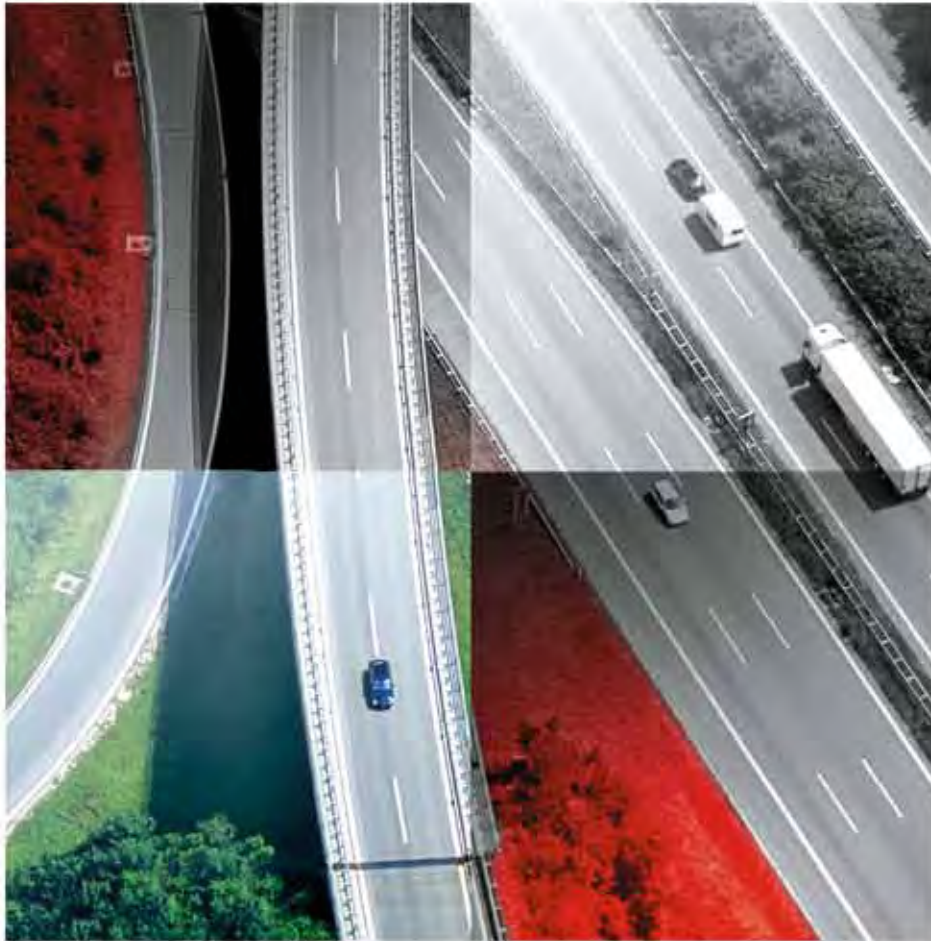


# 2014

## BITUMEN HANDBOOK



BITUMEN HANDBOOK



Krzysztof Błażejowski  
Jacek Olszacki  
Hubert Peciakowski

2014

## **Bitumen Handbook**

Authors:

PhD Eng. Krzysztof Błażejowski

PhD Eng. Jacek Olszacki

M.Sc. Eng. Hubert Peciakowski

Copyright by ORLEN Asphalt sp. z o.o.

ul. Chemików 7

09-411 Płock, Poland

[www.orlen-asfalt.pl](http://www.orlen-asfalt.pl)

2014

Both the Authors and ORLEN Asphalt Sp. z o.o. have exercised due diligence to ensure that the information contained herein is accurate and reliable. However, they shall not be liable for any consequences of the use of information contained in this document, in particular for any loss of any type and form. The reader shall be solely responsible for the use of these data.

# CONTENTS

|   |           |
|---|-----------|
| INTRODUCTION .....  | 6         |
| ABOUT ORLEN Asphalt .....   | 7         |
| <b>1. BITUMEN PRODUCTION .....</b>                                    | <b>10</b> |
| 1.1. Bitumen production technologies at ORLEN Asphalt .....           | 11        |
| 1.1.1. Production of Paving Grade and Multigrade Paving Bitumens..... | 11        |
| 1.1.2. Polymer modification of bitumens .....                         | 12        |
| <b>2. KEY PROPERTIES AND TESTING OF BITUMEN .....</b>                 | <b>14</b> |
| 2.1. Introduction .....   | 14        |
| 2.2. Primary properties.....  | 14        |
| 2.2.1. Penetration at 25°C.....                                       | 14        |
| 2.2.2. Softening Point R&B.....                                       | 15        |
| 2.2.3. Fraass breaking point .....                                    | 16        |
| 2.2.4. Flash Point, Cleveland cup method .....                        | 18        |
| 2.2.5. Solubility .....   | 19        |
| 2.2.6. Plasticity range .....   | 20        |
| 2.2.7. Penetration Index Ip.....                                      | 20        |
| 2.2.8. Viscosity .....  | 21        |
| 2.2.9. Density.....   | 29        |
| 2.2.10. Short-term ageing (RTFOT).....                                | 29        |
| 2.2.11. Properties after RTFOT.....                                   | 31        |
| 2.3. Additional properties of polymer-modified bitumen.....           | 31        |
| 2.3.1. Elastic recovery.....  | 31        |
| 2.3.2. Elastic recovery at 25°C after RTFOT ageing .....              | 33        |
| 2.3.3. Force ductility (cohesion).....                                | 33        |
| 2.3.4. Storage stability of modified bitumens.....                    | 35        |
| 2.3.5. Polymer modified bitumens microstructure .....                 | 36        |
| 2.4. Other bitumen properties.....                                    | 36        |
| 2.4.1. N-heptane insoluble content.....                               | 36        |
| 2.4.2. Paraffin content .....   | 36        |
| 2.4.3. Bitumen adhesion to mineral aggregates.....                    | 37        |
| 2.4.4. PAV ageing .....   | 38        |
| 2.4.5. BBR method.....  | 39        |
| 2.4.6. DSR method.....  | 40        |
| 2.4.7. MSCR method.....   | 42        |
| <b>3. DISCUSSION OF STANDARDS EN 12591 and EN 14023.....</b>          | <b>44</b> |
| 3.1. Introduction .....   | 44        |
| 3.2. Bituminous binder classification .....                           | 44        |
| 3.3. Requirements of EN 12591 and EN 14023 .....                      | 45        |
| 3.4. National application documents.....                              | 45        |
| 3.5. Evaluation of conformity in EN 12591 and EN 14023.....           | 48        |

|  |            |
|--|------------|
| <b>4. PAVING GRADE BITUMENS ACC. TO EN 12591 .....</b>                 | <b>50</b>  |
| 4.1. General description.....  | 50         |
| 4.2. Properties.....   | 51         |
| 4.2.1. Paving Grade Bitumen 20/30 .....                                | 51         |
| 4.2.2. Paving Grade Bitumen 35/50 .....                                | 54         |
| 4.2.3. Paving Grade Bitumen 50/70 .....                                | 57         |
| 4.2.4. Paving Grade Bitumen 70/100 .....                               | 59         |
| 4.2.5. Paving Grade Bitumen 100/150 .....                              | 62         |
| 4.2.6. Paving Grade Bitumen 160/220 .....                              | 64         |
| <b>5. POLYMER MODIFIED BITUMENS ORBITON ACC. TO EN 14023.....</b>      | <b>66</b>  |
| 5.1. General description.....  | 66         |
| 5.2. Intended use.....   | 67         |
| 5.3. Properties.....   | 67         |
| 5.3.1. ORBITON PMB 10/40-65 .....                                      | 67         |
| 5.3.2. ORBITON PMB 25/55-60 .....                                      | 71         |
| 5.3.3. ORBITON PMB 45/80-55 .....                                      | 74         |
| 5.3.4. ORBITON PMB 45/80-55 EXP.....                                   | 77         |
| 5.3.5. ORBITON PMB 45/80-65 .....                                      | 80         |
| 5.3.6. ORBITON PMB 65/105-60 .....                                     | 83         |
| 5.4. Other properties of Polymer Modified Bitumens ORBITON .....       | 86         |
| <b>6. HIGHLY MODIFIED BITUMENS ORBITON HIMA .....</b>                  | <b>88</b>  |
| 6.1. Introduction.....   | 88         |
| 6.2. Operating principle of HiMA .....                                 | 88         |
| 6.3. ORBITON HiMA product family .....                                 | 90         |
| 6.4. ORBITON HiMA test results .....                                   | 90         |
| 6.5. Experimental section in Poland .....                              | 93         |
| 6.6. Conclusion .....  | 93         |
| <b>7. TEST RESULTS OF BITUMEN IN ASPHALT MIXTURES .....</b>            | <b>94</b>  |
| 7.1. Introduction.....   | 94         |
| 7.2. Rutting resistance tests.....                                     | 94         |
| 7.2.1. Comparative testing of all binders with AC 16S .....            | 95         |
| 7.2.2. Comparative tests between individual binders .....              | 96         |
| 7.3. Low-temperature cracking resistance tests .....                   | 103        |
| 7.3.1. TSRST method .....  | 103        |
| 7.3.2. Comparative testing of all binders with AC 16S .....            | 103        |
| 7.4. Conclusions.....  | 105        |
| <b>8. BITUMEN PROPERTIES ACCORDING TO SUPERPAVE .....</b>              | <b>106</b> |
| 8.1. Superpave.....  | 106        |
| 8.2. Low-temperature properties testing.....                           | 107        |
| 8.3. Testing of properties at high and intermediate temperatures ..... | 108        |
| 8.4. PG system adjustment – MSCR test .....                            | 111        |
| 8.5. Classification of ORLEN Asphalt bitumens acc. to PG System .....  | 113        |

|  |            |
|--|------------|
| <b>9. BITUMEN APPLICATION TECHNOLOGY .....</b>                             | <b>116</b> |
| 9.1. Laboratory guidelines .....   | 116        |
| 9.1.1. Determination of process temperatures.....                          | 116        |
| 9.1.2. Bitumen samples at the lab .....                                    | 118        |
| 9.1.3. Bitumen adhesion to mineral aggregates.....                         | 119        |
| 9.2. Bitumen storage .....   | 119        |
| 9.3. Asphalt mixture production .....                                      | 121        |
| 9.4. Asphalt mixture transport .....                                       | 122        |
| 9.5. Placement .....   | 123        |
| 9.6. Roadbase preparation .....  | 123        |
| 9.7. Process temperatures.....   | 124        |
| <br>   |            |
| <b>10. OTHER BITUMEN PROPERTIES .....</b>                                  | <b>125</b> |
| 10.1. Bitumen density.....   | 125        |
| 10.2. Bitumen solubility.....  | 126        |
| 10.3. Data for mechanistic pavement design.....                            | 126        |
| <br>   |            |
| <b>11. QUALITY CONTROL OF BITUMEN DELIVERIES ACC. TO EN ISO 4259 .....</b> | <b>127</b> |
| 11.1. Quality acceptance rules for deliveries acc. to EN ISO 4259.....     | 127        |
| 11.2. Determination of requirements.....                                   | 127        |
| 11.3. Product evaluation by bitumen recipient.....                         | 128        |
| 11.4. Disputes.....  | 129        |
| 11.5. Breaking point example.....  | 129        |
| 11.6. Reproducibility values for selected bitumen properties .....         | 129        |
| <br>   |            |
| <b>12. OCCUPATIONAL SAFETY .....</b>                                       | <b>130</b> |
| 12.1. Introduction.....  | 130        |
| 12.2. Burns (skin, eye contact) .....                                      | 130        |
| 12.3. Fire.....  | 131        |
| 12.4. Foaming in the presence of water .....                               | 132        |
| 12.5. Bitumen vapours (bitumen mist, smoke) .....                          | 132        |
| 12.5.1. Hydrogen sulphide.....   | 133        |
| <br>   |            |
| <b>LITERATURE .....</b>  | <b>134</b> |
| <br>   |            |
| <b>AUTHORS OF THE BITUMEN HANDBOOK .....</b>                               | <b>135</b> |



## INTRODUCTION

### Dear Readers

It is our pleasure to submit to you our technical publication *Bitumen Handbook 2014* – prepared and published by ORLEN Asphalt in English. This is the 6th edition of the Bitumen Handbook series. Four editions were published in Polish in 2007, 2009, 2011 and 2014, and one in Romanian in 2013.

*The Bitumen Handbook* is a structured compilation based on our expertise in bitumens, which also provides information about their effective application. It is the outcome of work and the experience of our Technology, Research and Development Department, and is prepared for those dealing with bituminous mixture design, production and placement in their everyday work. The contents of this publication and the way the information is provided may also prove useful for those who are interested in the current bitumen offering of ORLEN Asphalt and bitumen properties. This publication also outlines the current standardisation status of bitumens for road pavements.

We hope that this Handbook will address the ever-growing need for expertise concerning the application of bitumens not only in Poland, but also in other countries of Central and Eastern Europe, and provide a valuable insight into bituminous materials for road construction. ORLEN Asphalt is always ready to cooperate and help in selecting the products best suited to your specific requirements.

We are proud that we can share this valuable collection of information with you.

## ABOUT ORLEN Asphalt

### About us

ORLEN Asphalt Sp. z o.o. is a member of the PKN ORLEN SA Group, and one of Poland's largest bitumen manufacturers and sellers. The company was registered under the name ORLEN Asphalt on 21 July 2003. At present the only shareholder of the company is PKN ORLEN SA.

ORLEN Asphalt is the sole shareholder of a Czech company ORLEN Asphalt Česká republika, formed following the acquisition of all shares in Paramo Asphalt, selling bitumen manufactured by refineries in Litvinov and Pardubice. 17 April 2013 saw the official registration of the company's Romanian branch – ORLEN Asphalt Sp. z o.o. PLOCK – Sucursala Bucuresti, helping the company to continue its robust development on southern markets.

The core business of our company has always been the production of and trade in paving-grade bitumens, ORBITON polymer-modified bitumens, Bitrex multigrade bitumens and industrial bitumens. ORLEN Asphalt applies state-of-the-art technology solutions as a reliable manufacturer of bitumens, and is perfectly positioned to supply its products for major road projects, harnessing its modern and efficient installations, operated by qualified personnel.

### Quality above all

We offer premium product and service quality to our customers. Our products are manufactured to European standards. ORLEN Asphalt is a benchmark example of a modern business which focuses not only on the production process control and product quality improvement, but also cares about customer service quality.

With our customers' needs in mind and out of responsibility for the quality of bituminous pavements, we provide our customers with technical consultancy on bitumens – their selection, properties and applications.

We strive to embrace all aspects of our operations with our motto "Bitumens – quality in every detail". Our customers are provided with both unrivalled product quality and top customer service standards.

We are a transparent company and all our operations follow the rules of corporate governance in place at the PKN ORLEN SA Group, as well as the principles of corporate social responsibility, supporting the development of our staff and protecting the natural environment.

Since 2005 we have been using an integrated Management System based on ISO 9001, ISO 14001 and OHSAS 18001 standards.



## Market recognition

The high quality of products on offer from ORLEN Asphalt is confirmed by numerous honours and distinctions awarded by recognised industry organisations and the media. Positive recognition of our products began in 2004 with a distinction for our ORBITON elastomer-modified bitumens in the EUROPRODUCT competition, organised under the auspices of the Polish Minister of Economy and the Polish Agency for Enterprise Development. ORBITON polymer-modified bitumens were also awarded the Gold Medal at the 11th International Road Construction Fair Autostrada-Polska, and the “High Level” prize in the “Proven Product” category, awarded by “Magazyn Autostrady” magazine and the Polish Association of Transport Engineers and Technicians for outstanding achievements and products. In 2011 ORLEN Asphalt was honoured with the QI Golden Emblem for top product quality awarded in the programme implemented under the auspices of the Polish Minister of Regional Development, the Polish Agency for Enterprise Development and the Polish ISO 9000 Forum Club.

The company was also twice honoured with the “Construction Company of the Year” title. Our BITREX multigrade bitumen production technology also won the market’s recognition, winning the Gold Medal at the International Invention Fair IWIS 2007.

## Our products

Our current offer comprises paving-grade bitumens, ORBITON polymer-modified bitumens, BITREX multigrade bitumens and oxidised (industrial) bitumens. Following the consolidation of the bitumen segment, our product offering was extended to include bitumens from the Czech Republic (Pardubice and Litvínov production centres) and Lithuania (Mažeikiai production centre).



## Bitumen products by production location:

| Type of bitumen            | Płock   | Trzebinia   | Litvínov                   | Pardubice   | Mazeikiai                                      |
|----------------------------|---|---|----------------------------|---|--|
| Paving Grade Bitumens      | 20/30<br>35/50<br>50/70<br>70/100<br>100/150<br>160/220   | 20/30<br>35/50<br>50/70<br>70/100<br>100/150<br>160/220   | 50/70<br>70/100<br>160/220 | 20/30<br>30/45<br>35/50<br>50/70<br>70/100<br>160/220 | 35/50<br>50/70<br>70/100<br>100/150<br>160/220 |
| Polymer Modified Bitumens  | ORBITON 10/40-65<br>ORBITON 25/55-55 EXP<br>ORBITON 25/55-60<br>ORBITON 45/80-55<br>ORBITON 45/80-55 EXP<br>ORBITON 45/80-65<br>ORBITON 65/105-60 | ORBITON 10/40-65<br>ORBITON 25/55-60<br>ORBITON 25/55-60 EXP<br>ORBITON 25/55-65 EXP<br>ORBITON 45/80-55<br>ORBITON 45/80-55 EXP<br>ORBITON 45/80-65<br>ORBITON 65/105-60 | —                          | —   | —  |
| Multigrade Paving Bitumens | BITREX 20/30<br>BITREX 35/50<br>BITREX 50/70  | BITREX 35/50<br>BITREX 50/70  | —                          | VMT 25<br>VMT 45<br>VMT 65                            | —  |
| Hard Paving Grade Bitumens | —   | —   | —                          | AP 15 (10/20)<br>AP 25 (20/30)                        | —  |
| Oxidised Bitumens          | 80/15<br>95/35  | 95/35   | —                          | 85/15<br>85/25<br>85/40<br>95/35<br>105/15            | Special Bitumen<br>BNK 40/180                  |



## Chapter 1

# BITUMEN PRODUCTION

ORLEN Asphalt is a manufacturer of several bitumen types: paving-grade bitumens, ORBITON modified bitumens, BITREX multigrade bitumens and oxidised (industrial) bitumens. Bitumen production operations are carried out at multiple production centres:

- in Poland: Płock and Trzebinia,
- in Lithuania: Mažeikių,

all of which have implemented the Integrated Management System based on ISO 9001, ISO 14001 and OHSAS 18001 standards.



Figure 1.1. ISO Certificates for ORLEN Asphalt and ORLEN Lietuva

In accordance with European requirements, ORLEN Asphalt and ORLEN Lietuva have implemented the Factory Production Control (FPC) system, and all production sites (Płock, Trzebinia and Mažeikių) hold FPC certificates for the paving-grade bitumens they produce (Table 1.1).

Table 1.1. List of FPC Certificates for the Production Sites.

| Bitumen type  | FPC Certificate Number      |                             |                             |                                  |                  |
|---|-----------------------------|-----------------------------|-----------------------------|----------------------------------|------------------|
|   | Site in Płock               | Site in Trzebinia           | Site in Mažeikių            | Site in Pardubice                | Site in Litvínov |
| Paving Grade Bitumens EN 12591  | 1434-CPD-0107 <sup>1)</sup> | 1434-CPD-0108 <sup>1)</sup> | 1567-CPD-0064 <sup>1)</sup> | 1020-CPD-090027697 <sup>1)</sup> | 1023-CPD-0233F   |
| Polymer Modified Bitumens EN 14023  | 1434-CPD-0133 <sup>1)</sup> | 1434-CPD-0134 <sup>1)</sup> | —                           | —                                | —                |
| Multigrade Paving Bitumens <sup>2)</sup> (National certificates or EN 13924-2 as of 2014) | F-013-BG-015                | F-013-BG-026                | —                           | 13 0720 SV/AO                    | —                |

1) Certificate designation may change from CPD to CPR following the next update  
 2) National certificate in Poland and the Czech Republic (no EN standard for this bitumen, the European standard for this type (EN 13924-2) is expected to be issued in 2014)

Bitumens from ORLEN Asphalt are manufactured from conventional raw material sources, and specifically from crude oil processing residue. It is vacuum residue coming from the distillation tower plant.

## 1.1. Bitumen production technologies at ORLEN Asphalt

### 1.1.1. Production of Paving Grade and Multigrade Paving Bitumens

ORLEN Asphalt manufactures Paving Grade Bitumens and BITREX Multigrade Paving Bitumens using systems where vacuum residue from crude oil processing is oxidised. They are classified as <sup>1</sup> *semi-blown or air-rectified* bitumens.

Bitumen oxidation is a very complex chemical and physical process. In terms of chemical processes, it involves intense polymerisation and condensation leading to particle growth. Chemical reactions taking place concurrently produce oxygen compounds, with the associated dehydrogenative condensation causing C-C (carbon-carbon) bonds to appear. This produces resins and asphaltenes at the expense of naphthenic aromatic hydrocarbons. The reaction types and mechanism depend on the reaction's temperature. The physical nature of the reaction is evidenced by the stripping of lighter hydrocarbons from the liquid to the gaseous phase through steam distillation. It is an exothermic process, which means that heat is produced by the reaction.

#### 1.1.1.1. Biturox technology vacuum residue continuous oxidation system

Paving Grade and BITREX Multigrade Paving Bitumens are manufactured by ORLEN Asphalt mainly in the continuous oxidation process based on BITUROX® technology, licensed from Austrian company Pörner. The process involves continuous, uninterrupted feedstock batching to the reactors and continuous discharge of products to storage tanks. The process continuity ensures product homogeneity. The process is also marked by optimum utilisation of oxygen for oxidation and excellent hydrodynamic properties of the reaction.

The heart of the system is composed of Biturox® reactors. They are cylindrical, vertical pressure vessels fitted with a central cylinder and an agitator with three turbines on a single shaft, placed inside the cylinder. Air forming large bubbles moves upwards inside the cylinder, and is collected at two levels by coalescing plates and broken into smaller bubbles by the agitator's turbines. This renews the reaction surfaces. Intense oxidation takes place in the entire volume, using less air and over a shorter residence time. The air flow is selected so that the quantity of oxygen in the off gas is 2÷5% (v/v)<sup>2</sup>. Air movement and operation of the agitator produce liquid circulation in the reactor – an upward movement in the inner cylinder, and a downward movement in the external cylinder space. The heat of oxidation is collected from the reactor by the evaporation of process water, injected directly to process air immersion pipes. The quantity of process water precisely controls the process temperature. The resultant steam helps to remove undesirable by-products from the bitumen, such as gases and light oxidised distillate and increases production safety. Bitumen is discharged from the external cylinder space of the reactor, from the level above the feedstock inlet, and is cooled in bitumen coolers. Subsequently, it is sent to bitumen storage tanks, where it is mixed and tested for quality. Bitumen distribution to road and rail tankers takes place at sealed loading stations. The process is controlled by the distributed DCS system.

1) According to Eurobitume's nomenclature, paving-grade bitumens manufactured by ORLEN Asphalt are "semi-blown" or "air-rectified" bitumens, with the Penetration Index lesser than or equal to 2.0, whereas industrial bitumens are ("oxidised") bitumens, and their Penetration Index is greater than 2.0 [source: *Physical differentiation between air-rectified and oxidised bitumens. Technical Committee Task Force. Eurobitume, 15.04.2011*]

2) (v/v) means volumetric proportions, whereas (m/m) stands for weight proportions



Figure 1.2. Continuous oxidation system – Biturox® reactors (photo by ORLEN Asphalt)

#### 1.1.1.2. Vacuum residue periodic oxidation system – oxidisers

The periodic oxidation plant at ORLEN Asphalt is primarily used for the production of industrial-grade bitumens (*oxidised bitumens*) and special bitumens, but it can just as well be used for paving-grade and multigrade bitumens.

Unlike Biturox® reactor oxidation, the production in the oxidisers is a batch process, involving the batching of the feedstock to the oxidiser, oxidation of the feedstock and pressing of the finished product. The oxidiser is less sophisticated in terms of technology than the Biturox® reactor.

### 1.1.2. Polymer modification of bitumens

Bitumen modification is designed to increase the temperature range in which bitumen demonstrates viscoelastic properties. ORLEN Asphalt applies the physical method for modification, involving mutual, mechanical mixing of bitumen and polymer, with potential addition of crosslinkers.

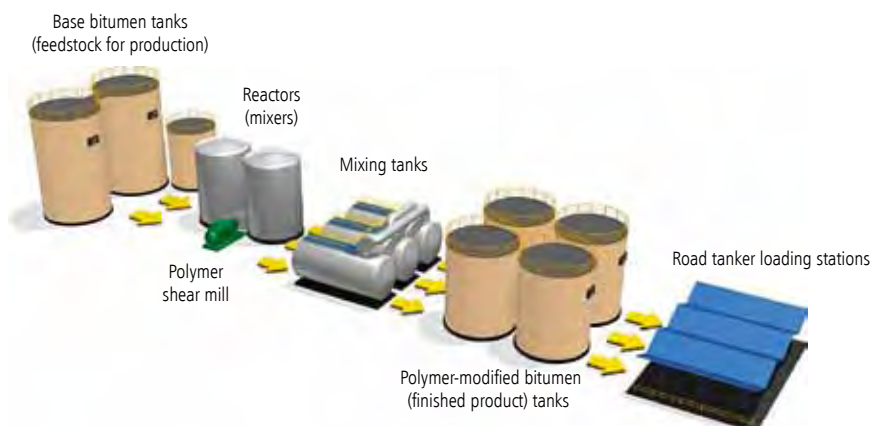
#### 1.1.2.1. Bitumen polymer modification system

The primary feedstock for polymer-modified bitumen production at ORLEN Asphalt comprises special bitumens, so-called base bitumens, whose properties are appropriate for SBS elastomer modification. The modifier added to bitumen in the production process is usually Styrene-Butadiene-Styrene block copolymer (SBS), and therefore this bitumen type is also referred to as *elastomer-modified bitumen*.

Modified bitumen production involves the insertion of the polymer into hot bitumen, milling of the mixture in a high-shear mill and its final dissolution and homogenisation.

The production technology was developed and is supervised by ORLEN Asphalt's Technology, Research and Development Department, which also develops formulas for new products.

ORLEN Asphalt operates two polymer-modified bitumen production lines – in Plock (launched in 2006) and in Trzebinia. Figure 1.3 shows the diagram for the Plock site's bitumen modification system.



**Figure 1.3.** Plock site bitumen modification system diagram

Both systems (Plock and Trzebinia sites) are automatically controlled. The Plock system is controlled by DCS (Figure 1.4), which enables full production process control and historical process data readout.



**Figure 1.4.** DCS system for the Plock site ORBITON modified bitumen production (photo by ORLEN Asphalt)

The application of elastomer (modifier) in the production process helps to achieve substantial benefits in terms of bitumen properties at both high and low temperatures. Polymer-modified bitumens are used for bituminous pavements carrying heavy traffic, at particular locations – such as bridge decks, and for special bituminous mixtures, such as porous asphalt mix. Performance of this bitumen type markedly exceeds that of conventional paving-grade bitumen in the applications mentioned above.

More details are provided in the chapters on:

- ORBITON modified bitumens (Chapter 5),
- Bituminous mixture test results (Chapter 7),
- *Superpave* test results (Chapter 8).

## Chapter 2

# KEY PROPERTIES AND TESTING OF BITUMEN

### 2.1. Introduction

This chapter lists and briefly discusses key properties of bitumen and their testing methods. The authors' intention is to help readers analyse the relevant standards and technical documents.

Photographs in this chapter were made at an accredited laboratory owned by ORLEN Laboratorium sp. z o.o., supporting bitumen production control for the Plock plant.

### 2.2. Primary properties

#### 2.2.1. Penetration at 25°C

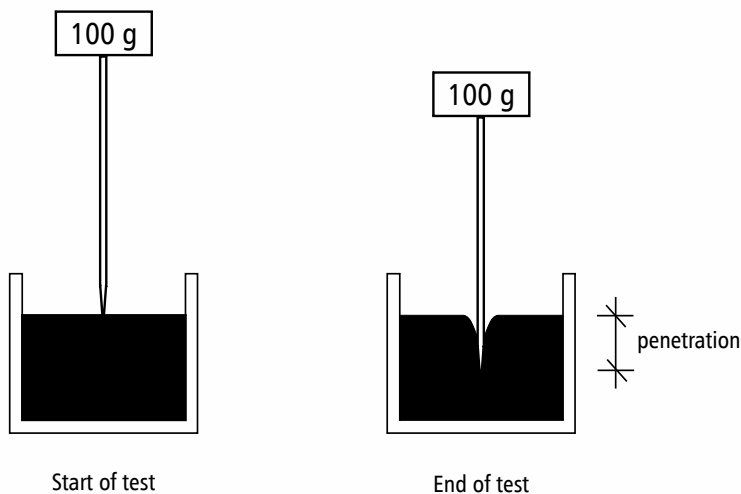
The primary method for bitumen classification in the European standardisation framework is the penetration test at 25°C.

The test involves the determination of bitumen consistency expressed as the distance that a standardised steel needle penetrates vertically into a bitumen sample at a specific temperature. The needle load is 100 g, and the loading time equals 5 seconds. The penetration unit is [0.1 mm], which corresponds to the needle penetration depth in the bitumen sample.

Interpretation of the results is easy: e.g. we know that bitumen with a penetration of 200 [0.1 mm] is softer than bitumen with a penetration of 100 [0.1 mm] since, in the former case, the needle penetration depth is 20 mm, while in the latter it is 10 mm. In general terms, the greater the penetration, the softer the bitumen.

The test can be conducted at various temperatures, although 25°C is adopted for bitumen classification purposes.

The penetration test is conducted according to PN-EN 1426 *Bitumen and bituminous binders. Determination of needle penetration*.



**Figure 2.1.**  
Penetration test principle



**Figure 2.2.** General view of the penetration test equipment with a bitumen sample (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)



**Figure 2.3.** View of a bitumen sample after the test (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

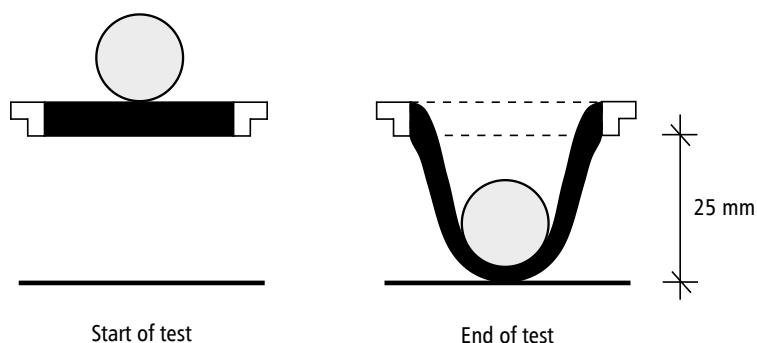
### 2.2.2. Softening Point R&B

The softening point specifies bitumen properties at high service temperatures and represents a conventional, approximate upper limit of the viscoelastic consistency.

The test involves the determination of the “conventional” temperature at which asphalt acquires a specific consistency. Bitumen softening point testing is usually performed by the ring and ball method (R&B in short).

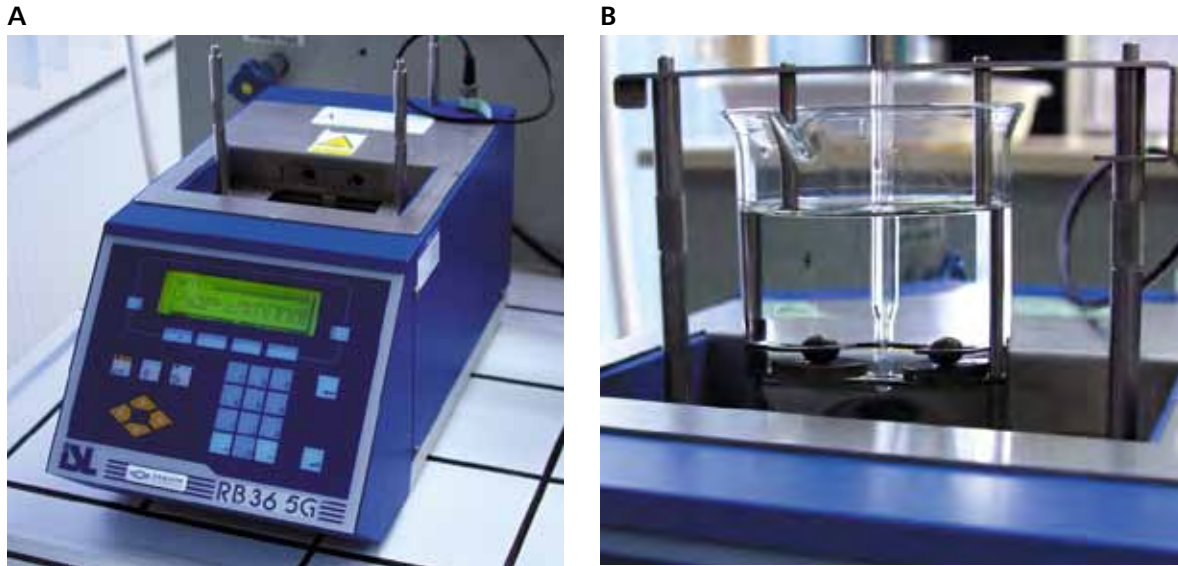
Two bitumen samples placed in metal rings are heated in a controlled manner in a liquid (distilled water for the expected R&B from 28 to 80°C, glycerine for the R&B from 80 to 150°C), placed in a glass beaker, with each bitumen-filled ring supporting a steel ball. The softening point is adopted as the average temperature at which both bitumen rings soften to the point that each ball, covered in bitumen and overcoming its resistance, travels a distance of 25.0 mm ± 0.4 mm. The result of the softening point test is expressed in [°C].

The R&B (ring and ball) softening point test is carried out in accordance with PN-EN 1427 *Bitumen and bituminous binders. Determination of the softening point. Ring and Ball method.*

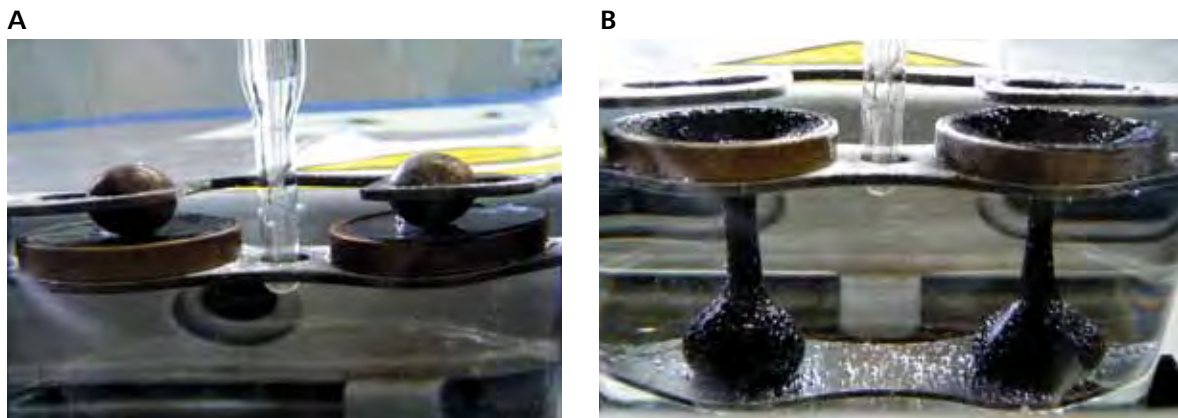


**Figure 2.4.** R&B softening point test principle





**Figure 2.5.** General view of the automatic R&B softening point test equipment with a bitumen sample (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)



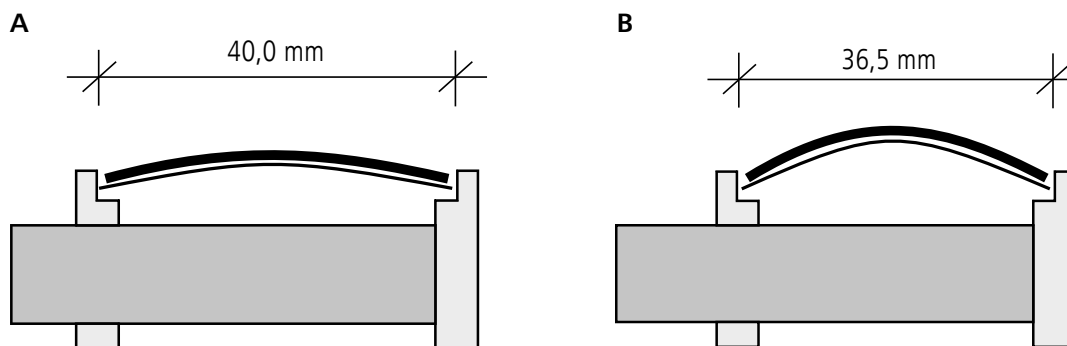
**Figure 2.6.** View of a bitumen sample before and after the test (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

### 2.2.3. Fraass breaking point

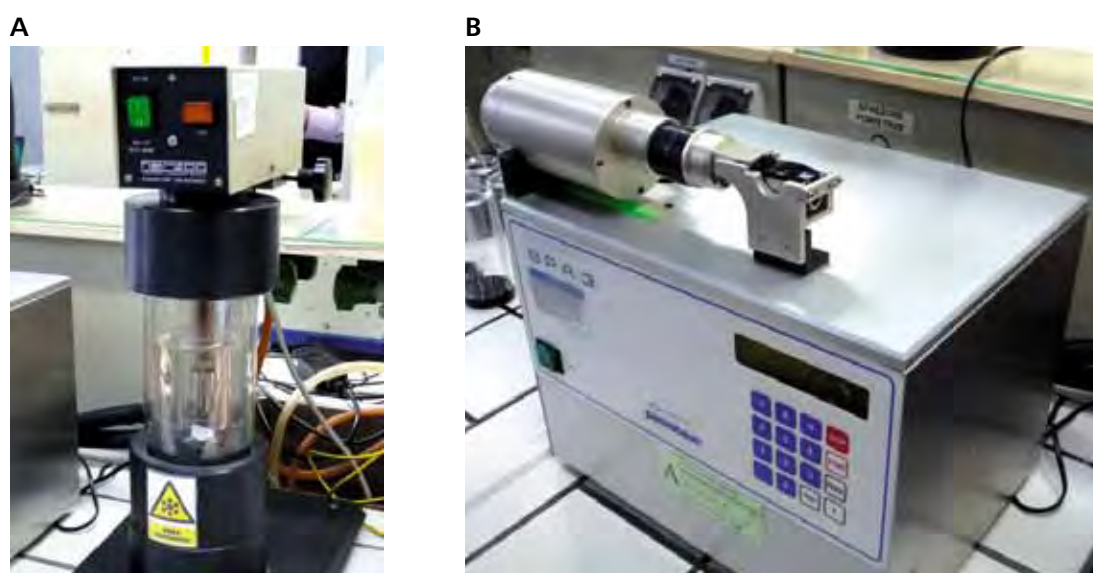
The breaking point determines low-temperature bitumen properties and represents an approximate (conventional) lower limit of viscoelastic consistency.

The breaking point test is conducted according to PN-EN 12593 *Bitumen and bituminous binders. Determination of the Fraass breaking point*. The test proposed by A. Fraass involves the determination of the temperature at which a 0.5 mm thick bitumen layer breaks when spread on a thin, steel plate, 20x41 mm, placed in the test set described below.

The bitumen sample is placed in the test set and subjected to cyclic, mechanical bending and relieving. The bending occurs every 1°C while the air temperature around the sample is constantly decreased at a rate of 1°C/minute. The bitumen layer is examined after each plate bending and any cracks are recorded. The tests ends when the first visible sample crack is observed. The result of the Fraass breaking point is expressed in [°C].



**Figure 2.7.** Fraass breaking point test principle, A) plate with bitumen prior to bending, B) plate with bitumen after bending – the moment of examination whether any bitumen cracks are observed



**Figure 2.8.** General view of the Fraass breaking point test set with a bitumen sample, A) semi-automatic set, B) automatic set (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)



**Figure 2.9.** View of a bitumen sample on the plate placed on the semi-automatic set after the test – a bitumen layer crack can be seen (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

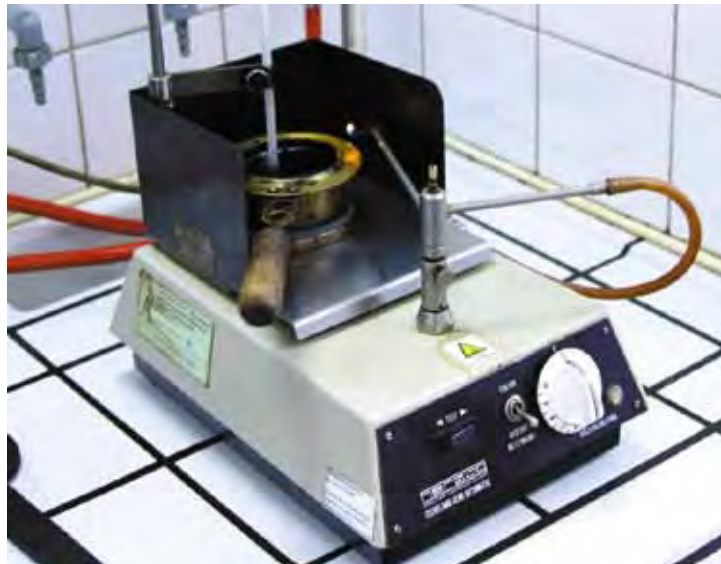
The applicability of the Fraass method has been challenged for many years as it arguably does not translate into the actual behaviour of bitumen in the pavement in winter. Therefore, many countries, such as the US, currently use the BBR method (see Section 2.4.5), or develop new methods for low-temperature binder properties testing (ABCD method or other).

### 2.2.4. Flash Point, Cleveland cup method

Flash point testing belongs to a group of tests related to bituminous binder application safety. This method helps to verify whether the binder contains flammable, volatile components.

Flash point testing is conducted according to PN-EN ISO 2592 *Determination of flash and fire points. Cleveland open cup method*. The test involves the determination of the temperature at which there is instantaneous flash of bitumen sample vapours in an open cup.

Prior to the test, an ambient atmospheric pressure reading is taken from the laboratory barometer and recorded. The cup with a bitumen sample is preheated at a temperature increase rate of  $14 \div 17^\circ\text{C}/\text{min}$ . When the sample temperature is about  $56^\circ\text{C}$  smaller than the expected flash point, bitumen heating is reduced so that the temperature increase rate for the last  $23^\circ\text{C}$  is  $5 \div 6^\circ\text{C}/\text{min}$ . Then, the test flame is passed over the bitumen in the cup until the flame causes the vapour to flash and the flame spreads over the bitumen surface. The flash point, expressed in  $[\text{C}]$  and determined at the ambient atmospheric pressure, is adjusted to standardised atmospheric pressure using the appropriate mathematical equation.



**Figure 2.10.** General view of the Cleveland open cup flash point test equipment (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

There is also a flash point testing method using the so-called Martens-Pensky closed cup [PN-EN ISO 2719:2007]. The values obtained using that method are typically smaller than those obtained from the Cleveland open cup method.

## 2.2.5. Solubility

This test checks whether the binder contains any solid contamination.

The solubility test is conducted according to PN-EN 12592 *Bitumen and bituminous binders. Determination of solubility*. The test involves the determination, in percentage terms (relative to the entire bitumen sample), of that part of bitumen mass which dissolves in a specific solvent.

A bitumen sample is dissolved in the solvent and filtered through a layer of glass powder in a sintered crucible. Undissolved material from bitumen is washed, dried and weighed. Then, the result of the solubility test is calculated, expressed as the percentage mass of the dissolved bitumen fraction relative to the mass of the entire sample [% m/m].

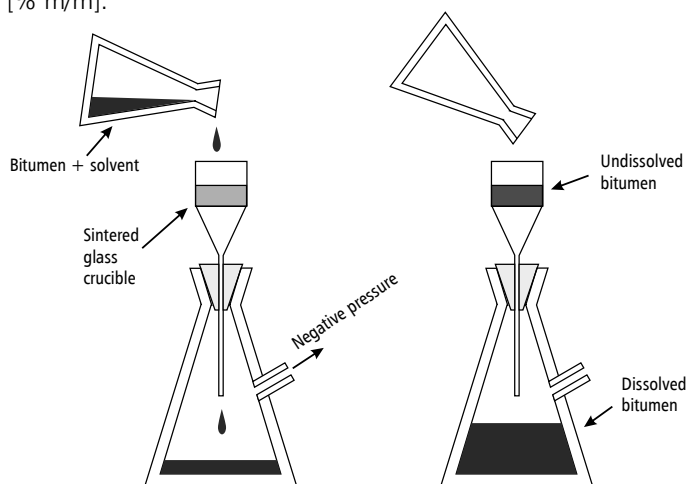


Figure 2.11. Solubility test principle



Figure 2.12.

General view of the solubility test set (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)



Figure 2.13.

View of a bitumen sample after the test (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

Bitumen solubility data can be used to determine bitumen contamination by solids, e.g. coke, and to determine value of “T” as per Section A.4, that is to determine the content of dissolved bitumen after the extraction of the bituminous mixture acc. to PN-EN 12697-1 *Bituminous mixtures. Test methods for hot mix asphalt. Part 1: Soluble binder content*.

The results of bitumen solubility tests are provided in Chapter 9.

### 2.2.6. Plasticity range

The plasticity range is the difference between the Softening Point R&B and the Fraass Breaking Point [°C]. By definition, the plasticity range is the range within bituminous binder retains its viscoelastic properties.

From the standpoint of the binder’s user, the classical bitumen theory emphasises that the range should be as broad as possible, i.e. marked by the lowest possible breaking point and highest possible softening point. In contemporary research, however, the applicability of the breaking point and softening point is relatively often challenged. Instead, testing e.g. by the Dynamic Shear Rheometer (DSR) or the Bending Beam Rheometer (BBR) is proposed as more suitable for the determination of binder properties (see Sections 2.4.5 – 2.4.6).

Therefore:

$$\text{Plasticity range} = T_{R\&B} - T_{Fraass}$$

### 2.2.7. Penetration Index Ip

The Penetration Index Ip is the measure of temperature sensitivity of bitumen, and is calculated using the equation taking the known value of penetration at 25°C (100 g, 5 s), determined in accordance with PN-EN 1426, and the Softening Point R&B determined acc. to PN-EN 1427. The Penetration Index is a dimensionless quantity [-]. The lower the Penetration Index, the faster the binder changes its consistency as the temperature changes (shows greater temperature sensitivity).

The calculation of the Penetration Index acc. to PN-EN 12591 is based on the assumption that bitumen at the softening point R&B has penetration equal to 800 [0.1 mm].

Ip is calculated from the equation provided in Annex A to PN-EN 12591.

$$I_p = \frac{20 \times t_{R\&B} + 500 \times \lg P - 1952}{t_{R\&B} - 50 \times \lg P + 120}$$

$t_{R\&B}$  – R&B softening temperature in degrees Celsius;

$\lg P$  – common logarithm of penetration at 25°C [0.1 mm].

## 2.2.8. Viscosity

Bitumen viscosity is one of the most important process and service properties. There are various definitions of viscosity and its testing methods. This section provides a brief description of viscosity as a phenomenon, the required definitions and conversion factors, as well as gives an overview of the various testing methods.

Bitumens are complex liquids in rheological terms. Their viscosity may vary depending on:

- temperature change,
- shear rate,
- test duration,
- testing method,
- test set used in a given method.

In other words, this means that **the comparability of viscosity as determined by different testing methods can be retained only if specific measurement conditions are strictly complied with (the same temperature, appropriately selected test sets, shear rate, test duration)**. In other cases, the comparison and substitution of viscosity test results is incorrect and may lead to erroneous conclusions.

The resistance of a liquid body to all irreversible changes of position of its volumetric constituents is referred to as **viscosity** [19]. For bitumen, viscosity can be defined as the internal friction between particles when one bitumen layer moves relative to another. The higher the temperature of bitumen, the lower its viscosity [4]. This relationship is used to determine temperatures for bitumen pumping, mixing with aggregate and pavement compaction. Figure 2.14 shows a schematic relationship between bitumen viscosity and its temperature.

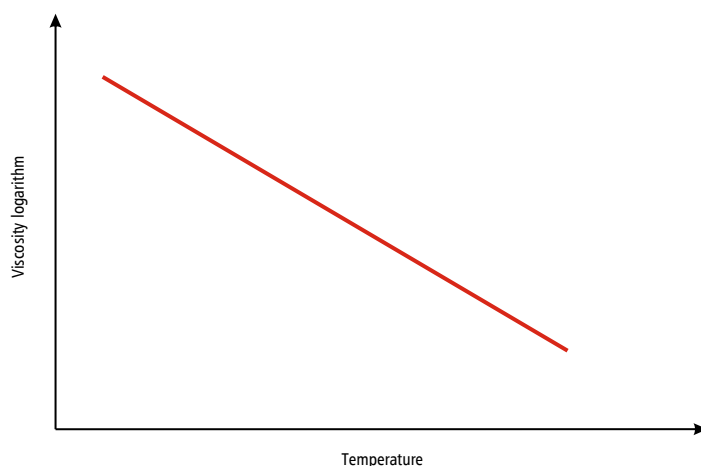


Figure 2.14. Relationship between bitumen viscosity and temperature

### 2.2.8.1. Viscosity measurement principle

Isaac Newton was the first to formulate the primary rule for viscosity measurement, known as Newton's law of viscosity, expressed by the following equation [19]:

$$\tau = \eta \cdot \gamma$$

shear stress = viscosity · shear rate

The model of a rotating spindle immersed in a container holding a bitumen sample, shown in Figure 2.15, helps to define both shear stress and shear rate.

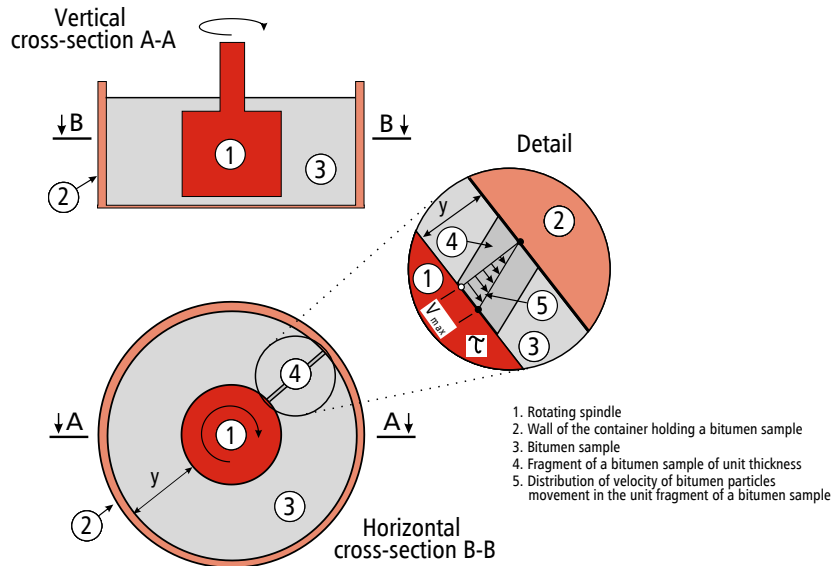


Figure 2.15. Model of the rotating spindle immersed in a container holding a bitumen sample

$$\text{shear stress} \Rightarrow \tau = \frac{F}{A} = \left[ \frac{N}{m^2} \right] = Pa \text{ [paskal]}$$

$$\text{shear rate} \Rightarrow \gamma = \frac{V_{\max}}{y} [s^{-1}]$$

### 2.2.8.2. Types of viscosity and their interrelations

**Dynamic viscosity** is the ratio of shear stress and shear rate. Dynamic viscosity is a measure of liquid flow resistance and is typically referred to as liquid viscosity [PN-EN 12596 *Bitumen and bituminous binders – Determination of dynamic viscosity by vacuum capillary*].

$$\text{dynamic viscosity} \Rightarrow \eta = \frac{\tau}{\gamma} = \left[ \frac{N}{m^2} \cdot s \right] = [Pa \cdot s]$$

The SI dynamic viscosity unit is "Pascal · second" [Pa·s]. "milipascal · second" [mPa·s] is also often used [1].

$$1 Pa \cdot s = 1000 mPa \cdot s$$

Formerly, dynamic viscosity unit from the CGS system was used, namely the "poise" [P], named after French physicist Jean L. M. Poiseuille [10]:

$$1 Pa \cdot s = 10 P$$

"centipoise" [cP] was also used:

$$1 P = 100 cP$$

**Kinematic viscosity**, also referred to as kinetic viscosity, is the ratio of dynamic viscosity and liquid density. Dynamic viscosity is a measure of liquid flow resistance under gravity [PN-EN 12595 *Bitumen and bituminous binders – Determination of kinematic viscosity*]. Kinematic and dynamic viscosity are therefore interrelated.

$$\text{kinematic viscosity} \Rightarrow \nu = \frac{\eta}{\rho} = \left[ \frac{m^2}{s} \right]$$

$$\text{density} \Rightarrow \rho = \frac{kg}{m^3} = \left[ \frac{N \cdot s^2}{m^4} \right]$$

Kinematic viscosity unit is "metre<sup>2</sup>/second" [m<sup>2</sup>/s]. "millimetre<sup>2</sup>/second" [mm<sup>2</sup>/s] is also often used:

$$1 m^2 / s = 1\,000\,000 \text{ mm}^2 / s$$

Formerly, a kinematic viscosity unit known as "Stokes" [St] or "Centistokes" [cSt] was used, named after Irish physicist George Gabriel Stokes [19]:

$$1 \text{ St} = 100 \text{ cSt} \quad \text{and} \quad 1 \text{ St} = 1 \frac{cm^2}{s}$$

"Centistokes" is equivalent to "millimetre<sup>2</sup>/second"

$$1 \text{ mm}^2 / s = 1 \text{ cSt}$$

### 2.2.8.3. Viscosity testing methods

Viscosity testing can be conducted by various methods and instruments, also at various temperatures. Bitumen tests are typically conducted at 60, 90 and 135°C, which corresponds to a specific process temperature (service, rolling and pumping). Viscosity testing at ORLEN Asphalt is often conducted at 160°C or 180°C if the test is performed on modified bitumen.

Characteristics of test sets and rules for kinematic and dynamic viscosity testing are provided below for the selected, frequently used methods:

- kinematic viscosity testing with BS/IP/RF viscometer,
- dynamic viscosity testing with Cannon-Manning vacuum capillary,
- dynamic viscosity testing with rheometer by cone and plate method,
- dynamic viscosity testing with Brookfield rotary viscometer.

#### Kinematic viscosity testing with BS/IP/RF viscometer

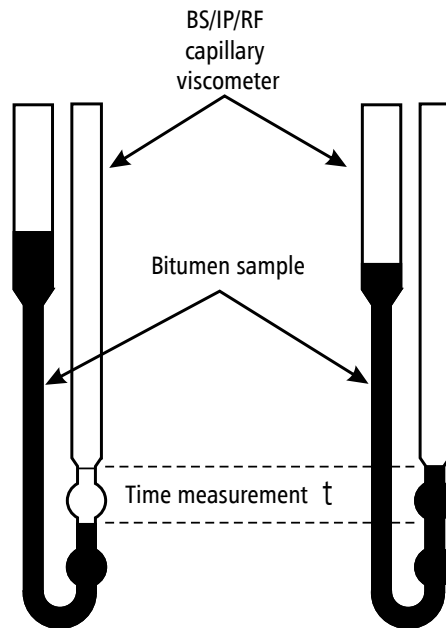
General view of the BS/IP/RF capillary viscometer and apparatus in which the test shown on Figure 2.16 is conducted.



**Figure 2.16.**  
General view of the BS/IP/RF capillary viscometer and apparatus used for kinematic viscosity testing (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)



Kinematic viscosity testing with the BS/IP/RF viscometer is conducted according to PN-EN 12595 *Bitumen and bituminous binders. Determination of kinematic viscosity*. The test involves the determination of the flow time of a specific liquid through a capillary of a calibrated viscometer at a pre-set test temperature (efflux time) [PN-EN 12595]. Figure 2.17 shows the principle of the test duration determination.



**Figure 2.17.** Test duration determination principle in the BS/IP/RF capillary viscometer

Kinematic viscosity is calculated as the product of the efflux time expressed in seconds and the viscometer calibration constant using the following equation [PN-EN 12595]:

$$v = C \cdot t \text{ [mm}^2 / \text{s]}$$

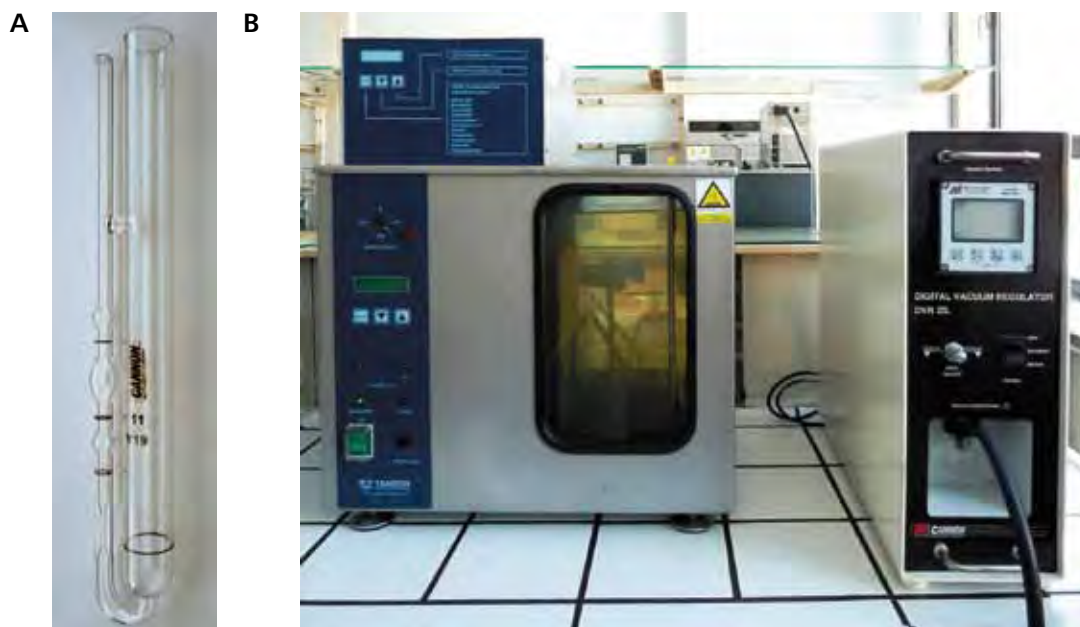
where:

C – viscometer calibration constant expressed in [mm<sup>2</sup>/s<sup>2</sup>]

t – efflux time expressed in [s]

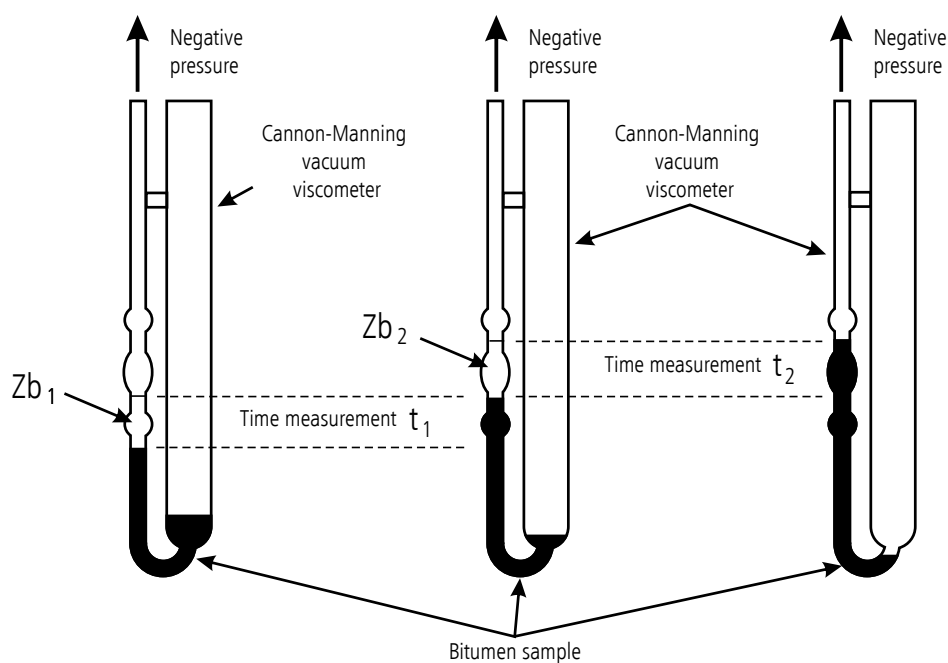
### Dynamic viscosity testing with the Cannon-Manning vacuum capillary

Dynamic viscosity testing is conducted according to PN-EN 12596 *Bitumen and bituminous binders. Determination of dynamic viscosity by vacuum capillary*. Dynamic viscosity testing by vacuum capillary as per PN-EN 12596 involves the application of the difference in flow time of bitumens having different viscosities through a capillary under pre-set negative pressure and temperature. Knowing the flow time of a predetermined bitumen volume and the viscometer calibration factor, we can determine dynamic viscosity using the appropriate equations. General view of the Cannon-Manning vacuum viscometer and apparatus in which the test shown on Figure 2.18 is conducted.



**Figure 2.18.** General view of the Cannon-Manning vacuum viscometer and apparatus used for dynamic viscosity testing (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

The test involves the determination of flow times  $t_1$  and  $t_2$  of a specific volume of liquid by the vacuum method through a capillary under pre-set negative pressure and temperature conditions [PN-EN 12596]. Figure 2.19 shows the principle of the test duration determination.



**Figure 2.19.** Test duration determination principle in the Cannon-Manning vacuum viscometer

Dynamic viscosity is calculated as the arithmetic mean of the products of flow times expressed in seconds ( $t_1$  and  $t_2$ ) and the relevant viscometer calibration factors using the following equations [PN-EN 12596]:

$$\eta_1 = K_1 \cdot t_1 [Pa \cdot s]$$

$$\eta_2 = K_2 \cdot t_2 [Pa \cdot s]$$

$$\eta = \frac{\eta_1 + \eta_2}{2} [Pa \cdot s]$$

where:

$\eta_1, \eta_2$  – namic viscosities calculated on the basis of flow times through tanks  $Zb_1$  and  $Zb_2$

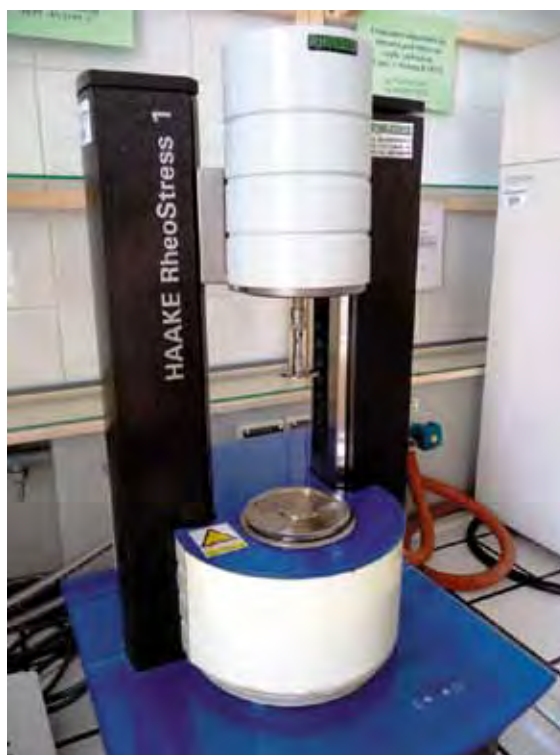
$K_1, K_2$  – selected calibration factors for tanks  $Zb_1$  and  $Zb_2$  expressed in pascals [Pa]

$t_1, t_2$  – flow times through tanks  $Zb_1$  and  $Zb_2$  expressed in [s]

$\eta$  – dynamic viscosity determined in the Cannon-Manning vacuum viscometer

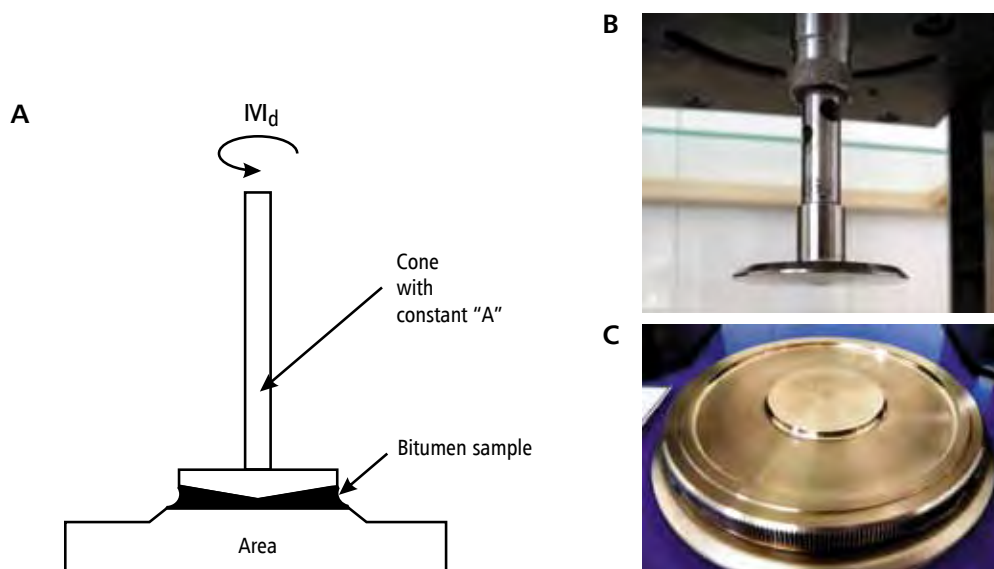
### Dynamic viscosity testing with the rheometer by cone and plate method

Dynamic viscosity testing with the rheometer by cone and plate method is conducted according to PN-EN 13702 *Bitumen and bituminous binders. Determination of dynamic viscosity of modified bitumen. Part 1: Cone and plate method*. General view of the rheometer for the testing of minimum dynamic viscosity by cone and plate method is shown on Figure 2.20.



**Figure 2.20.** General view of the rheometer for the testing of minimum dynamic viscosity by cone and plate method (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

The test involves the determination of torque for a pre-set shear rate [PN-EN 13702-1]. The testing system configuration and the general view of its constituents is shown on Figure 2.21.



**Figure 2.21.** Testing system configuration with a bitumen sample during the test (A) and general view of its components – cone (B) and plate (C)

Using a known torque and cone factor, the testing system calculates viscosity based on the following equation [PN-EN 13702-1]:

$$\eta = \frac{A \cdot M_d}{\gamma} [Pa \cdot s]$$

where:

A – cone factor expressed [m<sup>-3</sup>]

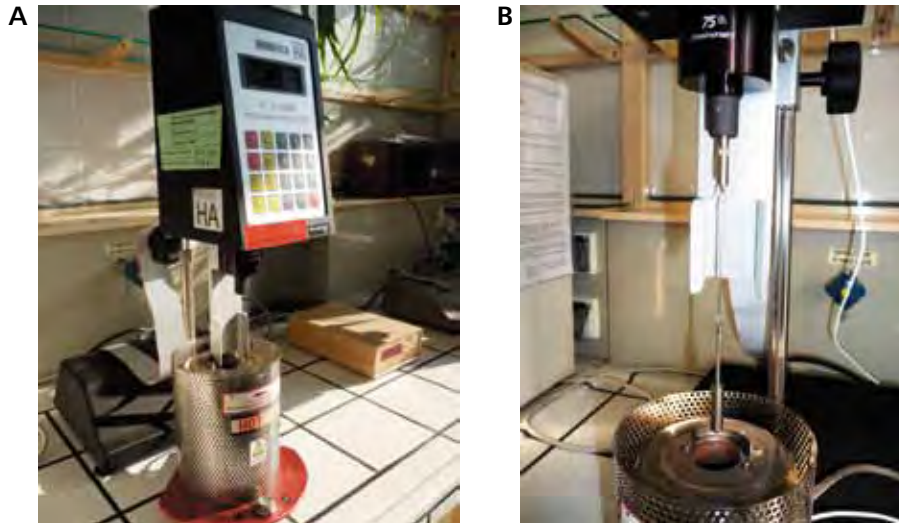
M<sub>d</sub> – torque expressed in [N·m]

γ – shear rate expressed in [s<sup>-1</sup>]

The final result of dynamic viscosity testing, expressed in [Pa·s] or [mPa·s] is calculated as the arithmetic mean of two determinations and is obtained along with the corresponding shear rate and test temperature [PN-EN 13702-1].

### Dynamic viscosity testing with the Brookfield rotary viscometer

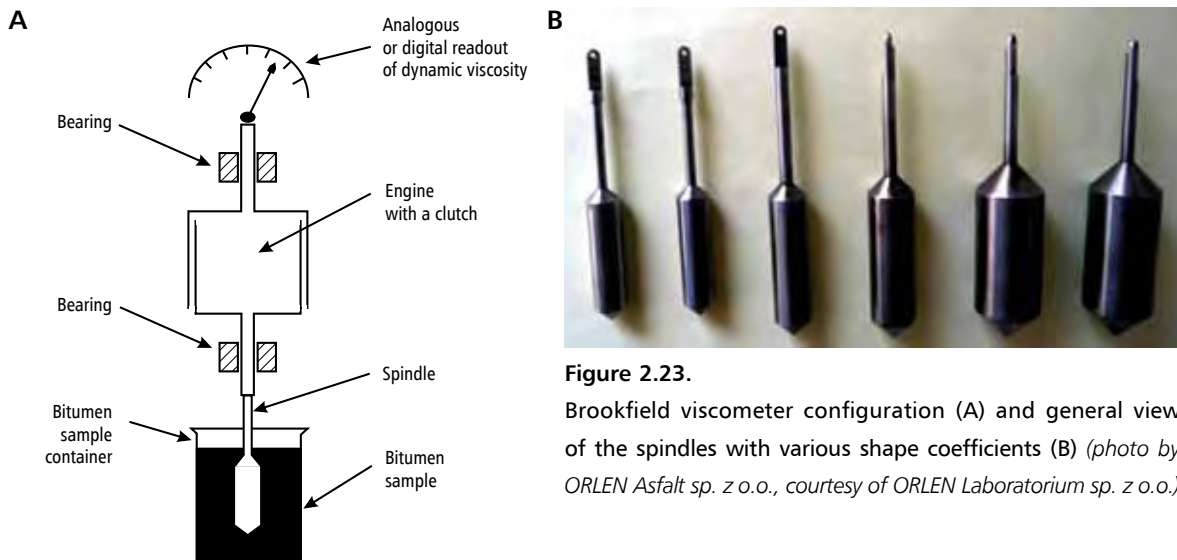
Dynamic viscosity testing is conducted according to PN-EN 13302 *Bitumen and bituminous binders. Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus* or ASTM D 4402 *Standard Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer*. Brookfield dynamic viscosity testing applies the difference in the resistance of a rotating spindle immersed in bitumen with different viscosity. Resistance increases as bitumen viscosity increases, while the spindle's torque remains within the appropriate pre-set range. A view of the Brookfield viscometer for dynamic viscosity testing and the related testing system is shown on Figure 2.22.



**Figure 2.22.** General view of the Brookfield viscometer (A) and close-up on the spindle and a heated container for the bitumen sample (B) (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

Dynamic viscosity testing involves the determination of the relationship between relative resistance to the rotation of the spindle in a special container holding the tested sample at the pre-set spindle rotational speed. Dynamic viscosity of the tested liquid is read out directly on the viscometer, whose spindle torque must be within a specific range. If the condition is not met, the spindle is replaced with another one, having a different shape coefficient [PN-EN 13302].

The Brookfield viscometer configuration and the general view of the spindles with various shape coefficients is shown on Figure 2.23. The shape of the spindle (spindle numbers are typically used) should be stated with the Brookfield apparatus viscosity test result.



**Figure 2.23.** Brookfield viscometer configuration (A) and general view of the spindles with various shape coefficients (B) (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

The final result of dynamic viscosity testing, expressed in [Pa·s], [mPa·s] [PN-EN 13302] or [cPa] [ASTM D 4402] is calculated as the arithmetic mean of three determinations.

The results of paving-grade and modified bitumen viscosity tests are provided in Chapters 4 and 5.

### 2.2.9. Density

Bitumen density testing is conducted according to PN-EN 15326 *Bitumen and bituminous binders. Measurement of density and specific gravity. Capillary-stoppered pycnometer method*. The test involves the determination of the ratio of density of the tested bituminous binder and the test fluid density under the same temperature conditions.

A sample of bitumen with precisely determined volume is placed in a pycnometer at 25°C. After temperature balance is achieved, the sample is weighed with the appropriate precision. Then, the process is repeated with the test fluid, i.e. exactly the same volume of the test fluid (with known density) is weighed. Density and specific gravity are calculated from the appropriate equation on the basis of the known difference in the masses. The unit of density is [kg/m<sup>3</sup>].

In former bitumen standards, the standard for density testing was PN-EN ISO 3838 *Crude petroleum and liquid or solid petroleum products – Determination of density or relative density – Capillary-stoppered pycnometer and graduated bicapillary pycnometer methods*.

Bitumen density is required, among others, to calculate volumetric parameters of bituminous mixtures as per PN-EN 12697-8 *Bituminous mixtures. Test methods for hot mix asphalt. Part 8: Determination of void characteristics of bituminous specimens*.

### 2.2.10. Short-term ageing (RTFOT)

The most intense bitumen ageing processes take place when bitumen is mixed with hot aggregate in the batching plant's mixer. Temperature at that time is the highest and the layer of bitumen on the aggregate is very thin. Under those circumstances the evaporation of light fractions and bitumen oxidation is the fastest and most intense, and bitumen rapidly hardens (ages). The process is referred to as **short-term ageing**. The next step is long-term ageing, taking place in the pavement over many years of road service life (see PAV test described in Section 2.4.4).

Bitumen hardens (its stiffness increases) as a result of ageing, which means that, among other things:

- its penetration drops,
- its softening point increases,
- its break point increases (deteriorates),
- its viscosity increases.

Bitumen ageing should not be passed over in this discussion on bitumen as a construction material. Importantly, bitumen incorporated in the pavement has already undergone short-term ageing. Tests on bitumen sensitivity to ageing are therefore very relevant. Short-term ageing (at a high temperature) is a vital consideration.

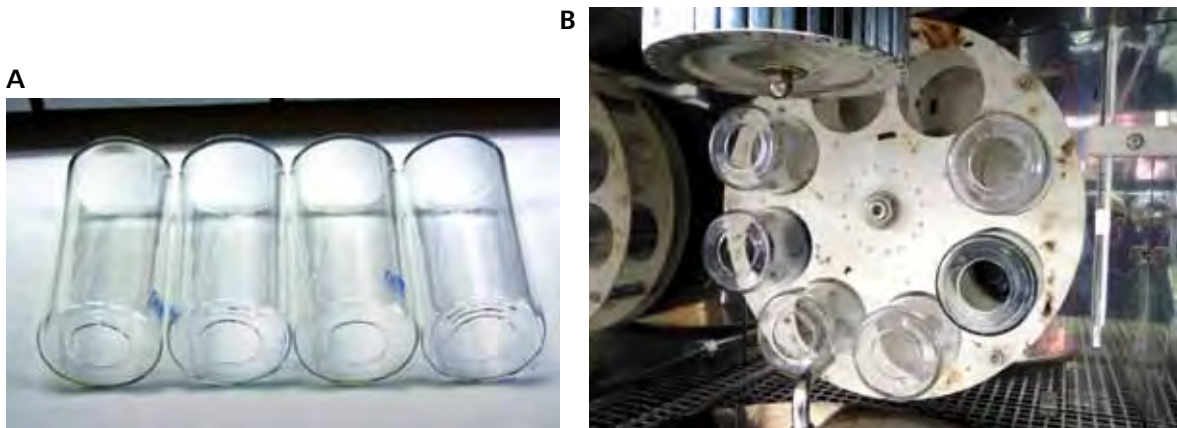
From the perspective of pavement quality, it is required to check the effects of short-term ageing on primary bitumen properties, such as penetration, softening point and elastic recovery (for modified bitumen). For this reason, a number of test methods have been developed to replicate the ageing process and produce bituminous binder samples "after ageing" for further testing.

One of the most commonly used methods for short-term ageing is RTFOT (*Rolling Thin Film Oven Test*) conducted in accordance with PN-EN 12607-1 *Bitumen and bituminous binders. Determination of the resistance to hardening under influence of heat and air. Part 1: RTFOT method*. It involves subjecting a thin layer of bituminous binder to hot air flowing around it for a specified period of time.

Bitumen samples placed in pre-weighed glass containers are installed in a special rotating disc inside a drier for the RTFOT test at the temperature of 163°C with air blowing. After a specific time the samples in the glass containers are taken out of the drier and cooled down to ambient temperature. The binder in the glass containers has thus undergone the short-term ageing simulation test and can be used for subsequent tests.



**Figure 2.24.** General view of the RTFOT short-term ageing test set (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)



**Figure 2.25.** View of glass containers for bitumen samples (A) and containers installed in the RTFOT drier (B) (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

## 2.2.11. Properties after RTFOT

A certain quantity of aged bitumen is obtained from the RTFOT test. Subsequently, tests are carried out to verify whether the binder's properties have changed as a result of short-term ageing.

### 2.2.11.1. Retained penetration after RTFOT

Bitumen penetration drops as a result of short-term ageing. The bitumen sample obtained from the RTFOT is tested for penetration at 25°C according to PN-EN 1426. Then, the value of penetration after ageing is calculated [%] as a percentage share of the initial bitumen penetration before the RTFOT ageing (assuming that non-aged bitumen penetration is 100%).

### 2.2.11.2. Increase and decrease in softening point after RTFOT

Bitumen softening point after short-term ageing usually increases. The bitumen sample obtained from the RTFOT is tested for softening point according to PN-EN 1427. Then, the increase in softening point after ageing is calculated [°C] as the difference between the softening point of the sample after the RTFOT ageing and non-aged bitumen's softening point. The requirement limiting the increase in the softening point after ageing applies to each type of bitumen used for hot mixes: paving-grade, modified and multigrade.

It may happen that the softening point after RTFOT is lower for some modified bitumens, which depends on the actual modifier and modification technology. Therefore, the specification for modified bitumens as per PN-EN 14023 provides an optional requirement concerning determination of this property. For instance, the requirement is included in the Polish national annex to PN-EN 14023:2011.

### 2.2.11.3. Mass change after RTFOT (absolute)

Bitumen mass may change (increase or decrease) as a result of short-term ageing. The change of mass after ageing is determined according to PN-EN 12607-1 (RTFOT). It is calculated as the mass difference between fresh bitumen and the sample after the RTFOT. The final result is the absolute percentage mass difference between the sample before and after the ageing test.

## 2.3. Additional properties of polymer-modified bitumen

The following sections discuss tests developed specifically for polymer-modified bitumen.

### 2.3.1. Elastic recovery

No conventional polymer-modified bitumen testing is able to determine elastic properties of that material. This has given rise to the partially adapted ductility test. It is called the elastic recovery test.

Elastic recovery testing is conducted according to PN-EN 13398 *Bitumen and bituminous binders. Determination of the elastic recovery of modified bitumen*. The test involves the determination of a conventional expression of bitumen elasticity being the distance between ends of a stretched and cut sample under pre-set conditions.



A bitumen sample is stretched at a specified temperature (typically at 25°C or 10°C) at a constant speed of 50 mm/min., until its elongation reaches 200 mm. A bitumen thread thus formed is cut in half. After 30 minutes, the distance between both ends of the cut sample is measured. Then, elastic recovery is calculated as a percentage share of elongation [%].

The following equation is used for the calculation of elastic recovery (RE):

$$R_E = \frac{d}{L} 100 \quad [\%]$$

where:

**d** - distance between ends of the cut sample [mm],

**L** - sample elongation, typically 200 mm (it can be less if the sample breaks too early) [mm].

Elastic recovery is expressed as a percentage, where 0% stands for no elasticity and 100% for full recovery to the original shape. Elastomer-modified bitumen (depending on the quantity of elastomer) demonstrates elastic recovery in excess of 50%. The test is the primary test for the operation (and presence) of elastomer. The test result is provided with the accuracy of up to 1%.

If the samples cannot be extended to 200 mm (thread breaking too early), the result is specified for that shorter elongation, and the relevant comments are added to the test result. These results can also be considered correct based on the standard.

**Note:** the mould for elastic recovery testing samples is different from the mould for the force ductility test (see 2.3.3).

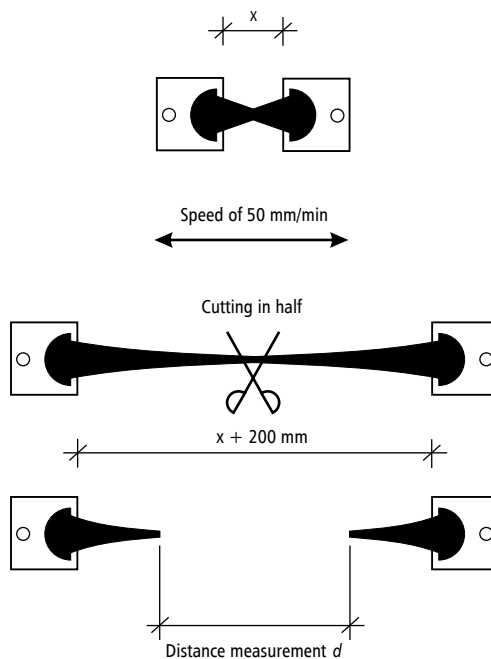
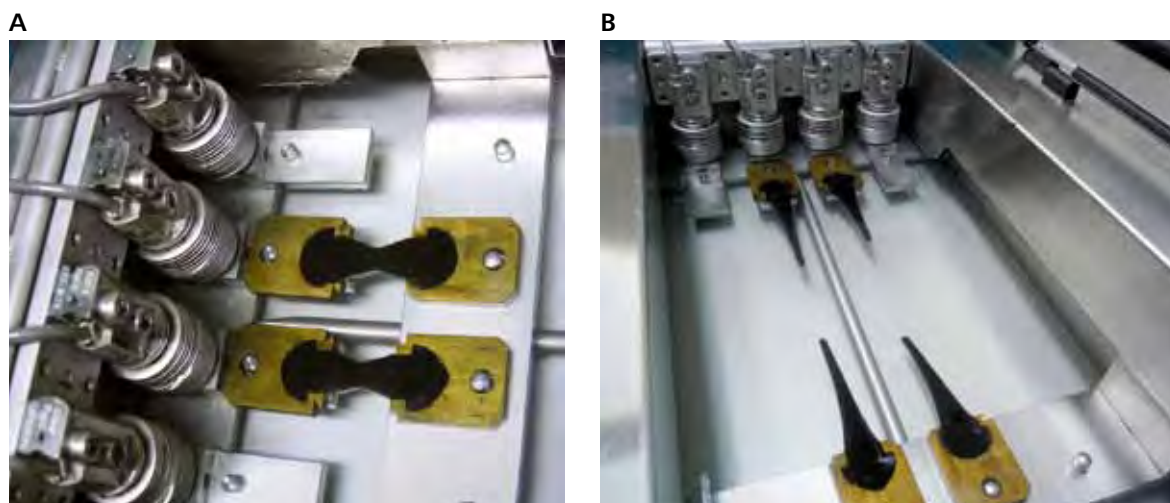


Figure 2.26. Elastic recovery test principle



**Figure 2.27.** General view of the elastic recovery test set  
(photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)



**Figure 2.28.** View of a bitumen sample before (A) and in the course of sample recovery after cutting (B)  
(photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

### 2.3.2. Elastic recovery at 25°C after RTFOT ageing

Testing for bitumen elastic recovery after the RTFOT ageing acc. to PN-EN 12607-1 is conducted in accordance with PN-EN 13398, and the testing method does not differ from non-aged bitumen testing as discussed in Section 2.3.1. The result of the test answers the question to what extent the polymer (elastomer) remains effective after ageing, and consequently to what extent it will be effective in reality.

### 2.3.3. Force ductility (cohesion)

If the level of bitumen cohesion is sufficiently high, it can transmit ductility forces in the pavement. It is assumed that this makes the pavement more resistant to cracks.

Force ductility testing (at a small tension rate) is conducted according to PN-EN 13589 *Bitumen and bituminous binders. Determination of the tensile properties of modified bitumen by the force ductility method*. The test involves the determination of the force required for sample tension (up to the appropriate elongation) at a specified temperature.

A properly shaped sample is placed in the ductilometer in water bath having the appropriate temperature (specified for each type of modified bitumen). The sample is then continuously stretched at a rate of 50 mm/min., until it reaches elongation of at least 1333% (400 mm). Sensors record the applied force throughout the tensile process. The penetration test is conducted according to PN-EN 13703 *Bitumen and bituminous binders. Determination of deformation energy on the basis of data readout from the apparatus*. The unit for cohesion is [J/cm<sup>2</sup>].

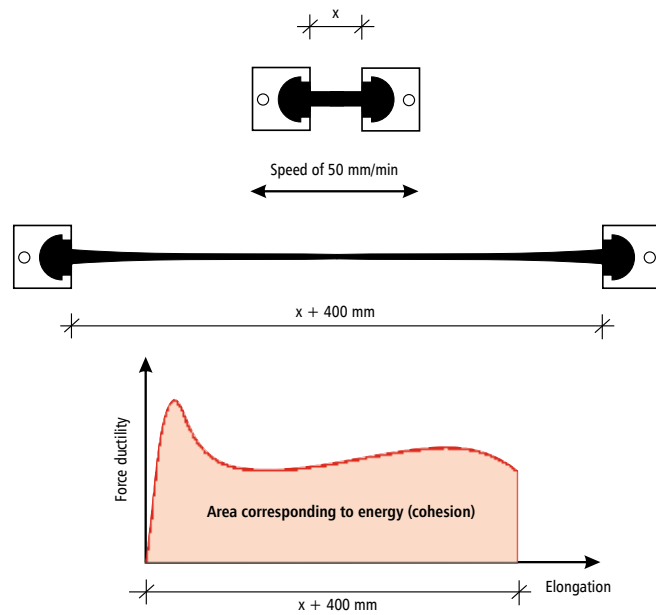


Figure 2.29. Force ductility test principles – energy-to-elongation chart

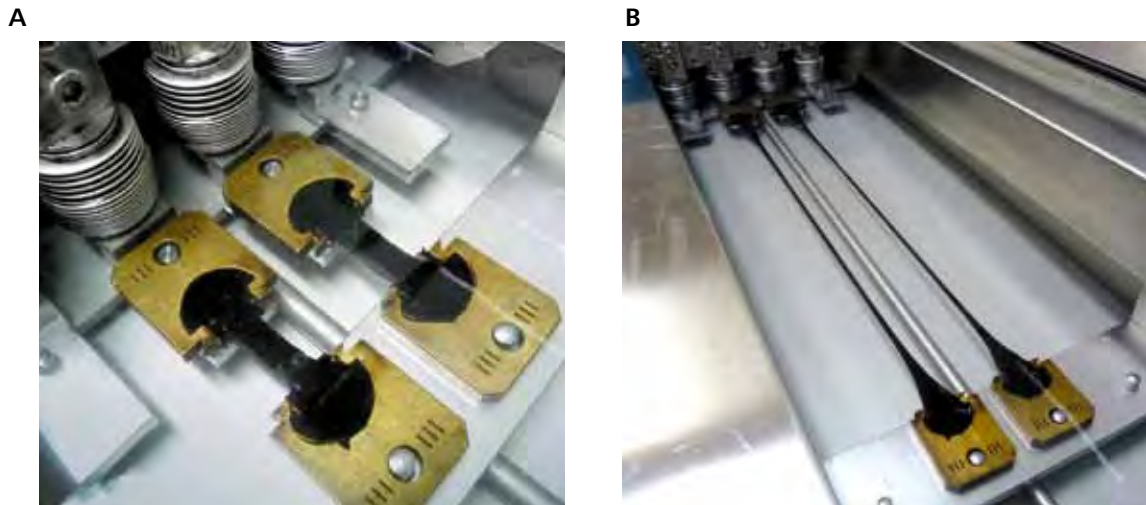


Figure 2.30. View of a bitumen sample before (A) and after the test (elongated to 400 mm) in the force ductility testing apparatus (B) (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

### 2.3.4. Storage stability of modified bitumens

This test is a production test and is of significance for the determination of modified bitumen usability period. As already stated, the production of polymer-modified bitumen involves the batching of the appropriate polymer and achievement of the required product parameters by employing the appropriate technology. Although the description of the process is very simplified, manufacturers of modified bitumen have to face the related problems, such as bitumen and polymer incompatibility, which may separate polymer from bitumen some time after production.

The purpose of the stability test is to check whether modified bitumen is exposed to the risk of polymer separation from bitumen. Stability testing in the course of modified bitumen storage is conducted according to PN-EN 13399 *Bitumen and bituminous binders. Determination of storage stability of modified bitumen*. This popular test is referred to as the “tube test”, because binder samples are poured into metal tubes.

A modified bitumen sample placed in an aluminium tube is heated vertically to and held at 180°C for 72 hours. The tube is then left to cool. After cooling, the aluminium coating of the tube is removed and the sample is cut into three roughly equal parts. The central sample part is thrown out. The softening point test is conducted for the upper and lower section of the sample (PN-EN 1427), and, potentially, for penetration at 25°C according to PN-EN 1426. The result of the stability test is assumed to be the difference between softening point values (or penetration at 25°C) obtained for the upper and lower modified bitumen samples.



**Figure 2.31.** General view of the filled and sealed tube for the “tube test” and of the modified bitumen sample cut into three sections (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

### 2.3.5. Polymer modified bitumens microstructure

The test is conducted according to PN-EN 13632 *Bitumen and bituminous binders. Visualisation of polymer dispersion in polymer-modified bitumen*. The purpose of the test is to obtain information about how the polymer is dispersed in the bitumen. The test is conducted on a fresh fracture of sampled modified bitumen under the fluorescent microscope with a UV lamp, through image analysis in the reflected light.

The standardised result is provided as a collection of letter codes:

- Phase continuity: **P**: Continuous polymer phase  
**B**: Continuous bitumen phase  
**X**: Continuity of both (crosslinking)
- Phase description: **H**: Homogeneous  
**I**: Heterogeneous
- Size description: **S**: Small (< 10  $\mu\text{m}$ )  
**M**: Medium (from 10  $\mu\text{m}$  to 100  $\mu\text{m}$ )  
**L**: Large (> 100  $\mu\text{m}$ )
- Shape description: **r**: round, cylindrical  
**s**: Elongated  
**o**: Other

ORBITON modified bitumen characteristics in terms of polymer dispersion are provided in Chapter 5, Table 5.14.

## 2.4. Other bitumen properties

### 2.4.1. N-heptane insoluble content

N-heptane insoluble content testing is conducted according to ASTM D 4124 *Standard Test Method for Separation of Asphalt into Four Fractions*. The test involves the determination of the percentage share of insolubles in bitumen subjected to a solvent (n-heptane in this case). It is assumed that the result specifies the content of asphaltenes in the binder.

A bituminous binder sample is dissolved in n-heptane (solvent). The solution thus formed is filtered through a layer of glass powder (from sintered glass) in a crucible. Undissolved material from bitumen is then washed, dried and weighed. N-heptane insoluble content is calculated as a percentage share [% m/m] relative to the sample prior to dissolving.

Currently, this requirement is rarely applied.

### 2.4.2. Paraffin content

The testing for paraffin content in bitumen is conducted according to PN-EN 12606 *Bitumen and bituminous binders. Determination of the paraffin wax content. Part 1: Method by distillation*. The test involves the determination of the percentage share of paraffins in the bitumen distillate obtained in a strictly specified distillation process.

A bitumen sample is preheated in a porcelain vessel and placed in a distillation flask. Then, bitumen in the flask is subjected to specified distillation conditions in the heating process. The distillate thus obtained is cooled down to room temperature and weighed. Then, the distillate is dissolved in the mixture of ether and ethanol in the appropriate proportions. The resultant mixture is cooled to -20°C. Paraffin is precipitated in the filtering process. Appropriately elutriated and extracted paraffin is weighed and, on the basis of the information thus obtained, paraffin content in bitumen is calculated as the percentage share relative to the bitumen sample mass [% m/m].

There is also another method for the determination of paraffin in bitumen which is, however, used more rarely: PN-EN 12606-2 *Bitumen and bituminous binders. Determination of the paraffin wax content. Part 2: Method by extraction*. The values obtained using this method are typically greater than for the distillation method.

To note, the European standard PN-EN 12591 and the remaining standards, namely PN-EN 14023 and PN-EN 13924, no longer contain the requirements for the paraffin wax content.

### 2.4.3. Bitumen adhesion to mineral aggregates

Adhesion “is the formation of the bond between surface layers of two bodies (solid or liquid) which are in contact [4]”.

Good bitumen adhesion to the surface of aggregate is one of the factors in the durability of pavement courses.

Factors affecting adhesion include:

- Aggregate moisture rate.
- Aggregate dusting.
- Aggregate grain microtexture.
- Aggregate pH (silica content).
- Bitumen physical and chemical properties.

Next to the factors referred to above, a precondition for good adhesion is the appropriate viscosity of bitumen – it must be sufficiently liquid to coat the aggregate. Tests conducted by ORLEN Asphalt have also demonstrated that adhesion improves the longer the asphalt mix is kept in the silo. In other words, bitumen adheres better to aggregate grain surfaces after ageing.

Bitumen adhesion to aggregate can be tested using PN-EN 12697-11 *Bituminous mixtures. Test methods for hot mix asphalt. Part 11: Determination of the affinity between aggregate and bitumen*.

According to the standard, the affinity between aggregate and bitumen is specified as the visually determined rate of bitumen coating of the grains subjected to the following procedure:

- mixing in water in rolled bottles over a specific time (method A),
- immersion in water for 48 hours (method B),
- using acids (HCl or HG) and phenolphthalein solution as the marker, and boiling in water for 10 minutes (method C).

Appropriate-fraction aggregate is used for the test. The aggregate is washed, dried and mixed with the binder until uniform coating is obtained. The test procedures, depending on the method, are specified in the standard.



**Figure 2.32.** Bottles for testing binder elutriation from aggregate (method A) on the rolling machine acc. to PN-EN 12697-11 (photo by ORLEN Asphalt sp. z o.o.)



**Figure 2.33.** Aggregate samples after the test (method A) as per PN-EN 12697-11 (photo by ORLEN Asphalt sp. z o.o.)

### Tests to verify binder adhesion to aggregate as part of bituminous mixture tests

Next to direct testing of bitumen adhesion to aggregate as described above, various testing methods are used for the determination of bituminous mixture resistance to water and frost, which indirectly addresses the question of the bitumen-aggregate bond stability. The test used in Poland is based on PN-EN 12697-12 *Bituminous mixtures. Test methods for hot mix asphalt. Part 12: Determination of the water sensitivity of bituminous specimens*, or the curing of bituminous mixtures using the impact method (so-called Marshall samples) and then their splitting. The ITSr index thus obtained is a measure of the bituminous mixture's resistance to water and frost (if the freezing cycle is used). A similar, although not identical method comes from the American AASHTO T 283 – the so-called *modified Lottman test*.

#### 2.4.4. PAV ageing

Section 2.2.10 on short-term ageing discusses the RTFOT method. The section also mentions another type of ageing, namely **long-term ageing**. It occurs in the course of service life of bituminous pavements and involves the effect on the binder of oxygen, UV radiation, substances present in precipitation water, chemicals for de-icing, etc. Long-term ageing causes bitumen properties to gradually change over the pavement's service life. This type of ageing leads to a gradual, slow bitumen hardening.

To test binder sensitivity to long-term ageing, the PAV (Pressure Ageing Vessel unit has been designed. PAV testing can be conducted according to ASTM D6521/AASHTO R28 *Standard Practice for Accelerated Ageing of Asphalt Binder Using a Pressure Ageing Vessel (PAV)* or acc. to PN-EN 14769 *Bitumen and bituminous binders. Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV)*.

Bitumen subjected to PAV testing is pre-tested using the RTFOT, which means that long-term ageing (on the road) is preceded by short-term ageing (in the mixing plant). The vessel with bitumen is pressurised to 2.1 MPa over 20 hours at the temperature depending on the type of PG<sup>1</sup> (90°C, 100°C or 110°C). The entire process is designed to simulate 7-10 years of bitumen ageing in the pavement.

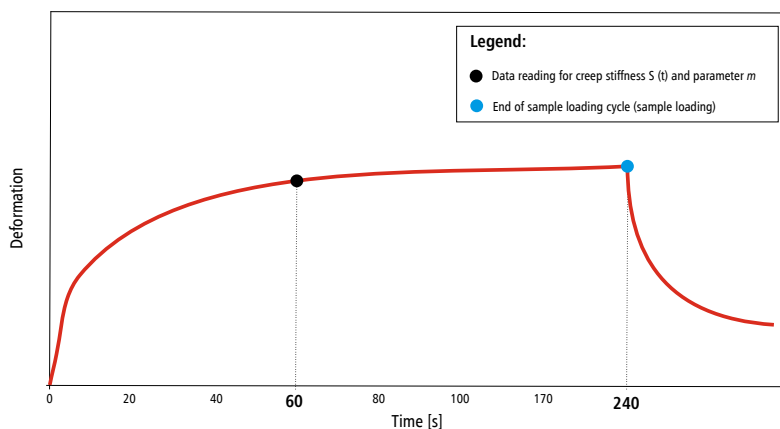
Bitumen samples after PAV ageing are used to measure bitumen properties at low temperatures (cracks) and intermediate temperatures (fatigue) as per the *Superpave* methodology. European standards containing requirements for paving-grade binders do not yet contain PAV testing requirements.

### 2.4.5. BBR method

The BBR method (*Bending Beam Rheometer*) is used in the US (as part of the Performance Grade system) as well as in Europe (as an additional test) to determine low-temperature bitumen properties.

The method is standardised in the US as ASTM D6648 *Standard Test Method for Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)* and in Europe under PN-EN 14771 *Bitumen and bituminous binders. Determination of the flexural creep stiffness. Bending Beam Rheometer (BBR)*.

The BBR test is fully automatic and takes place in pre-set temperature cycles, e.g. -10, -16, -22, -28, -34°C. A properly formed bitumen beam (dimensions in cross-section: 12.70±0.05 mm in height, 6.35±0.5 mm in width), freely supported (the distance between the supports is 127±0.02 mm) is subjected to a constant, vertical load at the central point between the supports. The operation of constant force causes the beam to deform (bend). Using strength relationships, the test apparatus calculates the so-called “creep stiffness”  $S(t)$  as a function of time, as well as the so-called parameter “ $m$ ”, showing how stiffness changes over the loading time. Data for the calculations are taken at the 60th second of the loading process in each temperature cycle. The nature of the sample deformation curve and the calculation data readout moment is shown in Figure 2.34.

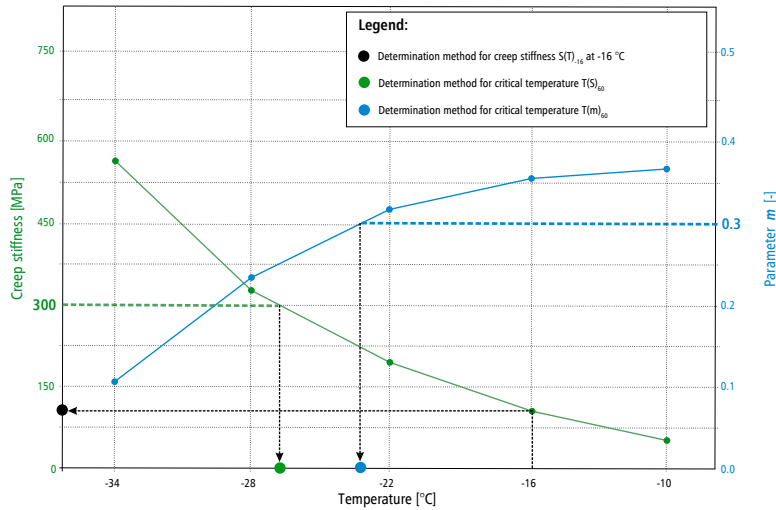


**Figure 2.34.** Nature of the sample deformation curve and the creep stiffness  $S(t)$  and parameter “ $m$ ” calculation data readout moment in a single temperature cycle of the loading process

On obtaining full calculation data (creep stiffness and parameter  $m$ ) from the analysed temperature cycles, e.g. -10, -16, -22, -28, -34°C, the so-called “lower critical temperatures”  $T(S)_{60}$ <sup>2</sup> and  $T(m)_{60}$ <sup>3</sup> are determined, as well as creep stiffness at -16°C,  $S(T)_{-16}$ . The determination method for “critical temperatures”  $T(S)_{60}$ ,  $T(m)_{60}$  and creep stiffness  $S(T)_{-16}$  are shown in Figure 2.35.

- 1) The American Performance Grade (PG) system is described in Chapter 7.
- 2) Critical temperature  $T(S)_{60}$  is determined on the assumption that creep stiffness  $S(60)$  should be  $\leq 300$  MPa, which should ensure resistance to cracks of the bituminous mixture using the tested bitumen, and therefore  $T(S)_{60}$  is the temperature at which binder stiffness is exactly 300 MPa [4].
- 3) Critical temperature  $T(m)_{60}$  is determined on the assumption that the value of parameter  $m(60)$  should be  $\geq 0.3$ , which is related to the fact that bitumens with high parameter  $m$  ( $\geq 0.3$ ) demonstrate a more effective relaxation of stresses present in the binder when temperatures drop [4]. Therefore,  $T(m)_{60}$  is the temperature at which parameter  $m$  equals 0.3.





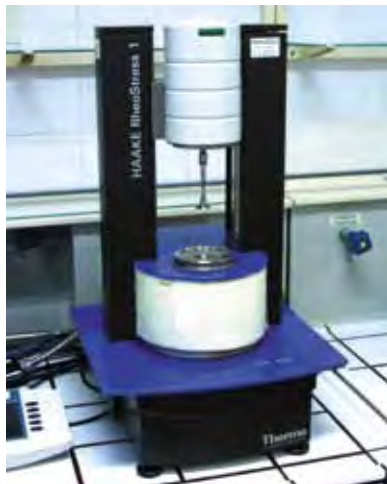
**Figure 2.35.** Determination method for critical temperatures  $T(S)_{60}$ ,  $T(m)_{60}$ , and creep stiffness  $S(T)_{-16}$  on the basis of data obtained in temperature cycles at -10, -16, -22, -28 and -34°C.

Results of BBR tests –  $(T(S)_{60}, T(m)_{60}$  and creep stiffness  $S(T)_{-16}$ ) for binders manufactured by ORLEN Asphalt are provided in Chapters 4 and 5.

### 2.4.6. DSR method

The determination of complex rheological properties of bitumen is currently possible with the use of the Dynamic Shear Rheometer (DSR), Figure 2.36. Parameters most commonly tested in that rheometer include the bitumen stiffness module and phase angle, tested in various temperature and frequency ranges.

The method is standardised as AASHTO T 315 *Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)* and as PN-EN 14770 *Bitumen and bituminous binders. Determination of complex shear modulus and phase angle. Dynamic Shear Rheometer (DSR)*.



**Figure 2.36.** General view of the DSR (photo by ORLEN Asphalt sp. z o.o., courtesy of ORLEN Laboratorium sp. z o.o.)

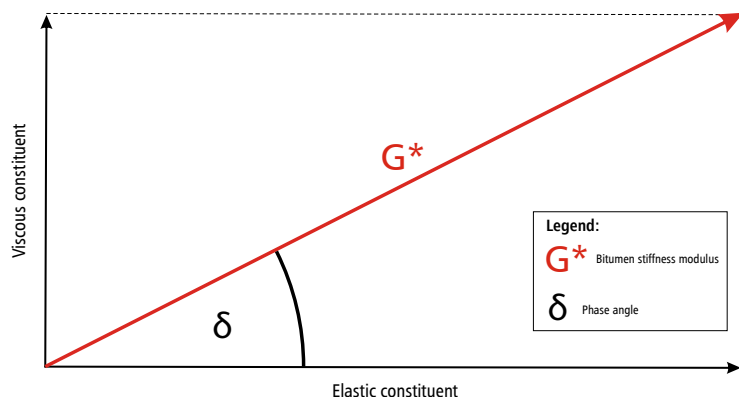
Stiffness modulus<sup>4</sup> ( $G^*$ ) and phase angle ( $\delta$ ) testing in the DSR is fully automatic (similarly to the BBR test) and occurs in pre-set temperature cycles, e.g. from 10 to 82°C, in steps of e.g. 6°C. An appropriately formed bitumen “cylinder” with the following dimensions:

- 8 mm in diameter and 2 mm in height for tests at 10÷46°C,
- 25 mm in diameter and 1 mm in height for tests at 46÷82°C,

4) Complex shear modulus  $G^*$

is subjected to oscillating shear in the DSR at the frequency of 10 rad/s (1.59Hz). Using the theory on viscoelastic bodies, which assumes that the stiffness modulus comprises “viscous” and “elastic” constituents, with their interdependencies shown on Figure 2.36, the testing equipment calculates the following relationships:

- $G^*/\sin\delta$  for the tested non-aged bitumen sample,
- $G^*/\sin\delta$  for the tested RTFOT-aged bitumen sample,
- $G^* \cdot \sin\delta$  for the tested RTFOT+PAV-aged bitumen sample.

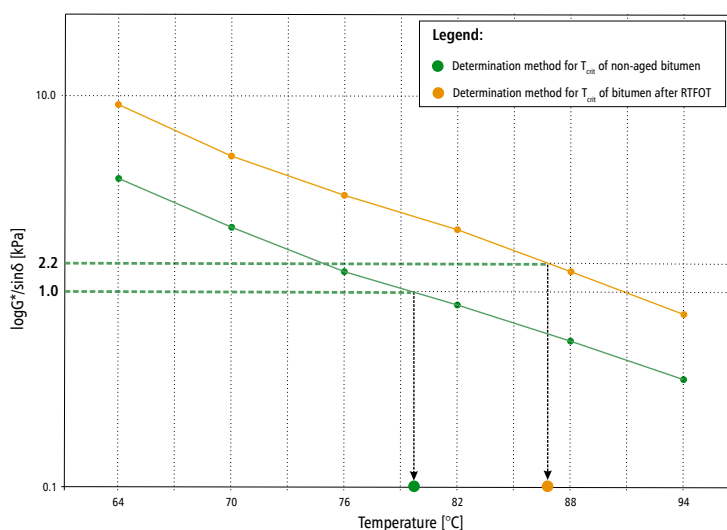


**Figure 2.37.**  
The “viscous” and “elastic” constituents of bitumen and their interdependencies

On obtaining full results of the calculations ( $G^*/\sin\delta$  and  $G^*\sin\delta$  for non-aged bitumen, bitumen after RTFOT and after RTFOT+PAV) from the analysed temperature cycles, e.g. from 10 to 82°C in steps of e.g. 6°C, the so-called “upper critical temperatures” are determined, i.e.:

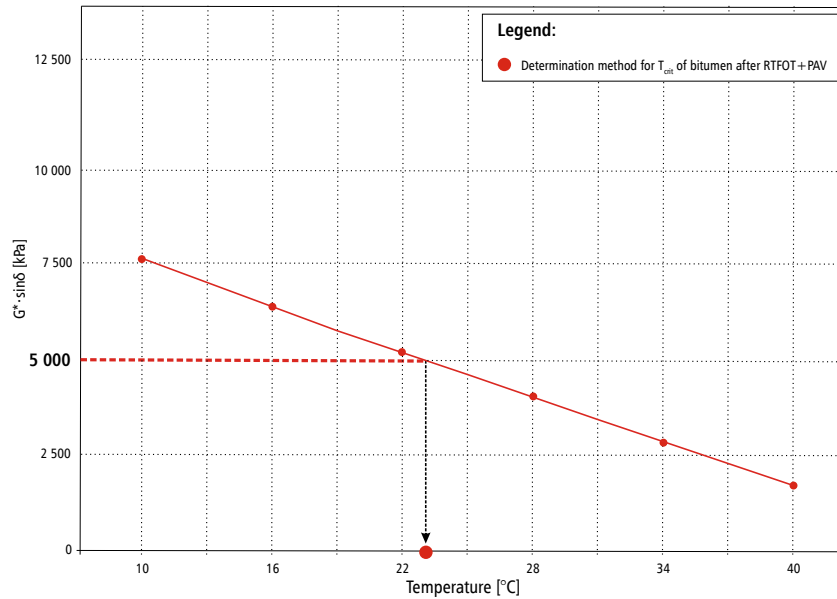
- $T_{crit}$  (for non-aged bitumen, the temperature at which  $G^*/\sin\delta = 1.0$  kPa)<sup>5</sup>
- $T_{crit}$  (for RTFOT-aged bitumen, the temperature at which  $G^*/\sin\delta = 2.2$  kPa)<sup>6</sup>
- $T_{crit}$  (for RTFOT+PAV-aged bitumen, the temperature at which  $G^* \cdot \sin\delta = 5\,000$  kPa)<sup>7</sup>

Determination method for critical temperature  $T_{crit}$  (for non-aged bitumen, bitumen after RTFOT and RTFOT+PAV) is shown on Figure 2.38.



**Figure 2.38.**  
Determination method for critical temperature  $T_{crit}$  for non-aged bitumen and bitumen after RTFOT on the basis of data from temperature cycles, e.g. from 10 to 82°C in steps of 6°C

5) In order for the bituminous mixture to resist rutting, it is assumed that  $G^* \cdot \sin\delta$  for fresh bitumen should not be smaller than 1.0 kPa [www.pavementinteractive.org]  
 6) In order for the bituminous mixture to resist rutting, it is assumed that  $G^*/\sin\delta$  for RTFOT-aged bitumen should not be smaller than 2.2 kPa [www.pavementinteractive.org]  
 7) In order for the bituminous mixture to resist fatigue cracks, it is assumed that  $G^* \cdot \sin\delta$  for RTFOT+PAV-aged bitumen should not be greater than 5 000 kPa [www.pavementinteractive.org], whereas the most recent US research for roads loaded with the heaviest traffic requires 6 000 kPa



**Figure 2.39.** Determination method for critical temperature  $T_{crit}$  for bitumen after RTFOT+PAV on the basis of data from temperature cycles, e.g. from 10 to 82°C in steps of 6°C

Results of bitumen tests in the DSR ( $T_{crit}$  for non-aged bitumen, bitumen after RTFOT and RTFOT+PAV) are provided in Chapters 4 and 5.

### 2.4.7. MSCR method

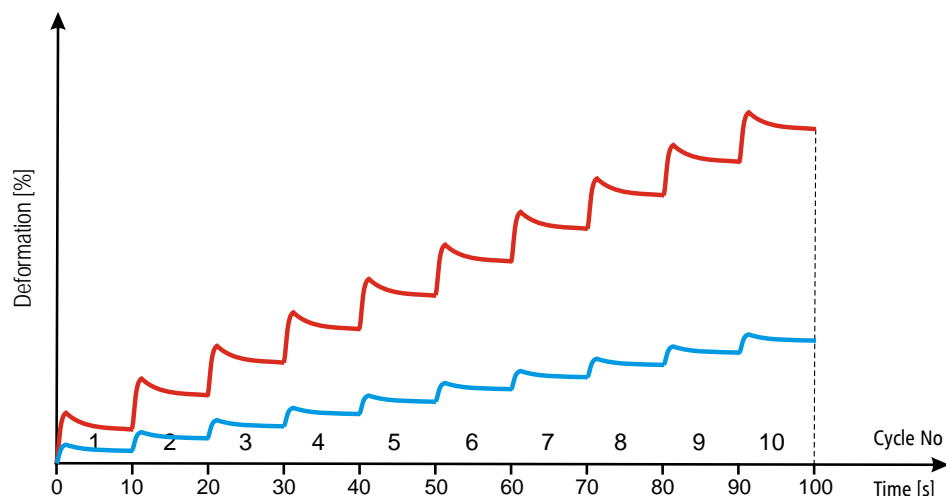
The MSCR (Multiple Stress Creep Recovery test) method involves the measurement of certain binder properties in order to determine the impact of the binder on resistance to rutting and evaluate the rate and efficiency of polymer modification. The test is conducted on an appropriately configured DSR shown in Figure 2.34.

The MSCR test is conducted according to the following standards: AASHTO TP 70: *Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)* and ASTM D7405: *Standard Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer*.

A cylindrical bitumen sample, 1 mm in height and 25 mm in diameter, is placed between a rotor and a heated base in the DSR, and subjected to the stress application cycle over 1 second and rest period over 9 seconds. There are 10 stress and relief cycles in total. It can be argued that two mechanisms are analysed in the entire testing process:

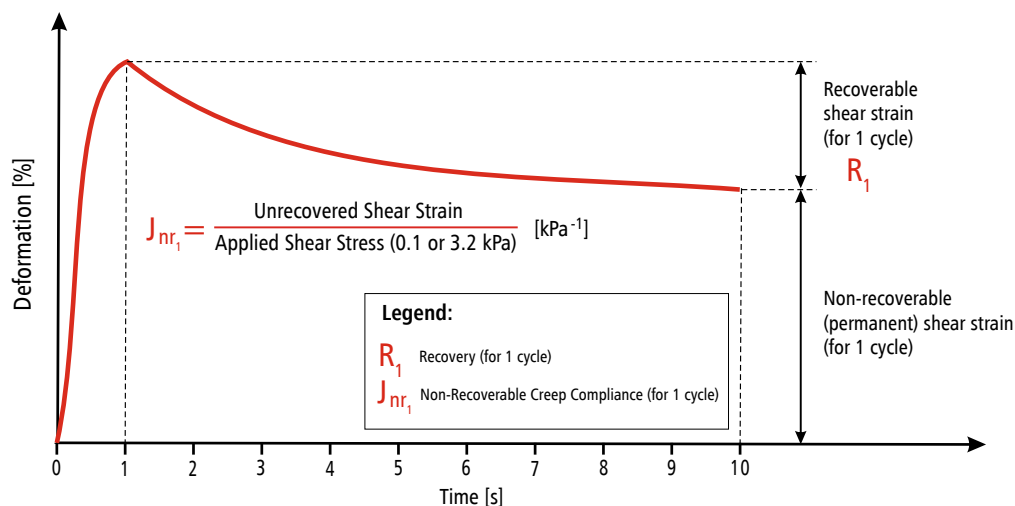
- binder sample creep mechanism – during the 1-second stress application,
- binder sample recovery mechanism – during the 9-second rest period (after the stress is removed).

The nature of the sample deformation curve in the MSCR test (10 stress and relief cycles) is shown in Figure 2.40.



**Figure 2.40.** Nature of the sample deformation curve in the MSCR test (stress of 0.1 or 3.2 kPa) for two different bitumens. The nature of the deformation allows for the comparison of bitumen properties. Bitumen highlighted in blue on the figure is marked by better properties than bitumen highlighted in red (sensitivity to deformation is different).

The test is conducted with a binder after the RTFOT ageing, i.e. with two stress values, e.g. 0.1 kPa and 3.2 kPa (values indicated in ASTM D 7405) at the highest temperature at which the pavement with the binder should work. In effect, two pairs of results are obtained: “non-recoverable creep compliance”, that is parameter  $J_{nr}$  in  $[kPa^{-1}]$  and “recovery”  $R$  [%] for two stress values (0.1 kPa and 3.2 kPa). Both properties are determined after each of the 10 cycles. The final result is a pair of parameters:  $J_{nr}$  and  $R$ , which are calculated as arithmetic means from the results obtained in 10 cycles. The nature of  $J_{nr}$  and  $R$  properties over one cycle is shown on Figure 2.41.



**Figure 2.41.** Nature of  $J_{nr}$  and  $R$  properties over one stress and rest period

In addition, on the basis of  $J_{nr}$  and  $R$  determined for two stress values – 0.1 kPa and 3.2 kPa, additional parameters are determined, namely  $J_{nr, diff}$  and  $R_{diff}$  which are measures of binder sensitivity to stress changes.

Results of bitumen tests with the MSCR method ( $J_{nr}$  and  $R$  for 0.1 kPa,  $J_{nr}$  and  $R$  for 3.2 kPa and  $J_{nr, diff}$  and  $R_{diff}$ ) are provided in Chapters 4 and 5.



## Chapter 3

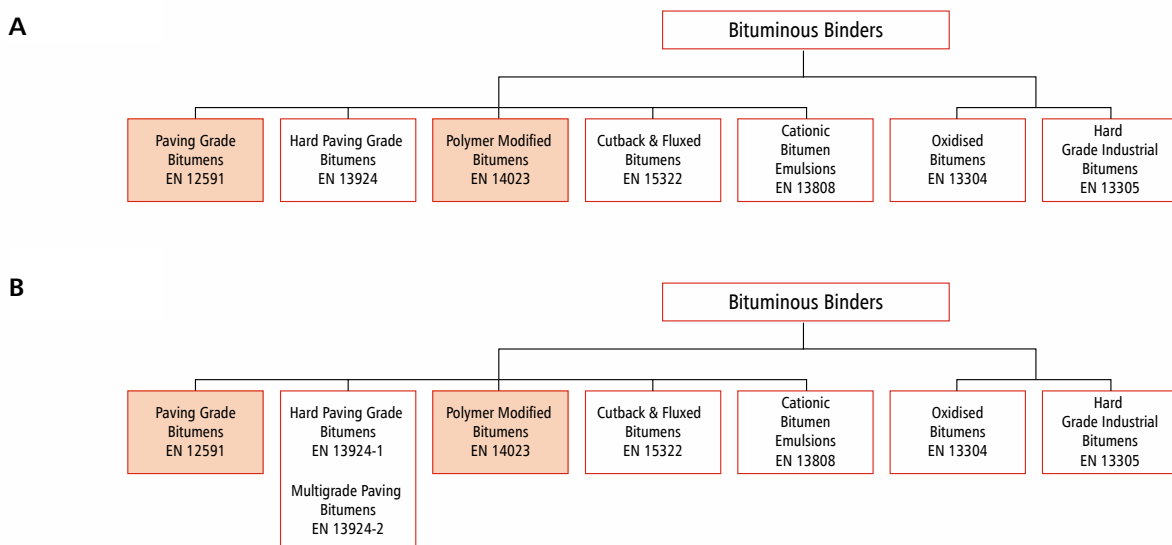
# DISCUSSION OF STANDARDS EN 12591 and EN 14023

### 3.1. Introduction

EN 12591 and EN 14023 are included in a set of European standards on bituminous binders. EN 12591 applies to paving-grade bitumen, and its most recent update comes from 2009 (EN 12591:2009), while EN 14023 discusses modified bitumen and was last updated in 2010 (EN 14023:2010).

EN 12591:2009 and EN 14023:2010 are developed on the basis of mandate, i.e. standardisation request from the European Commission. Originally, the standards supported essential requirements of the Construction Products Directive (Directive 89/106/EEC), repealed as of 30 June 2013 by Regulation No 305/2011 of the European Parliament and of the Council. As of 1 July 2013, construction products (including bituminous binders) are governed by Regulation No 305/2011.

Figure 3.1 presents statuses of the reference standards (current status and target status in 2014) in the European standardisation framework for bituminous binders.



**Figure 3.1.** Assignment of European standards to different types of binders. (A) Current status – prior to the publication of EN 13924-2. (B) Target status (2014) – upon publication of EN 13924-2 and change from EN 13924 to EN 13924-1. Standards discussed in this chapter are highlighted in colour.

### 3.2. Bituminous binder classification

Table 3.1. provides the classification of bituminous binders manufactured to European standards EN 12591 and EN 14023.

**Table 3.1.** Classification of bituminous binders manufactured to European standards EN 12591 and EN 14023

| Bituminous binder  | Paving Grade Bitumen                                    | Polymer Modified Bitumen  |
|--|---|---|
| Reference document   | EN 12591:2009   | EN 14023:2010   |
| Standard designation of bituminous binder  | <b>XX/YY</b>  | <b>PMB XX/YY-Z</b>  |
| Type of bituminous binder manufactured by ORLEN Asphalt  | 20/30<br>35/50<br>50/70<br>70/100<br>100/150<br>160/220 | ORBITON 10/40-65<br>ORBITON 25/55-55 EXP <sup>1)</sup><br>ORBITON 25/55-60<br>ORBITON 25/55-60 EXP <sup>2)</sup><br>ORBITON 25/55-65 EXP <sup>3)</sup><br>ORBITON 45/80-55<br>ORBITON 45/80-55 EXP <sup>4)</sup><br>ORBITON 45/80-65<br>ORBITON 65/105-60 |
| <p>1) ORBITON modified bitumen subtype for the German market<br/>                 2) ORBITON modified bitumen subtype for the Czech market<br/>                 3) ORBITON modified bitumen subtype for the Czech, Hungarian, Romanian and Slovakian market<br/>                 4) ORBITON modified bitumen subtype for the Lithuanian, Latvian and Estonian market</p> <p><b>Notes to designations:</b></p> <p>XX – lower penetration limit at 25°C for a given bitumen type [0.1 mm]<br/>                 YY – upper penetration limit at 25°C for a given bitumen type [0.1 mm]<br/>                 Z – lower softening point (R&amp;B) limit [°C] as per EN 1427<br/>                 PMB – stands for <i>polymer modified bitumen</i> (usually replaced by the manufacturer’s trade name)</p> |   |   |

### 3.3. Requirements of EN 12591 and EN 14023

EN 12591 and EN 14023 provide the rules for the selection of properties and the corresponding test methods, as well as the scope of requirements for bitumen designed for the construction and maintenance of roads, airfields and other pavements for wheeled traffic.

EN 12591:2009 “Bitumen and bituminous binders. Specifications for paving grade bitumens” is partially a classification standard, i.e. it delegates to member states of CEN (*European Committee for Standardization*) the right to select certain combinations of requirements, however, most of the requirements are fixed.

EN 14023:2010 “Bitumen and bituminous binders. Specification framework for polymer modified bitumens” does not set forth fixed requirements for the individual grades of bitumen (as most provisions of EN 12591:2009 do), but is a classification standard. It means that it provides a collection of properties and assigns various requirement levels to them (referred to as classes).

### 3.4. National application documents

EN 12591 and EN 14023 assume that each Member State of CEN makes the selection of properties and their assigned requirement levels in the so-called Application Documents to the discussed standards. Member States develop such documents as e.g. “National Annexes” to the standards, or as information about the selected requirements published in general, separate documents on bituminous pavement construction materials and technologies. This enables each Member State to specify its own requirements to be met by bituminous binders used in its territory. The differentiation results for diverse climate conditions in various parts of Europe and many other technology factors. “National annexes” and documents on bituminous pavement construction materials and technologies providing the requirements for bitumen are referred to below as “national requirements”.

Tables 3.2. and 3.3. provide example requirements for paving grade bitumen 35/50 which are **highlighted in red and bold**. Specifications for the remaining paving grade bitumen types (so-called National Annexes) for application in the specific Member State territory are established in a similar manner.

**Table 3.2** Requirements for paving grade bitumen with penetration from 20 × 0.1 mm to 220 × 0.1 mm acc. to EN 12591:200 (properties for all paving grade bitumens listed in the table). Example selection of properties for paving grade bitumen 35/50

| Property   | Test method                  | Unit   | Paving grade bitumen type |                          |                   |                   |                    |                    |
|--|------------------------------|--------|---------------------------|--------------------------|-------------------|-------------------|--------------------|--------------------|
|  |                              |        | 20/30                     | 35/50                    | 50/70             | 70/100            | 100/150            | 160/220            |
| Penetration at 25°C  | PN-EN 1426                   | 0,1 mm | 20-30                     | <b>35-50</b>             | 50-70             | 70-100            | 100-150            | 160-220            |
| Softening Point  | PN-EN 1427                   | °C     | 55-63                     | <b>50-58</b>             | 46-54             | 43-51             | 39-47              | 35-43              |
| Resistance to hardening, at 163°C  | PN-EN 12607-1 (RTFOT method) | %      |                           |                          |                   |                   |                    |                    |
| Retained penetration   |                              |        | ≥ 55                      | <b>≥ 53</b>              | ≥ 50              | ≥ 46              | ≥ 43               | ≥ 37               |
| Softening point increase – option 1 or Softening point increase – option 2 <sup>a</sup>  |                              |        | ≤ 8<br>or<br>≤ 10         | <b>≤ 8</b><br>or<br>≤ 11 | ≤ 9<br>or<br>≤ 11 | ≤ 9<br>or<br>≤ 11 | ≤ 10<br>or<br>≤ 12 | ≤ 11<br>or<br>≤ 12 |
| Change of mass <sup>b</sup> (absolute)   |                              |        | ≤ 0,5                     | <b>≤ 0,5</b>             | ≤ 0,5             | ≤ 0,8             | ≤ 0,8              | ≤ 1,0              |
| Flash point  | PN-EN ISO 2592               | °C     | ≥ 240                     | <b>≥ 240</b>             | ≥ 230             | ≥ 230             | ≥ 230              | ≥ 220              |
| Solubility   | PN-EN 12592                  | %      | ≥ 99,0                    | <b>≥ 99,0</b>            | ≥ 99,0            | ≥ 99,0            | ≥ 99,0             | ≥ 99,0             |
| <p>a – If option 2 is selected, it must be combined with the requirements for the Fraass breaking point or penetration index, or both, determined for the binder subjected to ageing.</p> <p>b – Change in mass may be a positive or negative value.</p> |                              |        |                           |                          |                   |                   |                    |                    |

**Table 3.3** Requirements for paving grade bitumen with penetration from 20 × 0.1 mm to 220 × 0.1 mm acc. to EN 12591:2009 (properties relating to laws and regulations or other national requirements). Example selection of properties for paving grade bitumen 35/50 (continued)

| Property   | Test method         | Unit               | Paving grade bitumen type             |   |                                       |                                       |                                       |                                       |
|--|---------------------|--------------------|---------------------------------------|---|---------------------------------------|---------------------------------------|---------------------------------------|---------------------------------------|
|  |                     |                    | 20/30                                 | 35/50                                       | 50/70                                 | 70/100                                | 100/150                               | 160/220                               |
| Penetration index  | PN-EN 12591 Annex A | –                  | -1.5 to +0.7<br>or<br>NR <sup>b</sup> | -1.5 to +0.7<br>or<br><b>NR<sup>b</sup></b> | -1.5 to +0.7<br>or<br>NR <sup>b</sup> | -1.5 to +0.7<br>or<br>NR <sup>b</sup> | -1.5 to +0.7<br>or<br>NR <sup>b</sup> | -1.5 to +0.7<br>or<br>NR <sup>b</sup> |
| Dynamic viscosity at 60°C  | PN-EN 12596         | Pa · s             | ≥ 440<br>or<br>NR <sup>b</sup>        | <b>≥ 225</b><br>or<br>NR <sup>b</sup>       | ≥ 145<br>or<br>NR <sup>b</sup>        | ≥ 90<br>or<br>NR <sup>b</sup>         | ≥ 55<br>or<br>NR <sup>b</sup>         | ≥ 30<br>or<br>NR <sup>b</sup>         |
| Fraass Breaking point <sup>a</sup>   | PN-EN 12593         | °C                 | NR <sup>b</sup>                       | <b>≤ -5</b><br>or<br>NR <sup>b</sup>        | ≤ -8<br>or<br>NR <sup>b</sup>         | ≤ -10<br>or<br>NR <sup>b</sup>        | ≤ -12<br>or<br>NR <sup>b</sup>        | ≤ -15<br>or<br>NR <sup>b</sup>        |
| Kinematic viscosity at 135°C   | PN-EN 12595         | mm <sup>2</sup> /s | ≥ 530<br>or<br>NR <sup>b</sup>        | <b>≥ 370</b><br>or<br>NR <sup>b</sup>       | ≥ 295<br>or<br>NR <sup>b</sup>        | ≥ 230<br>or<br>NR <sup>b</sup>        | ≥ 175<br>or<br>NR <sup>b</sup>        | ≥ 135<br>or<br>NR <sup>b</sup>        |
| <p>a – If option 2 is selected, it must be combined with the requirements for the Fraass breaking point or penetration index, or both, determined for the binder subjected to ageing.</p> <p>b – NR – no requirements, it can be used in the case where there are no national requirements for a given property at the location of its intended use.</p> |                     |                    |                                       |   |                                       |                                       |                                       |                                       |

National specifications for polymer-modified bitumen as per EN 14023 are designed in a similar manner; however, the number of properties and available classes (requirement levels) is much greater than in EN 12591.

Tables 3.4, 3.5 and 3.6 give examples of primary and additional requirements for modified bitumen ORBITON PMB 45/80-55 EXP, which are provided in frames in red. Specifications for other polymer modified bitumens are designed in a similar fashion.

**Table 3.4.** Rules for polymer-modified bitumen classification – Properties applicable to all polymer-modified bitumens (EN 14023:2010). Example selection of properties for modified bitumen ORBITON 45/80-55 EXP

| Property  |  | Test method             | Unit              | Classes for all polymer modified bitumens |            |            |            |             |             |              |             |               |               |  |
|---|--|-------------------------|-------------------|---|------------|------------|------------|-------------|-------------|--------------|-------------|---------------|---------------|--|
|   |  |                         |                   | 2   | 3          | 4          | 5          | 6           | 7           | 8            | 9           | 10            | 11            |  |
| Penetration at 25°C                               |  | EN 1426                 | 0,1 mm            | 25-55                                     | 45-80      | 40-100     | 65-105     | 75-130      | 90-150      | 120-200      | 200-300     |               |               |  |
| Softening point                                   |  | EN 1427                 | °C                | ≥ 80                                      | ≥ 75       | ≥ 70       | ≥ 65       | ≥ 60        | ≥ 55        | ≥ 50         | ≥ 45        | ≥ 40          |               |  |
| Cohesion <sup>a</sup>                             | Force ductility by ductilometer method a (tension of 50 mm/min) or | EN 13589 after EN 13703 | J/cm <sup>2</sup> | ≥ 3 at 5°C                                | ≥ 2 at 5°C | ≥ 1 at 5°C | ≥ 2 at 0°C | ≥ 2 at 10°C | ≥ 3 at 10°C | ≥ 0.5 at 5°C | ≥ 2 at 15°C | ≥ 0.5 at 20°C | ≥ 0.5 at 25°C |  |
|   | Direct tension <sup>a</sup> (tension of 100 mm/min) or             | EN 13587 after EN 13703 | J/cm <sup>2</sup> | ≥ 3 at 5°C                                | ≥ 2 at 5°C | ≥ 1 at 5°C | ≥ 3 at 0°C | ≥ 3 at 10°C |             |              |             |               |               |  |
|   | Vialit pendulum (impact test)                                      | EN 13588                | J/cm <sup>2</sup> | ≥ 0.7                                     |            |            |            |             |             |              |             |               |               |  |
| Purobility (Resistance to hardening) <sup>b</sup> | Retained penetration at 25°C after RTFOT                           | EN 12607-1              | %                 | ≥ 35                                      | ≥ 40       | ≥ 45       | ≥ 50       | ≥ 55        | ≥ 60        |              |             |               |               |  |
|   | Softening point increase after RTFOT                               |                         | %                 | ≤ 8                                       | ≤ 10       | ≤ 12       |            |             |             |              |             |               |               |  |
|   | Change of mass after RTFOT <sup>c</sup>                            |                         | °C                | ≤ 0.3                                     | ≤ 0.5      | ≤ 0.8      | ≤ 1.0      |             |             |              |             |               |               |  |
| Flash Point                                       |  | EN ISO 2592             | %                 | ≥ 250                                     | ≥ 235      | ≥ 220      |            |             |             |              |             |               |               |  |

a) Only one method for cohesion testing should be selected, depending on the final application. Determination of cohesion by Vialit method (EN 13588) should be selected only for binders designed for surface dressing.  
b) The primary test method is RTFOT at 163°C. For certain polymer-modified bitumens, whose viscosity is too high, RTFOT at the reference temperature of 163°C cannot be performed because the movement of bitumen layer must be ensured. In such cases, the test should be conducted at 180°C as per EN 12607-1.  
c) Change in mass may be a positive or negative value.

**Table 3.5.** Rules for polymer-modified bitumen classification – Properties resulting from laws and regulations or other regional requirements (EN 14023:2010)

| Property              |                         | Test method | Unit | Classes for regional requirements |                  |      |      |      |       |       |       |       |       |       |
|-----------------------|-------------------------|-------------|------|-----------------------------------|------------------|------|------|------|-------|-------|-------|-------|-------|-------|
|                       |                         |             |      | 0                                 | 1                | 2    | 3    | 4    | 5     | 6     | 7     | 8     | 9     | 10    |
| Fraass Breaking point |                         | EN 12593    | °C   | NR <sup>a</sup>                   | TBR <sup>b</sup> | ≤ 0  | ≤ -5 | ≤ -7 | ≤ -10 | ≤ -12 | ≤ -15 | ≤ -18 | ≤ -20 | ≤ -22 |
| Elastic recovery      | at 25°C or <sup>c</sup> | EN 13398    | %    | NR <sup>a</sup>                   | TBR <sup>b</sup> | ≥ 80 | ≥ 70 | ≥ 60 | ≥ 50  |       |       |       |       |       |
|                       | at 10°C                 | EN 13398    | %    | NR <sup>a</sup>                   | TBR <sup>b</sup> | ≥ 75 | ≥ 50 |      |       |       |       |       |       |       |

a) NR. No requirements – can be used in the case where there are no national requirements for a given property at the location of its intended use.  
b) TBR. To be declared – can be used in the case where there are no national requirements for a given property at the location of its intended use; however, a given property is considered useful for the description of polymer-modified bitumen.  
c) Where required, polymer-modified bitumens should meet the requirements for elastic recovery at 25 °C or 10 °C.



**Table 3.6.** Rules for polymer-modified bitumen classification – Properties resulting from laws and regulations or other national requirements (EN 14023:2010)

| Property   | Test method         | Unit   | Classes for the additional properties of polymer modified bitumens |                  |            |      |             |      |      |      |
|--|---------------------|--------|--|------------------|------------|------|-------------|------|------|------|
|  |                     |        | 0  | 1                | 2          | 3    | 4           | 5    | 6    | 7    |
| Plasticity range   | Subsection 5.2.8.4. | °C     | NR <sup>a</sup>  | <b>TBR</b>       | ≥ 85       | ≥ 80 | ≥ 75        | ≥ 70 | ≥ 65 | ≥ 60 |
| Softening point drop after EN 12607-1                        | EN 1427             | °C     | NR <sup>a</sup>  | TBR              | ≤ 2        | ≤ 5  |             |      |      |      |
| Elastic recovery at 25°C after EN 12607-1                    | EN 13398            | %      | NR <sup>a</sup>  | TBR              | ≥ 70       | ≥ 60 | <b>≥ 50</b> |      |      |      |
| Elastic recovery at 10°C after EN 12607-1                    | EN 13398            | %      | <b>NR<sup>a</sup></b>  | TBR              | ≥ 50       |      |             |      |      |      |
| Storage stability <sup>b</sup><br>Softening point difference | EN 13399<br>EN 1427 | °C     | NR <sup>a</sup>  | TBR <sup>b</sup> | <b>≤ 5</b> |      |             |      |      |      |
| Storage stability <sup>b</sup><br>Penetration difference     | EN 13399<br>EN 1426 | 0.1 mm | NR <sup>a</sup>  | TBR <sup>b</sup> | <b>≤ 9</b> | ≤ 13 | ≤ 19        | ≤ 26 |      |      |

a) NR. No requirements – can be used in the case where there are no national requirements for a given property at the location of its intended use.  
b) Storage conditions for polymer-modified bitumen should be indicated by the supplier. Homogeneity is required for polymer-modified bitumens. The inclination of polymer-modified bitumen's constituents to separate can be evaluated through the determination of storage stability (see EN 13399). If the product fails to meet the requirements of Table 3, classes from 2 to 5, the supplier should state what should be the storage method for polymer-modified bitumen in order to avoid the separation of its components and ensure product homogeneity.

### 3.5. Evaluation of conformity in EN 12591 and EN 14023

Annexes ZA to EN 12591:2009 and EN 14023:2010 establish the requirements for CE marking. Annexes ZA also provide the procedure for bituminous binder conformity assessment and the division of responsibilities between the manufacturer and the notified body, and contain a chapter on certification and declaration of performance (formerly declaration of conformity<sup>1)</sup>, CE marking and labelling.

Bituminous binders for road construction and surface dressing are covered by the conformity assessment system "2+". Under the system, the manufacturer is required to establish, document and maintain the Factory Production Control system, confirmed by the FPC Certificate (issued by the notified body). In addition, the manufacturer should have in place the sample testing plan and perform type examination for each product. The FPC system should comprise procedures, regular inspections and tests and/or assessments, and the results should be used for the finished product quality assessment. Numbers of FPC Certificates for production units of the ORLEN Asphalt Group are provided in Chapter 1.

The assessment of bituminous binder properties' conformity with the requirements of the standards under consideration and the values they establish (including grades) should be demonstrated by:

- performance of an initial type examination of each type of bitumen,
- implementation and operation of the Factory Production Control (FPC).

1) The effective Regulation No 305/2011 of the European Parliament and of the Council, repealing the Construction Products Directive (89/106/EEC), changes the name "declaration of conformity" into "declaration of performance", and provides the information it must contain.

Figure 3.2. shows example information accompanying the CE marking of paving-grade bitumen 50/70 manufactured by ORLEN Asphalt in 2013.


|   |   |
|---|---|
| <br>1434   | <p><i>CE conformity marking, comprising the CE mark as indicated in Directive 93/68/EEC</i></p> <p><i>Identification number of the notified body</i></p>  |
| <p><b>ORLEN Asphalt sp. z o.o.</b><br/> <b>PRODUKCJA PŁOCK</b><br/>                 PRODUCTION PŁOCK<br/>                 09-411 Płock, ul. Chemików 7<br/>                 Poland</p> <p><b>13</b></p> <p>3/CPR/2013</p>   | <p><i>Name or identification mark and the manufacturer's registered address</i></p> <p><i>Two last digits of the year in which the marking was placed</i></p> <p><i>Declaration of performance number</i></p> |
| <p><b>PN-EN 12591:2010</b></p> <p><b>Asfalt drogowy: 50/70</b><br/> <b>Paving grade bitumen: 50/70</b></p> <p>Penetracja w 25°C<br/>                 (Penetration at 25°C) ..... 50-70 0,1mm<br/>                 Temperatura mięknięcia<br/>                 (Softening Point R&amp;B) .....46 – 54°C<br/>                 Odporność na starzenie w 163°C (EN 12607-1)<br/>                 (Resistance to hardening at 163°C (EN 12607-1))<br/>                 Pozostała penetracja w 25°C po starzeniu<br/>                 (Retained penetration at 25°C after RTFOT) .....50%<br/>                 Wzrost temperatury mięknięcia po starzeniu<br/>                 (Increasing of Softening Point R&amp;B after RTFOT) .....9°C<br/>                 Zmiana masy po starzeniu<br/>                 (Change of mass after RTFOT) .....0,5%<br/>                 Temperatura zapłonu<br/>                 (Flash point (COC)) .....230°C<br/>                 Zawartość składników rozpuszczalnych<br/>                 (Solubility in toluene) .....99,0% m/m<br/>                 Lepkość dynamiczna w 60°C<br/>                 (Dynamic viscosity at 60°C) .....≥ 145 Pa*s<br/>                 Temperatura łamliwości<br/>                 (Fraass breaking point) .....-8°C<br/>                 Indeks penetracji<br/>                 (Penetration Index) .....NR<br/>                 Lepkość kinematyczna w 135°C<br/>                 (Kinematic viscosity at 135°C) .....NR</p> | <p><i>European Standard number</i></p> <p><i>Product description and information about verified properties</i></p>  |

Figure 3.2. CE marking for paving-grade bitumen 50/70 manufactured by ORLEN Asphalt in 2013



## Chapter 4

# PAVING GRADE BITUMENS ACC. TO EN 12591

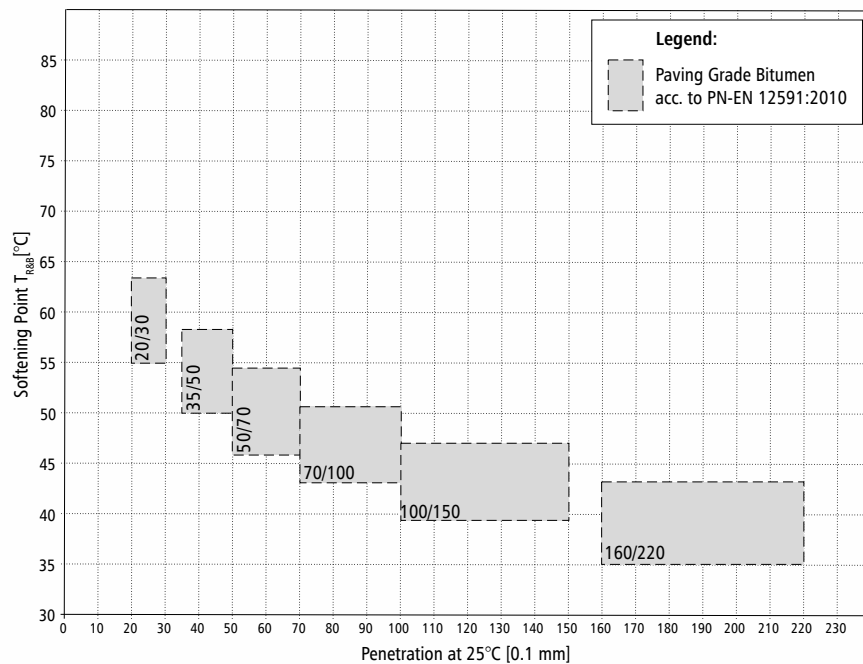
### 4.1. General description

Paving Grade Bitumens are the most popular binders for hot mix asphalt used for road pavement construction. In April 2009, CEN published the latest revision of EN 12591 (previous edition from 1999) adapted to the conformity assessment system 2+. From September 2010, ORLEN Asphalt has been manufacturing paving-grade bitumen in accordance with the requirements of EN 12591:2009, used in the Polish standardisation framework as PN-EN 12591:2010 with the National Annex NA. Bituminous binders produced in other countries also meet local (national) requirements.

ORLEN Asphalt manufactures the following types of Paving Grade Bitumens according to EN 12591:2009: 20/30, 35/50, 50/70, 70/100, 100/150 and 160/220. All of them are classified as paving-grade bitumens with the penetration range 20–220 [0.1 mm], tested at 25°C.

Bitumen 20/30 is among the hardest types according to EN 12591. Bitumens 35/50, 50/70 and 70/100 demonstrate high and medium hardness and are most commonly used for road works. Our offering also comprises soft bitumen, designated as 100/150 and 160/220.

A schematic comparison of primary bitumen properties for the two most popular bituminous binder parameters, namely penetration at 25°C and softening point TR&B is shown on Figure 4.1.



**Figure 4.1.** Schematic comparison of Paving Grade Bitumens acc. to EN 12591:2009 as a function of penetration at 25°C and softening point  $T_{R\&B}$

## 4.2. Properties

Subsequent sections of this chapter discuss all properties of paving-grade bitumens as per EN 12591, including additional details based on the American *Superpave (Performance Grade system)*. This chapter also provides approximate process temperatures for bitumen application in bituminous mixtures, viscosity data and viscosity dependence on temperature.

The classification of paving-grade bitumens intended for vehicle traffic on the basis of the MSCR test provided in this Chapter 4 is discussed in Chapter 7.

### 4.2.1. Paving Grade Bitumen 20/30

#### Intended use

**Paving Grade Bitumen 20/30** is the hardest paving-grade bitumen from the range currently manufactured by ORLEN Asphalt. Its high softening point and high sensitivity to low-temperature cracks renders it recommendable solely for the binder course and high modulus asphalt concrete base in regions with suitable climate. Courses with bitumen 20/30 should not be left over winter without applying a subsequent (covering) course.

#### Properties acc. to EN 12591:2009

**Table 4.1.** Properties of paving-grade bitumen 20/30

| Property                              | Test method           | Unit               | Requirement |
|---------------------------------------|-----------------------|--------------------|-------------|
| Penetration at 25°C                   | EN 1426               | 0.1 mm             | 20 – 30     |
| Softening Point R&B                   | EN 1427               | °C                 | 55 – 63     |
| Fraass Breaking Point                 | EN 12593              | °C                 | NR          |
| Flash point                           | EN ISO 2592           | °C                 | ≥ 240       |
| Solubility                            | EN 12592              | % (m/m)            | ≥ 99.0      |
| Change of mass after RTFOT (absolute) | EN 12607-1            | % (m/m)            | ≤ 0.5       |
| Retained penetration after RTFOT      | EN 12607-1<br>EN 1426 | %                  | ≥ 55        |
| Softening Point increase after RTFOT  | EN 12607-1<br>EN 1427 | °C                 | ≤ 8         |
| Kinematic viscosity at 135°C          | EN 12595              | mm <sup>2</sup> /s | ≥ 530       |
| Dynamic viscosity at 60°C             | EN 12596              | Pa*s               | ≥ 440       |

#### Fraass Breaking Point

ORLEN Asphalt has adopted a principle that, although not required by the standards, each production batch of bitumen 20/30 will also be tested for the breaking point. Table 4.2 presents statistical data from inspections in 2011–2013. Considering that the introduction of the Fraass Breaking Point requirement for bitumen samples after RTFOT is currently under discussion, Table 4.3 provides the results of the relevant tests.

**Table 4.2.** Results of the primary properties of bitumen 20/30: penetration at 25°C, R&B softening point and Fraass breaking point from 2011-2013

| Property  | Arithmetic means from tests on all production batches<br>(standard deviations in parentheses) |            |            |
|---|---|------------|------------|
|   | 2011  | 2012       | 2013       |
| Penetration at 25°C, 0.1 mm EN 1426                             | 27.8 (1.5)  | 27.9 (1.7) | 28.0 (1.2) |
| Softening Point R&B, °C EN 1427                                 | 61.8 (1.1)  | 62.4 (0.6) | 62.0 (0.6) |
| Fraass Breaking Point*), °C EN 12593                            | -7.5 (1.8)  | -7.5 (1.5) | -8.6 (2.3) |
| *) Property not required by the standard, tested as an addition |   |            |            |

**Table 4.3.** Means of bitumen 20/30 primary parameters after RTFOT acc. to EN 12607-1 for penetration at 25°C, R&B softening point and Fraass breaking point. Data from 2013.

| Property  | Arithmetic means<br>from tests conducted once per month<br>(standard deviations in parentheses) |
|---|---|
|   | 2013  |
| Penetration at 25°C, 0.1 mm acc. EN 1426 after RTFOT            | 19.8 (1.7)  |
| Softening Point R&B, °C acc. EN 1427 after RTFOT                | 68.8 (1.5)  |
| Fraass Breaking Point, °C, EN 12593, after RTFOT *)             | -7.1 (2.1)  |
| *) Property not required by the standard, tested as an addition |   |

### Properties acc. to Superpave

Bitumen 20/30 properties acc. to *Superpave* (tests conducted in 2009-2012).

- classification as per AASHTO MP 1: **PG 82-16**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (unaged bitumen)  $T_{crit} = 83.7^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 84.7^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\ 000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 26.0^\circ\text{C}$
- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  Pa  $T(S)_{60} = -14.7^\circ\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -8.1^\circ\text{C}$
  - stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 370.5$  MPa
- results and classification based on the MSCR (discussion in Chapter 7, Table 7.5)

| Temperature  | 64°C           |
|--|----------------|
| $J_{nr}$ 0.1 kPa   | 0.169          |
| $J_{nr}$ 3.2 kPa   | 0.185          |
| $J_{nr, diff}$   | 9.7            |
| R 0.1 kPa  | 33.4           |
| R 3.2 kPa  | 28.9           |
| R <sub>diff</sub>  | 13             |
| Final classification of suitability for road traffic<br>(at the test temperature) acc.to the most recent PG classification | E<br>(Extreme) |

## Viscosity dependence on temperature

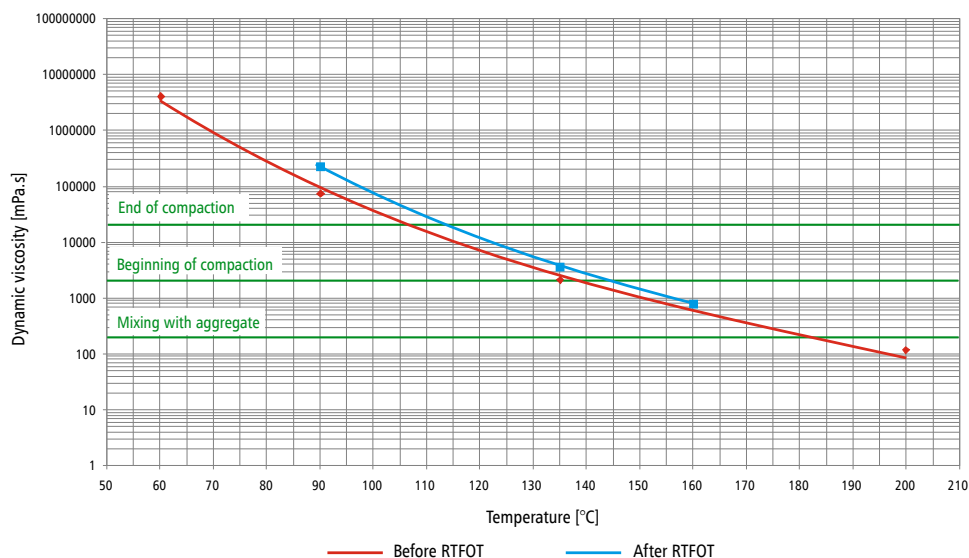


Figure 4.2. Viscosity dependence on temperature for Paving Grade Bitumen 20/30

Table 4.4. Example results of viscosity tests on bitumen 20/30 manufactured in 2012–2013. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type       | Test method                  | Reference document       | Equipment parameters | Unit               | Test temperature    | Example test result for viscosity |
|----------------------|------------------------------|--------------------------|----------------------|--------------------|---------------------|-----------------------------------|
| dynamic              | vacuum capillary             | EN 12596                 | —                    | Pa*s               | 60°C                | 3 313                             |
|                      | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle<br>No 21, 29 | Pa*s               | 90°C                | 70.80                             |
|                      |                              |                          |                      |                    | 135°C               | 2.06                              |
|                      |                              |                          | spindle<br>No 27     | Pa*s               | 160°C               | 0.43                              |
|                      |                              |                          |                      |                    | 90°C<br>after RTFOT | 209.00                            |
|                      |                              |                          |                      |                    |                     | 135°C<br>after RTFOT              |
| 160°C<br>after RTFOT |                              |                          |                      |                    |                     | 0.73                              |
| kinematic            | BS/IP/RF viscometer          | EN 12595                 | —                    | mm <sup>2</sup> /s | 135°C               | 1 655                             |

## Process temperatures

| At laboratory  |           |
|--|-----------|
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press)  | 155–160°C |
| At mixing plant  |           |
| Bitumen pumping temperature  | > 145°C   |
| Temperature of bitumen for bituminous mixture production   | 175–185°C |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 8 h)  | < 230°C   |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 4 h)  | < 240°C   |
| <b>Note:</b> it is recommended for MA production to use additives to reduce the process temperature (mixing with aggregate and placement) so that the mastic asphalt production takes place at a temperature below 200°C |           |
| At site  |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)   | 165°C     |

## Storage

### Short-term storage at high temperature (up to 10 days)

- recommended bitumen storage temperature:  $\leq 185^{\circ}\text{C}$

### Long-term storage at high temperature (over 10 days)

Bitumen storage at high temperatures over prolonged periods of time should be avoided. If it is necessary to store paving-grade bitumen 20/30 in the silo at a high temperature (up to  $185^{\circ}\text{C}$ ) for over 10 days, it is recommended to inspect the binder ageing rate before bitumen application for the production of bituminous mixtures. The following should be checked: penetration at  $25^{\circ}\text{C}$  (EN 1426) or softening point (EN 1427).

In the case of excessive bitumen ageing, the procedure for controlled product disposal should be initiated (FPC procedure compliant with EN 13108-21).

### Long-term storage at low temperature (over 10 days)

If it is required to store Paving Grade Bitumen 20/30 for a much longer period than 10 days, it is recommended to reduce the bitumen temperature and reheat before reuse. If a long storage period is envisaged without bituminous mixture production, bitumen storage at the ambient temperature is allowed. A precondition for such storage is fitting the silo with heating equipment having sufficient capacity to ensure subsequent bitumen heating without running the risk of local binder overheat during long-term heating.

## 4.2.2. Paving Grade Bitumen 35/50

### Intended use

Paving-grade bitumen 35/50 can be used for asphalt concrete in base and binder courses for light, medium and heavy traffic roads. Bitumen 35/50 should not be used for wearing courses.

### Properties acc. to EN 12591:2009

**Table 4.5.** Properties of paving-grade bitumen 35/50

| Property                                     | Test method           | Unit                     | Requirement |
|--|-----------------------|--------------------------|-------------|
| Penetration at $25^{\circ}\text{C}$          | EN 1426               | 0.1 mm                   | 35 – 50     |
| Softening Point R&B                          | EN 1427               | $^{\circ}\text{C}$       | 50 – 58     |
| Fraass Breaking Point                        | EN 12593              | $^{\circ}\text{C}$       | $\leq -5$   |
| Flash point                                  | EN ISO 2592           | $^{\circ}\text{C}$       | $\geq 240$  |
| Solubility                                   | EN 12592              | % (m/m)                  | $\geq 99.0$ |
| Change of mass after RTFOT (absolute)        | EN 12607-1            | % (m/m)                  | $\leq 0.5$  |
| Retained penetration after RTFOT             | EN 12607-1<br>EN 1426 | %                        | $\geq 53$   |
| Softening Point increase after RTFOT         | EN 12607-1<br>EN 1427 | $^{\circ}\text{C}$       | $\leq 8$    |
| Kinematic viscosity at $135^{\circ}\text{C}$ | EN 12595              | $\text{mm}^2/\text{s}$   | $\geq 370$  |
| Dynamic viscosity at $60^{\circ}\text{C}$    | EN 12596              | $\text{Pa}\cdot\text{s}$ | $\geq 225$  |

## Properties as per Superpave

Bitumen 35/50 properties acc. to *Superpave* (tests conducted in 2009–2012).

- classification acc. to AASHTO MP 1: **PG 70-16**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (unaged bitumen)  $T_{crit} = 73.2^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 74.2^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\ 000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 23.1^\circ\text{C}$
- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  Pa  $T(S)_{60} = -15.4^\circ\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -11.5^\circ\text{C}$
  - stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 338.5$  MPa
- results and classification based on the MSCR (discussion in Chapter 7, Table 7.5)

| Temperature  | 64°C              |
|--|-------------------|
| $J_{nr}$ 0.1 kPa   | 0.882             |
| $J_{nr}$ 3.2 kPa   | 1.004             |
| $J_{nr}$ diff  | 13.8              |
| R 0.1 kPa  | 12.5              |
| R 3.2 kPa  | 5.9               |
| R diff   | 53                |
| Final classification of suitability for road traffic (at the test temperature) acc. to the most recent PG classification | V<br>(Very Heavy) |

## Process temperatures

| At laboratory  |           |
|--|-----------|
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press)  | 140–145°C |
| At mixing plant  |           |
| Bitumen pumping temperature  | > 140°C   |
| Temperature of bitumen for bituminous mixture production   | 165–175°C |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 8 h)  | < 230°C   |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 4 h)  | < 240°C   |
| <b>Note:</b> it is recommended for MA production to use additives to reduce the process temperature (mixing with aggregate and placement) so that the mastic asphalt production takes place at a temperature below 200°C |           |
| At site  |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)   | 150°C     |



## Viscosity dependence on temperature

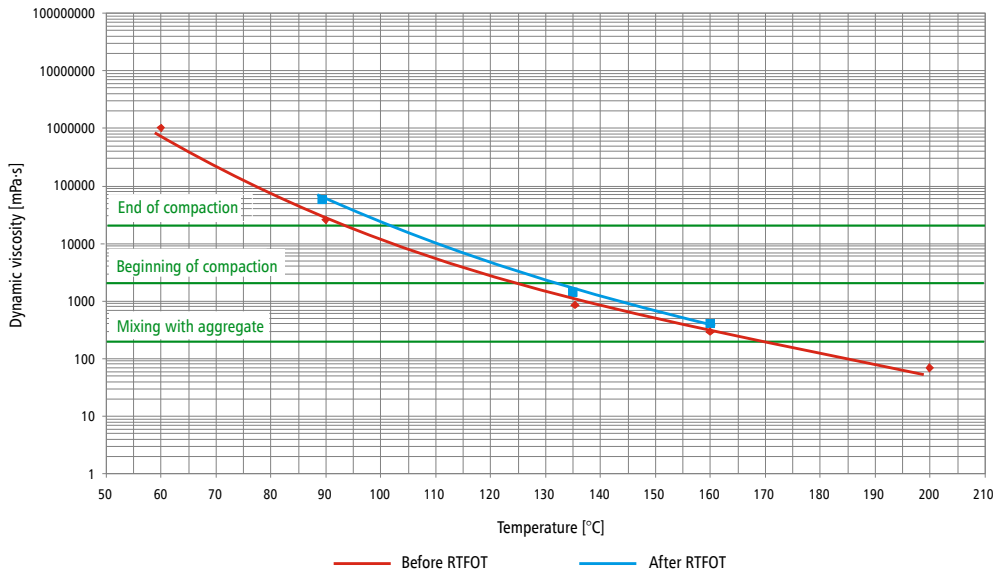


Figure 4.3. Viscosity dependence on temperature for Paving Grade Bitumen 35/50

Table 4.6. Example results of viscosity tests on bitumen 35/50 manufactured in 2012-2013. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit  | Test temperature     | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|-------|----------------------|-----------------------------------|
| dynamic        | vacuum capillary             | EN 12596                 | —                    | Pa*s  | 60°C                 | 758                               |
|                | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle<br>No 21, 29 | Pa*s  | 90°C                 | 23.91                             |
|                |                              |                          |                      |       | 135°C                | 0.82                              |
|                |                              |                          | spindle<br>No 27     | Pa*s  | 90°C<br>after RTFOT  | 55.00                             |
|                |                              |                          |                      |       | 135°C<br>after RTFOT | 1.42                              |
|                | 160°C<br>after RTFOT         | 0.38                     |                      |       |                      |                                   |
| kinematic      | BS/IP/RF viscometer          | EN 12595                 | —                    | mm²/s | 135°C                | 856                               |

### Storage

#### Short-term storage at high temperature (up to 10 days)

- recommended bitumen storage temperature:  $\leq 185^{\circ}\text{C}$

#### Long-term storage at high temperature (over 10 days)

Bitumen storage at high temperatures over prolonged periods of time should be avoided. If it is necessary to store paving-grade bitumen 35/50 in the silo at a high temperature (up to 185°C) for over 10 days, it is recommended to inspect the binder ageing rate before bitumen application for the production of bituminous mixtures. The following should be checked: penetration at 25°C (EN 1426) or softening point (EN 1427).

In the case of excessive bitumen ageing, the procedure for controlled product disposal should be initiated (FPC procedure compliant with EN 13108-21).

### Long-term storage at low temperature (over 10 days)

If it is required to store Paving Grade Bitumen 35/50 for a much longer period than 10 days, it is recommended to reduce the bitumen temperature and reheat before reuse. If a long storage period is envisaged without bituminous mixture production, bitumen storage at the ambient temperature is allowed. A precondition for such storage is fitting the silo with heating equipment having sufficient capacity to ensure subsequent bitumen heating without running the risk of local binder overheat during long-term heating.

## 4.2.3. Paving Grade Bitumen 50/70

### Intended use

Paving Grade Bitumen 50/70 can be used primarily for asphalt concrete and SMA in wearing courses for light and medium traffic, assuming the requirements for mix resistance to rutting are met.

### Properties acc. to EN 12591:2009

**Table 4.7.** Properties of paving-grade bitumen 50/70

| Property                              | Test method           | Unit               | Requirement |
|---------------------------------------|-----------------------|--------------------|-------------|
| Penetration at 25°C                   | EN 1426               | 0.1 mm             | 50 – 70     |
| Softening Point R&B                   | EN 1427               | °C                 | 46 – 54     |
| Fraass Breaking point                 | EN 12593              | °C                 | ≤ -8        |
| Flash point                           | EN ISO 2592           | °C                 | ≥ 230       |
| Solubility                            | EN 12592              | % (m/m)            | ≥ 99.0      |
| Change of mass after RTFOT (absolute) | EN 12607-1            | % (m/m)            | ≤ 0.5       |
| Retained penetration after RTFOT      | EN 12607-1<br>EN 1426 | %                  | ≥ 50        |
| Softening Point increase after RTFOT  | EN 12607-1<br>EN 1427 | °C                 | ≤ 9         |
| Kinematic viscosity at 135°C          | EN 12595              | mm <sup>2</sup> /s | ≥ 295       |
| Dynamic viscosity at 60°C             | EN 12596              | Pa*s               | ≥145        |

### Properties as per Superpave

Bitumen 50/70 properties acc. to *Superpave* (tests conducted in 2009-2012).

- classification as per AASHTO MP 1: **PG 64-22**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (fresh bitumen)  $T_{crit} = 67.7^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 67.8^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 20.5^\circ\text{C}$

- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa  $T(S)_{60} = -16.6^{\circ}\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -15.0^{\circ}\text{C}$
  - stiffness at  $-16^{\circ}\text{C}$   $S(T)_{-16} = 294$  MPa

- results and classification based on the MSCR (discussion in Chapter 7, Table 7.5)

| Temperature  | 58°C           | 64°C         |
|--|----------------|--------------|
| $J_{nr}$ 0.1 kPa   | 0.730          | 2.280        |
| $J_{nr}$ 3.2 kPa   | 0.810          | 2.588        |
| $J_{nr}$ diff  | 11.0           | 13.5         |
| R 0.1 kPa  | 11.5           | 4.5          |
| R 3.2 kPa  | 4.0            | 0.5          |
| R diff   | 66             | 89           |
| Final classification of suitability for road traffic (at the test temperature) acc. to the most recent PG classification | V (Very Heavy) | S (Standard) |

## Process temperatures

|   |           |
|---|-----------|
| <b>At laboratory</b>  |           |
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press) | 135–140°C |
| <b>At mixing plant</b>  |           |
| Bitumen pumping temperature   | > 130°C   |
| Temperature of bitumen for bituminous mixture production                                | 155–165°C |
| <b>At site</b>  |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)              | 145°C     |

## Viscosity dependence on temperature

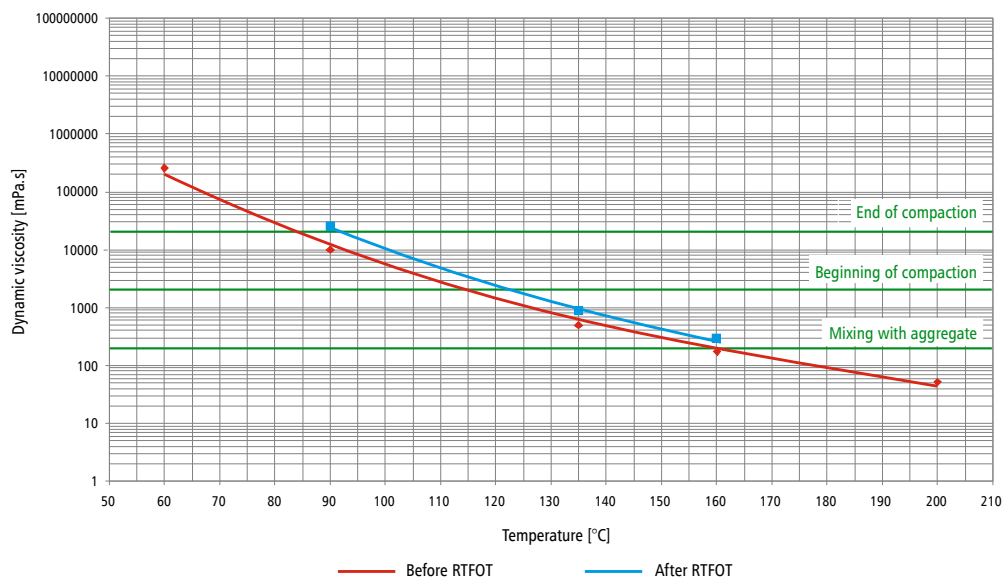


Figure 4.4. Viscosity dependence on temperature for Paving Grade Bitumen 50/70

**Table 4.8.** Example results of viscosity tests on bitumen 50/70 manufactured in 2012–2013. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit               | Test temperature    | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|--------------------|---------------------|-----------------------------------|
| dynamic        | vacuum capillary             | EN 12596                 | —                    | Pa*s               | 60°C                | 292                               |
|                | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle<br>No 21     | Pa*s               | 90°C                | 9.50                              |
|                |                              |                          |                      |                    | 135°C               | 0.46                              |
|                |                              |                          |                      |                    | 160°C               | 0.17                              |
|                |                              |                          |                      |                    | 90°C<br>after RTFOT | 22.59                             |
|                | spindle<br>No 27             | Pa*s                     | 135°C<br>after RTFOT | 0.81               |                     |                                   |
|                |                              |                          | 160°C<br>after RTFOT | 0.24               |                     |                                   |
| kinematic      | BS/IP/RF viscometer          | EN 12595                 | —                    | mm <sup>2</sup> /s | 135°C               | 505                               |

## Storage

### Short-term storage at high temperature (up to 10 days)

- recommended bitumen storage temperature:  $\leq 185^{\circ}\text{C}$

### Long-term storage at high temperature (over 10 days)

Bitumen storage at high temperatures over prolonged periods of time should be avoided. If it is necessary to store Paving Grade Bitumen 50/70 in the silo at a high temperature (up to 185°C) for over 10 days, it is recommended to inspect the binder ageing rate before bitumen application for the production of bituminous mixtures. The following should be checked: penetration at 25°C (EN 1426) or softening point (EN 1427).

In the case of excessive bitumen ageing, the procedure for controlled product disposal should be initiated (FPC procedure compliant with EN 13108-21).

### Long-term storage at low temperature (over 10 days)

If it is required to store paving-grade bitumen 50/70 for a much longer period than 10 days, it is recommended to reduce the bitumen temperature and reheat before reuse. If a long storage period is envisaged without bituminous mixture production, bitumen storage at the ambient temperature is allowed. A precondition for such storage is fitting the silo with heating equipment having sufficient capacity to ensure subsequent bitumen heating without running the risk of local binder overheat during long-term heating.

## 4.2.4. Paving Grade Bitumen 70/100

### Intended use

Paving Grade Bitumen 70/100 can be used to a limited extent for asphalt concrete and SMA in wearing courses, on the assumption that the mix resistance to rutting is confirmed. It can also be used for the production of asphalt emulsions.

## Properties acc. to EN 12591:2009

**Table 4.9.** Properties of Paving Grade Bitumen 70/100

| Property                              | Test method           | Unit               | Requirement |
|---------------------------------------|-----------------------|--------------------|-------------|
| Penetration at 25°C                   | EN 1426               | 0.1 mm             | 70 – 100    |
| Softening Point R&B                   | EN 1427               | °C                 | 43 – 51     |
| Fraass Breaking Point                 | EN 12593              | °C                 | ≤ -10       |
| Flash point                           | EN ISO 2592           | °C                 | ≥ 230       |
| Solubility                            | EN 12592              | % (m/m)            | ≥ 99.0      |
| Change of mass after RTFOT (absolute) | EN 12607-1            | % (m/m)            | ≤ 0.8       |
| Retained penetration after RTFOT      | EN 12607-1<br>EN 1426 | %                  | ≥ 46        |
| Softening Point increase after RTFOT  | EN 12607-1<br>EN 1427 | °C                 | ≤ 9         |
| Kinematic viscosity at 135°C          | EN 12595              | mm <sup>2</sup> /s | ≥ 230       |
| Dynamic viscosity at 60°C             | EN 12596              | Pa*s               | ≥ 90        |

## Properties acc. to Superpave

Bitumen 70/100 properties acc to *Superpave* (tests conducted in 2009–2012).

- classification as per AASHTO MP 1: **PG 58-22**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (ungaded bitumen)  $T_{crit} = 63.4^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 63.6^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\ 000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 19.1^\circ\text{C}$
- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa  $T(S)_{60} = -16.9^\circ\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -16.2^\circ\text{C}$
  - stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 285$  MPa
- results and classification based on the MSCR (discussion in Chapter 7, Table 7.5)

| Temperature  | 58°C            | 64°C           |
|--|-----------------|----------------|
| $J_{nr}$ 0.1 kPa   | 1.965           | 4.070          |
| $J_{nr}$ 3.2 kPa   | 2.273           | 4.560          |
| $J_{nr}$ diff  | 15.7            | 12.0           |
| R 0.1 kPa  | 2.7             | 1.7            |
| R 3.2 kPa  | 0.5             | 0.0            |
| R diff   | 81              | 100            |
| Final classification of suitability for road traffic (at the test temperature) acc. to the most recent PG classification | S<br>(Standard) | not classified |

## Process temperatures

| At laboratory   |           |
|---|-----------|
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press) | 130–135°C |
| At mixing plant   |           |
| Bitumen pumping temperature   | > 130°C   |
| Temperature of bitumen for bituminous mixture production                                | 150–160°C |
| At site   |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)              | 140°C     |

## Viscosity dependence on temperature

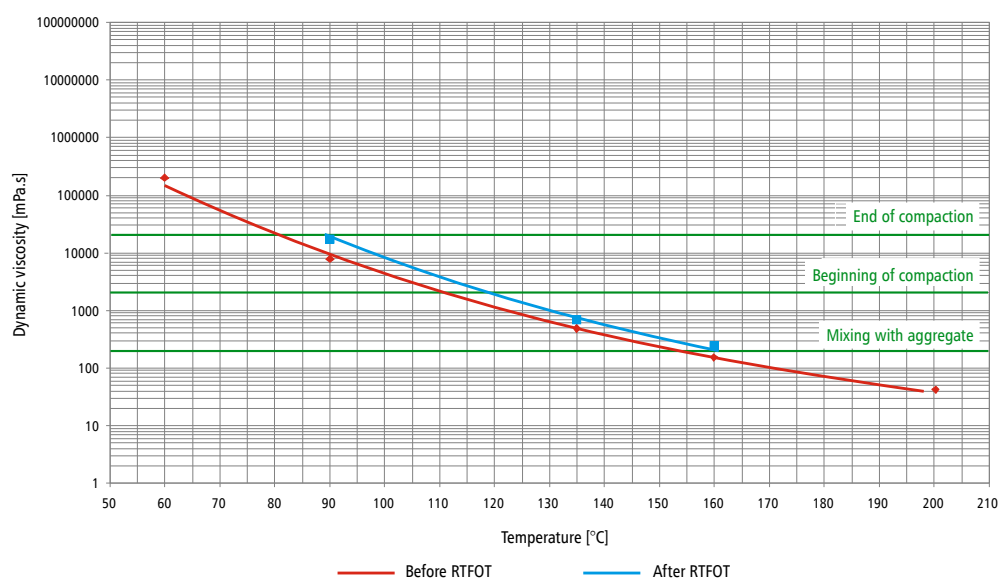


Figure 4.5. Viscosity dependence on temperature for Paving Grade Bitumen 70/100

Table 4.10. Example results of viscosity tests on bitumen 70/100 manufactured in 2012–2013. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature   | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|------|--------------------|-----------------------------------|
| dynamic        | vacuum capillary             | EN 12596                 | —                    | Pa*s | 60°C               | 149                               |
|                | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle No 21        | Pa*s | 90°C               | 7.47                              |
|                |                              |                          |                      |      | 135°C              | 0.41                              |
|                |                              |                          | spindle No 27        | Pa*s | 90°C after RTFOT   | 17.53                             |
|                |                              |                          |                      |      | 135°C after RTFOT  | 0.64                              |
|                | kinematic                    | BS/IP/RF viscometer      | EN 12595             | —    | mm <sup>2</sup> /s | 160°C after RTFOT                 |
| 135°C          |                              |                          |                      |      |                    | 372                               |

## Storage

### Short-term storage at high temperature (up to 10 days)

- recommended bitumen storage temperature:  $\leq 180^{\circ}\text{C}$

### Long-term storage at high temperature (over 10 days)

Bitumen storage at high temperatures over prolonged periods of time should be avoided. If it is necessary to store Paving Grade Bitumen 70/100 in the silo at a high temperature (up to  $180^{\circ}\text{C}$ ) for over 10 days, it is recommended to inspect the binder ageing rate before bitumen application for the production of bituminous mixtures. The following should be checked: penetration at  $25^{\circ}\text{C}$  (EN 1426) or Softening Point (EN 1427).

In the case of excessive bitumen ageing, the procedure for controlled product disposal should be initiated (FPC procedure compliant with EN 13108-21).

### Long-term storage at low temperature (over 10 days)

If it is required to store Paving Grade Bitumen 70/100 for a much longer period than 10 days, it is recommended to reduce the bitumen temperature and reheat before reuse. If a long storage period is envisaged without bituminous mixture production, bitumen storage at the ambient temperature is allowed. A precondition for such storage is fitting the silo with heating equipment having sufficient capacity to ensure subsequent bitumen heating without running the risk of local binder overheat during long-term heating.

## 4.2.5. Paving Grade Bitumen 100/150

### Intended use

Paving Grade Bitumen 100/150 is a binder intended primarily for the production of asphalt emulsion for various applications.

### Properties acc. to EN 12591:2009

**Table 4.11.** Properties of Paving Grade Bitumen 100/150

| Property                                     | Test method           | Unit                     | Requirement |
|--|-----------------------|--------------------------|-------------|
| Penetration at $25^{\circ}\text{C}$          | EN 1426               | 0.1 mm                   | 100 – 150   |
| Softening Point R&B                          | EN 1427               | $^{\circ}\text{C}$       | 39 – 47     |
| Fraass Breaking point                        | EN 12593              | $^{\circ}\text{C}$       | $\leq -12$  |
| Flash point                                  | EN ISO 2592           | $^{\circ}\text{C}$       | $\geq 230$  |
| Solubility                                   | EN 12592              | % (m/m)                  | $\geq 99.0$ |
| Change of mass after RTFOT (absolute)        | EN 12607-1            | % (m/m)                  | $\leq 0.8$  |
| Retained penetration after RTFOT             | EN 12607-1<br>EN 1426 | %                        | $\geq 43$   |
| Softening Point increase after RTFOT         | EN 12607-1<br>EN 1427 | $^{\circ}\text{C}$       | $\leq 10$   |
| Kinematic viscosity at $135^{\circ}\text{C}$ | EN 12595              | $\text{mm}^2/\text{s}$   | $\geq 175$  |
| Dynamic viscosity at $60^{\circ}\text{C}$    | EN 12596              | $\text{Pa}\cdot\text{s}$ | $\geq 55$   |

## Viscosity dependence on temperature

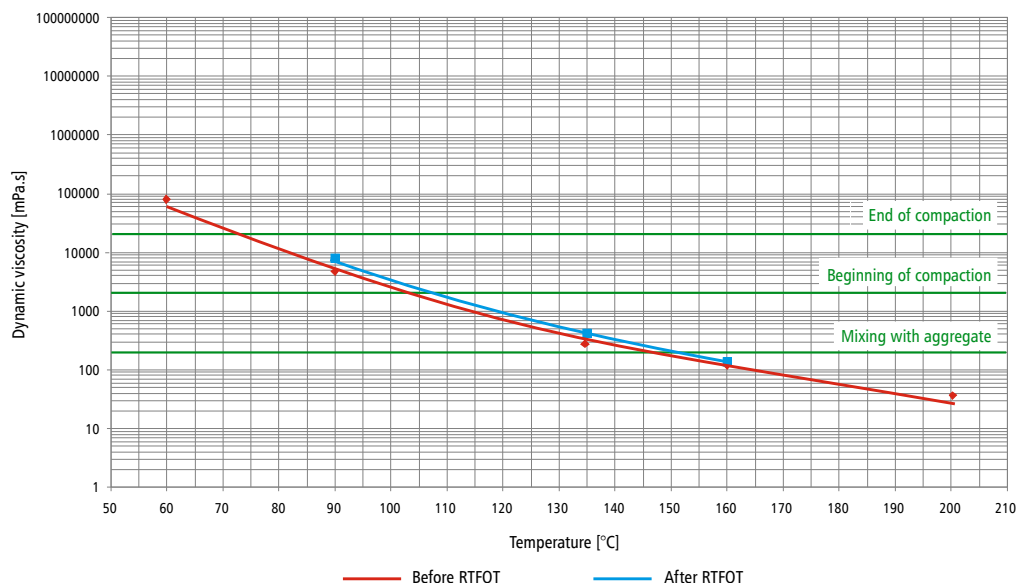


Figure 4.6. Viscosity dependence on temperature for Paving Grade Bitumen 100/150

Table 4.12. Example results of viscosity tests on bitumen 100/150 manufactured in 2012–2013. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type       | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature     | Example test result for viscosity |
|----------------------|------------------------------|--------------------------|----------------------|------|----------------------|-----------------------------------|
| dynamic              | vacuum capillary             | EN 12596                 | —                    | Pa*s | 60°C                 | 86.5                              |
|                      | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle<br>No 18, 21 | Pa*s | 90°C                 | 4.03                              |
|                      |                              |                          |                      |      | 135°C                | 0.26                              |
|                      |                              |                          | spindle<br>No 21     | Pa*s | 160°C                | 0.11                              |
|                      |                              |                          |                      |      | 90°C<br>after RTFOT  | 6.90                              |
|                      | kinematic                    | BS/IP/RF<br>viscometer   | EN 12595             | —    | mm <sup>2</sup> /s   | 135°C                             |
| 135°C<br>after RTFOT |                              |                          |                      |      |                      | 0.37                              |
|                      |                              |                          |                      |      | 160°C<br>after RTFOT | 0.13                              |

## Storage

### Short-term storage at high temperature (up to 10 days)

- recommended bitumen storage temperature:  $\leq 180^{\circ}\text{C}$

### Long-term storage at high temperature (over 10 days)

Bitumen storage at high temperatures over prolonged periods of time should be avoided. If it is necessary to store Paving Grade Bitumen 100/150 in the silo at a high temperature (up to 180°C) for over 10 days, it is recommended to inspect the binder ageing rate before bitumen application for the production of bituminous mixtures. The following should be checked: penetration at 25°C (EN 1426) or softening point (EN 1427).



### Long-term storage at low temperature (over 10 days)

If it is required to store paving-grade bitumen 100/150 for a much longer period than 10 days, it is recommended to reduce the bitumen temperature and reheat before reuse. If a long storage period is envisaged without bituminous mixture production, bitumen storage at the ambient temperature is allowed. A precondition for such storage is fitting the silo with heating equipment having sufficient capacity to ensure subsequent bitumen heating without running the risk of local binder overheat during long-term heating.

### 4.2.6. Paving Grade Bitumen 160/220

#### Intended use

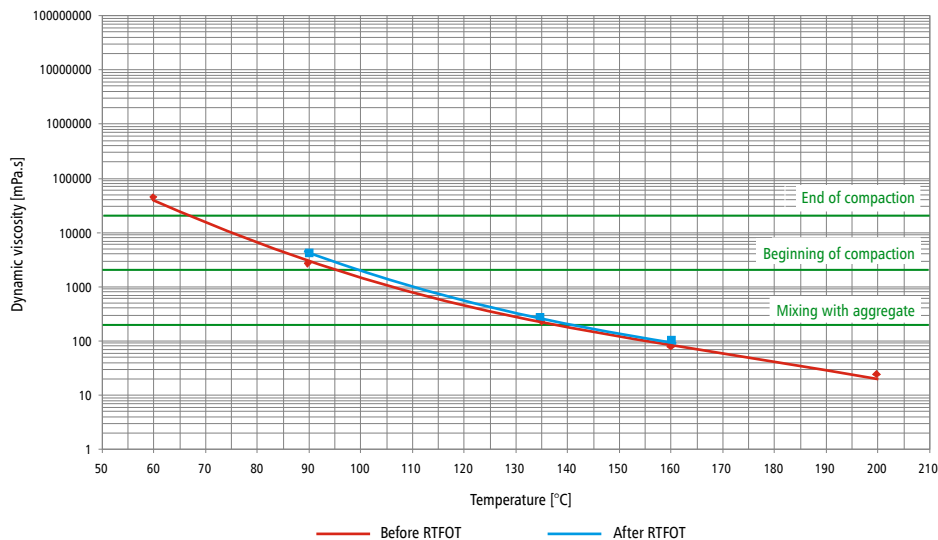
Paving Grade Bitumen 160/220 is a binder intended primarily for the production of bituminous emulsion for various applications.

#### Properties as per EN 12591:2009

**Table 4.13.** Properties of paving-grade bitumen 160/220

| Property                              | Test method           | Unit               | Requirement |
|---------------------------------------|-----------------------|--------------------|-------------|
| Penetration at 25°C                   | EN 1426               | 0.1 mm             | 160 – 220   |
| Softening Point R&B                   | EN 1427               | °C                 | 35 – 43     |
| Fraass Breaking Point                 | EN 12593              | °C                 | ≤ -15       |
| Flash point                           | EN ISO 2592           | °C                 | ≥ 220       |
| Solubility                            | EN 12592              | % (m/m)            | ≥ 99.0      |
| Change of mass after RTFOT (absolute) | EN 12607-1            | % (m/m)            | ≤ 1.0       |
| Retained penetration after RTFOT      | EN 12607-1<br>EN 1426 | %                  | ≥ 37        |
| Softening Point increase after RTFOT  | EN 12607-1<br>EN 1427 | °C                 | ≤ 11        |
| Kinematic viscosity at 135°C          | EN 12595              | mm <sup>2</sup> /s | ≥ 135       |
| Dynamic viscosity at 60°C             | EN 12596              | Pa*s               | ≥ 30        |

#### Viscosity dependence on temperature



**Figure 4.7.** Viscosity dependence on temperature for Paving Grade Bitumen 160/220

**Table 4.14.** Example results of viscosity tests on bitumen 160/220 manufactured in 2012–2013. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit               | Test temperature    | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|--------------------|---------------------|-----------------------------------|
| dynamic        | vacuum capillary             | EN 12596                 | —                    | Pa*s               | 60°C                | 70.0                              |
|                | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle<br>No 21     | Pa*s               | 90°C                | 2.66                              |
|                |                              |                          |                      |                    | 135°C               | 0.20                              |
|                |                              |                          |                      |                    | 160°C               | 0.08                              |
|                |                              |                          |                      |                    | 90°C<br>after RTFOT | 4.35                              |
|                |                              |                          | 135°C<br>after RTFOT | 0.27               |                     |                                   |
|                |                              |                          | 160°C<br>after RTFOT | 0.11               |                     |                                   |
| kinematic      | BS/IP/RF viscometer          | EN 12595                 | —                    | mm <sup>2</sup> /s | 135°C               | 233                               |

## Storage

### Short-term storage at high temperature (up to 10 days)

- recommended bitumen storage temperature:  $\leq 180^{\circ}\text{C}$

### Long-term storage at high temperature (over 10 days)

Bitumen storage at high temperatures over prolonged periods of time should be avoided. If it is necessary to store Paving Grade Bitumen 160/220 in the silo at a high temperature (up to 180°C) for over 10 days, it is recommended to inspect the binder ageing rate before bitumen application for the production of bituminous mixtures. The following should be checked: penetration at 25°C (EN 1426) or softening point (EN 1427).

### Long-term storage at low temperature (over 10 days)

If it is required to store Paving Grade Bitumen 160/220 for a much longer period than 10 days, it is recommended to reduce the bitumen temperature and reheat before reuse. If a long storage period is envisaged without bituminous mixture production, bitumen storage at the ambient temperature is allowed. A precondition for such storage is fitting the silo with heating equipment having sufficient capacity to ensure subsequent bitumen heating without running the risk of local binder overheat during long-term heating.

## Chapter 5

# POLYMER MODIFIED BITUMENS ORBITON ACC. TO EN 14023

### 5.1. General description

ORLEN Asphalt has been manufacturing ORBITON modified bitumens acc. to European Standard EN 14023 since early 2009.

Polymer Modified Bitumens represent a group of road paving binders designed specifically to counteract the most frequent road problems, such as deformations on roads carrying heavy and very heavy traffic and low-temperature cracks in winter, as well as to increase the pavement's fatigue resistance.

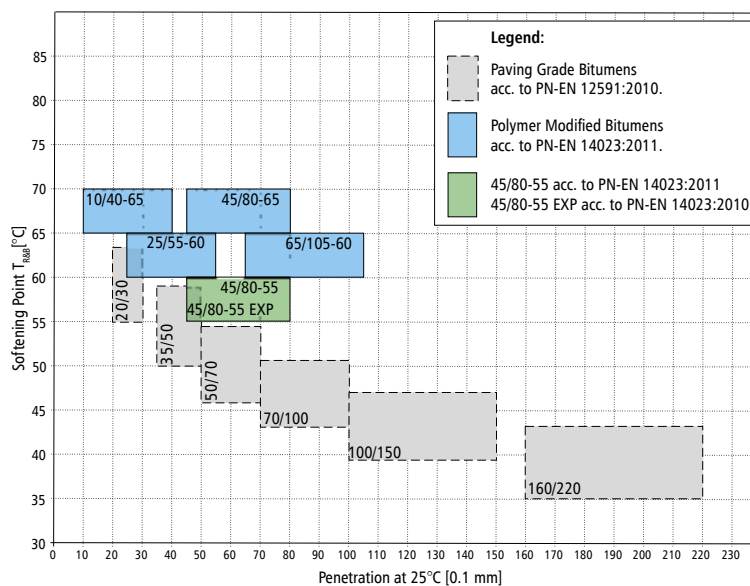
This Handbook discusses ORBITON modified bitumens manufactured acc. to PN-EN 14023:2011 and designed for use in road construction in Poland. ORLEN Asphalt also manufactures ORBITON modified bitumens acc. to the requirements indicated in the relevant National Annexes to EN 14023:2010 (e.g. for Romania, Lithuania, Latvia, Czech Republic, Slovakia, Germany, Hungary, etc.). The types of bitumens we manufacture are shown in Table 5.1.

**Table 5.1.** Types of modified bitumens manufactured by ORLEN Asphalt

| Types of ORBITON modified bitumens acc. to NA for Poland  | Types of ORBITON modified bitumens acc. to NA for EU countries, designations of product groups |
|---|--|
| 10/40-65<br>25/55-60<br>45/80-55<br>45/80-65<br>65/105-60 | 25/55-55 EXP *)<br>25/55-60 EXP *)<br>25/55-65 EXP *)<br>45/80-55 EXP                          |
| *) bitumen not discussed in this Handbook                 |  |

This Handbook discusses all types of ORBITON modified bitumens manufactured acc. to the Polish National Annex to PN-EN 14023:2011 (revision 2014) and ORBITON 45/80-55 EXP for Lithuanian, Latvian and Estonian markets.

Asphalt pavements where modified bitumen is used are more durable as compared to paving-grade bitumen pavements. Key differences between paving-grade bitumens and modified bitumens for the two primary binder parameters, namely penetration and Softening Point R&B, are schematically presented on Figure 5.1.



**Figure 5.1.** Schematic comparison of paving-grade bitumens and modified bitumens discussed in this Handbook in terms of penetration at 25°C and softening point  $T_{R\&B}$

## 5.2. Intended use

ORBITON modified bitumens represent a group of modern binders intended for use in pavements carrying heavy and very heavy traffic. Well-designed bituminous mixtures using those bitumens demonstrate better properties as compared with their counterparts having similar hardness (paving-grade and multigrade paving bitumen).

The range of applications for modified bitumens is very broad, both in terms of bituminous mixture type and road traffic category. The only limitation in the application of modified bitumens is their cost efficiency.

## 5.3. Properties

Subsequent sections of this chapter discuss all properties of modified bitumens acc. to EN 14023, including details based on the American Superpave (Performance Grade system). This chapter also provides approximate process temperatures for bitumen application in bituminous mixtures, viscosity data and viscosity dependence on temperature.

The MSCR-based classification is provided in Chapter 7.

### 5.3.1. ORBITON PMB 10/40-65

#### Intended use

**Modified bitumen ORBITON 10/40-65** is the hardest modified bitumen from the range currently manufactured by ORLEN Asphalt. Its very high softening point renders it suitable for high-rigidity courses – base and binder course made of AC WMS mix<sup>1</sup>. It can also be used for conventional asphalt concrete mixes. Resistance to rutting test results for mixtures with this type of bitumen demonstrate that it is recommended for pavements carrying slow and heavy traffic, such as hardstanding, slow traffic lanes, junctions. This type of bitumen is not recommended for wearing courses.

1) WMS designation is used in Poland to denote high modulus asphalt concrete (EME in France, HMB in the UK).

## Properties acc. to PN-EN 14023:2011

**Table 5.2.** Properties of ORBITON 10/40-65 modified bitumen acc. to PN-EN 14023:2011

| Property  | Test method                    | Unit              | Requirement      |
|---|--------------------------------|-------------------|------------------|
| Penetration at 25°C   | EN 1426                        | 0.1 mm            | 10 ÷ 40          |
| Softening Point R&B   | EN 1427                        | °C                | ≥ 65             |
| Elastic recovery at 25°C  | EN 13398                       | %                 | ≥ 60             |
| Fraass Breaking Point   | EN 12593                       | °C                | ≤ -5             |
| Flash Point   | EN ISO 2592                    | °C                | ≥ 235            |
| Force ductility (low tension rate)                                    | EN 13589<br>EN 13703           | J/cm <sup>2</sup> | ≥ 2 at 10°C      |
| Change of mass after RTFOT  | EN 12607-1                     | %                 | ≤ 0.5            |
| Increase in softening point after RTFOT                               | EN 1427                        | °C                | ≤ 8              |
| Retained penetration after RTFOT                                      | EN 1426                        | %                 | ≥ 60             |
| Elastic recovery at 25°C after RTFOT                                  | EN 12607-1,<br>EN 13398        | %                 | ≥ 50             |
| Storage stability:<br>Difference in softening point                   | EN 13399,<br>EN 1427           | °C                | ≤ 5              |
| Storage stability:<br>Difference in penetration at 25°C               | EN 13399,<br>EN 1427           | 0.1 mm            | NR <sup>b</sup>  |
| Plasticity range  | EN 14023<br>Subsection 5.2.8.4 | °C                | NR <sup>b</sup>  |
| Drop in softening point after RTFOT                                   | EN 12607-1,<br>EN 1427         | °C                | TBR <sup>a</sup> |
| <sup>a</sup> TBR – To Be Reported<br><sup>b</sup> NR – No Requirement |                                |                   |                  |

## Properties acc. to Superpave

ORBITON 10/40-65 properties acc. to *Superpave* (tests conducted in 2009-2012).

- classification acc. to AASHTO MP 1: **PG 82-16**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (unaged bitumen)  $T_{crit} = 88.5^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 83.8^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\ 000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 19.5^\circ\text{C}$
- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa  $T(S)_{60} = -17.2^\circ\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -8.6^\circ\text{C}$
  - stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 271.5$  MPa

- results and classification based on the MSCR (discussion in Chapter 7)

|   |                |
|---|----------------|
| <b>Temperature</b>  | <b>64°C</b>    |
| J <sub>nr</sub> 0.1 kPa   | 0.106          |
| J <sub>nr</sub> 3.2 kPa   | 0.140          |
| J <sub>nr</sub> diff  | 31.9           |
| R 0.1 kPa   | 68.6           |
| R 3.2 kPa   | 62.5           |
| R diff  | 9              |
| Final classification of suitability for road traffic (at the test temperature) as per the most recent PG classification | E<br>(Extreme) |

## Process temperatures

|   |             |
|---|-------------|
| <b>At laboratory:</b>   |             |
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press)   | 150 ± 155°C |
| <b>At mixing plant:</b>   |             |
| Bitumen pumping temperature   | > 150°C     |
| Temperature of bitumen for bituminous mixture production  | 180–190°C   |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 8 h)   | < 230°C     |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 4 h)   | < 240°C     |
| <b>Note:</b> it is recommended for MA production to use additives to reduce the process temperature (mixing with aggregate and placement) so that the mastic asphalt production takes place at a temperature below 200°C. |             |
| <b>At site</b>  |             |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)  | 160°C       |

## Viscosity dependence on temperature

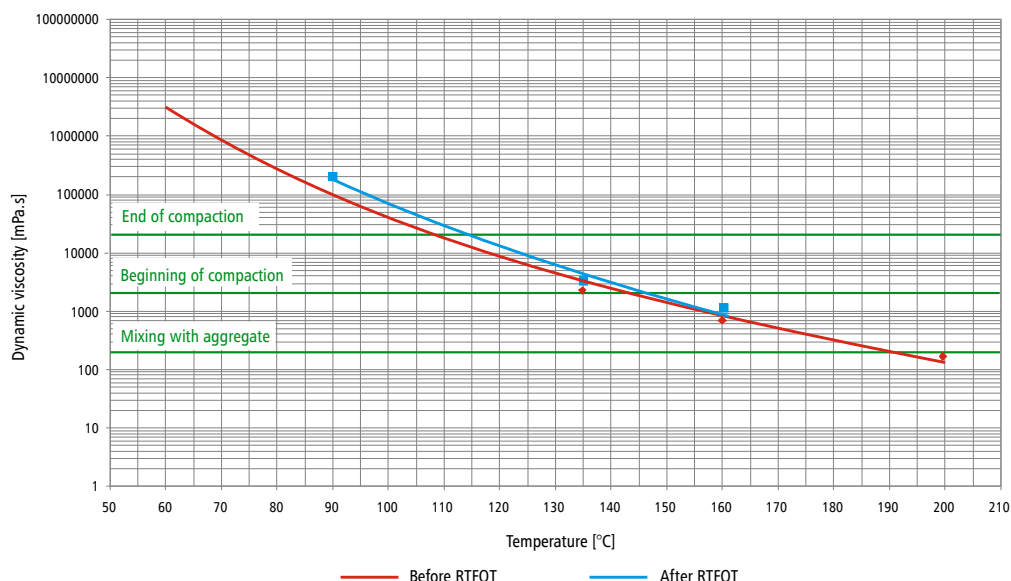


Figure 5.2. Viscosity dependence on temperature for modified bitumen ORBITON 10/40-65

**Table 5.3.** Example results of viscosity tests on bitumen ORBITON 10/40-65. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature  | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|------|-------------------|-----------------------------------|
| dynamic        | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle No 21        | Pa*s | 90°C              | 130.00                            |
|                |                              |                          |                      |      | 135°C             | 2.52                              |
|                |                              |                          |                      |      | 160°C             | 0.68                              |
|                |                              |                          | spindle No 27        | Pa*s | 90°C after RTFOT  | 202.00                            |
|                |                              |                          |                      |      | 135°C after RTFOT | 3.76                              |
|                |                              |                          |                      |      | 160°C after RTFOT | 0.98                              |

### Microstructure

- polymer dispersion code acc. to EN 13632: B/H/S/r or B/H/S/o

### Storage

#### Short-term storage at high temperature (up to 7 days)

- recommended bitumen storage temperature: 160–180°C
- guaranteed period of bitumen service life for asphalt production: 7 days

It is recommended to conduct basic inspection tests for modified bitumen properties after five days in order to make sure that the product has not lost its properties due to the stability loss of the bitumen-polymer combination caused by component separation. The test should be conducted after 5 days of storage and every subsequent 2 days (7th day, 9th day, etc.) or in other time intervals, depending on the actual needs:

- penetration at 25°C, EN 1426
- Softening Point R&B, EN 1427
- elastic recovery at 25°C, EN 13398

If the mixing plant is fitted with tanks with agitators, bitumen should be periodically mixed in the tank. Circulation can be used for that purpose.

#### Long-term storage at high temperature (over 7 days)

It is not recommended to store modified bitumen for more than 7 days. Where such storage is necessary, we recommend periodic testing of binder properties, e.g. every 2 days (scope of tests indicated above). It is desirable to mix bitumen in the tank for at least 6 hours per day. The recommended storage temperature is 150–160°C.

#### Long-term storage at low temperature (over 7 days)

Due to its significant hardness, it is not recommended to store this binder if it is cooled down to ambient temperature (e.g. over winter) since it is very difficult to melt it.

### 5.3.2. ORBITON PMB 25/55-60

#### Intended use

Modified bitumen ORBITON 25/55-60 is one of the most popular modified bitumen types. It is used for asphalt concrete base and binder courses and for high modulus asphalt concrete AC WMS. It can also be used for SMA wearing courses at sections carrying slow and heavy traffic and for mastic asphalt mixes.

#### Properties acc. to PN-EN 14023:2011

**Table 5.4.** Properties of ORBITON 25/55-60 modified bitumen as per PN-EN 14023:2011

| Property  | Test method                    | Unit              | Requirement      |
|---|--------------------------------|-------------------|------------------|
| Penetration at 25°C   | EN 1426                        | 0.1 mm            | 25 ÷ 55          |
| Softening Point R&B   | EN 1427                        | °C                | ≥ 60             |
| Elastic recovery at 25°C  | EN 13398                       | %                 | ≥ 60             |
| Fraass Breaking Point   | EN 12593                       | °C                | ≤ -10            |
| Flash Point   | EN ISO 2592                    | °C                | ≥ 235            |
| Force ductility<br>(low tension rate)                                 | EN 13589<br>EN 13703           | J/cm <sup>2</sup> | ≥ 2 at 10°C      |
| Change of mass after RTFOT  | EN 12607-1                     | %                 | ≤ 0.5            |
| Increase in softening point after RTFOT                               | EN 1427                        | °C                | ≤ 8              |
| Retained penetration after RTFOT                                      | EN 1426                        | %                 | ≥ 60             |
| Elastic recovery at 25°C after RTFOT                                  | EN 12607-1,<br>EN 13398        | %                 | ≥ 50             |
| Storage stability:<br>Difference in softening point                   | EN 13399,<br>EN 1427           | °C                | ≤ 5              |
| Storage stability:<br>Difference in penetration at 25°C               | EN 13399,<br>EN 1427           | 0.1 mm            | NR <sup>b</sup>  |
| Plasticity range  | EN 14023<br>Subsection 5.2.8.4 | °C                | NR <sup>b</sup>  |
| Drop in softening point after RTFOT                                   | EN 12607-1,<br>EN 1427         | °C                | TBR <sup>a</sup> |
| <sup>a</sup> TBR – To Be Reported<br><sup>b</sup> NR – No Requirement |                                |                   |                  |

#### Properties acc. to Superpave

ORBITON 25/55-60 properties acc. to *Superpave* (tests conducted in 2009-2012).

- classification as per AASHTO MP 1: **PG 76-22**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (fresh bitumen)  $T_{crit} = 83.1^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 80.5^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\ 000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 22.0^\circ\text{C}$



- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa
  - temperature at  $m(60) \geq 0.3$
  - stiffness at  $-16^\circ\text{C}$

$$T(S)_{60} = -16.9^\circ\text{C}$$

$$T(m)_{60} = -13.8^\circ\text{C}$$

$$S(T)_{-16} = 278 \text{ MPa}$$

- results and classification based on the MSCR (discussion in Chapter 7)

| Temperatura   | 58°C        | 64°C        | 70°C        |
|---|-------------|-------------|-------------|
| J <sub>nr</sub> 0.1 kPa   | 0.070       | 0.180       | 0.360       |
| J <sub>nr</sub> 3.2 kPa   | 0.070       | 0.208       | 0.440       |
| J <sub>nr</sub> diff  | 0           | 15.5        | 22.2        |
| R 0.1 kPa   | 68.3        | 63.8        | 58.2        |
| R 3.2 kPa   | 68.3        | 59.8        | 51.1        |
| R diff  | 0           | 6           | 12          |
| Final classification of suitability for road traffic (at the test temperature) as per the most recent PG classification | E (Extreme) | E (Extreme) | E (Extreme) |

### Process temperatures

| At laboratory:  |           |
|---|-----------|
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press)   | 145–150°C |
| At mixing plant:  |           |
| Bitumen pumping temperature   | > 150°C   |
| Temperature of bitumen for bituminous mixture production  | 175–185°C |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 8 h)   | < 230°C   |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 4 h)   | < 240°C   |
| <b>Note:</b> it is recommended for MA production to use additives to reduce the process temperature (mixing with aggregate and placement) so that the mastic asphalt production takes place at a temperature below 200°C. |           |
| At site   |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)  | 155°C     |

### Viscosity dependence on temperature

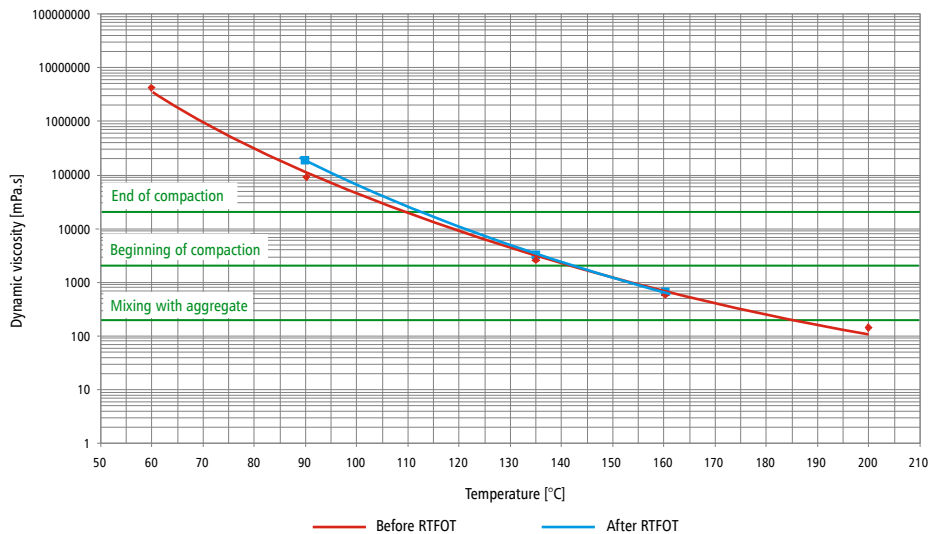


Figure 5.3. Viscosity dependence on temperature for modified bitumen ORBITON 25/55-60

**Table 5.5.** Example results of viscosity tests on bitumen ORBITON 25/55-60. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature  | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|------|-------------------|-----------------------------------|
| dynamic        | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle No 27        | Pa*s | 90°C              | 87.50                             |
|                |                              |                          |                      |      | 135°C             | 1.97                              |
|                |                              |                          |                      |      | 160°C             | 0.54                              |
|                |                              |                          | spindle No 27        | Pa*s | 90°C after RTFOT  | 182.00                            |
|                |                              |                          |                      |      | 135°C after RTFOT | 2.75                              |
|                |                              |                          |                      |      | 160°C after RTFOT | 0.68                              |

### Microstructure

- polymer dispersion code acc. to EN 13632: B/H/S/r or B/H/S/o

### Storage

#### Short-term storage at high temperature (up to 7 days)

- recommended bitumen storage temperature: 160–180°C
- guaranteed period of bitumen service life for asphalt production: 7 days

It is recommended to conduct basic inspection tests for modified bitumen properties after 5 days in order to make sure that the product has not lost its properties due to the stability loss of the bitumen-polymer combination caused by component separation. The test should be conducted after 5 days of storage and every subsequent 2 days (7th day, 9th day, etc.) or in other time intervals, depending on the actual needs:

- penetration at 25°C, EN 1426
- Softening Point R&B, EN 1427
- elastic recovery at 25°C, EN 13398

If the mixing plant is fitted with tanks with agitators, bitumen should be periodically mixed in the tank. Circulation can be used for that purpose.

#### Long-term storage at high temperature (over 7 days)

It is not recommended to store modified bitumen for more than 7 days. Where such storage is necessary, we recommend periodic testing of binder properties, e.g. every 2 days (scope of tests indicated above). It is desirable to mix bitumen in the tank for at least 6 hours per day. The recommended storage temperature is 150–160°C.

#### Long-term storage at low temperature (over 7 days)

Due to its significant hardness, it is not recommended to store this binder if it is cooled down to ambient temperature (e.g. over winter) since it is very difficult to melt it.

### 5.3.3. ORBITON PMB 45/80-55

#### Intended use

Modified bitumen ORBITON 45/80-55 is one of the most popular modified bitumen types used in Poland. It is intended for use in all bituminous mixtures for wearing courses (AC, SMA).

#### Properties acc. to PN-EN 14023:2011

**Table 5.6.** Properties of ORBITON 45/80-55 modified bitumen acc. to PN-EN 14023:2011

| Property  | Test method                    | Unit              | Requirement      |
|---|--------------------------------|-------------------|------------------|
| Penetration at 25°C                                     | EN 1426                        | 0.1 mm            | 45 – 80          |
| Softening Point R&B                                     | EN 1427                        | °C                | ≥ 55             |
| Elastic recovery at 25°C                                | EN 13398                       | %                 | ≥ 70             |
| Fraass Breaking Point                                   | EN 12593                       | °C                | ≤ -15            |
| Flash Point   | EN ISO 2592                    | °C                | ≥ 235            |
| Force ductility<br>(low tension rate)                   | EN 13589<br>EN 13703           | J/cm <sup>2</sup> | ≥ 3 at 5°C       |
| Change of mass after RTFOT                              | EN 12607-1                     | %                 | ≤ 0.5            |
| Increase in softening point after RTFOT                 | EN 1427                        | °C                | ≤ 8              |
| Retained penetration after RTFOT                        | EN 1426                        | %                 | ≥ 60             |
| Elastic recovery at 25°C after RTFOT                    | EN 12607-1,<br>EN 13398        | %                 | ≥ 50             |
| Storage stability:<br>Difference in softening point     | EN 13399,<br>EN 1427           | °C                | ≤ 5              |
| Storage stability:<br>Difference in penetration at 25°C | EN 13399,<br>EN 1427           | 0.1 mm            | NR <sup>b</sup>  |
| Plasticity range  | EN 14023<br>Subsection 5.2.8.4 | °C                | NR <sup>b</sup>  |
| Drop in softening point after RTFOT                     | EN 12607-1,<br>EN 1427         | °C                | TBR <sup>a</sup> |

<sup>a</sup> TBR – To Be Reported  
<sup>b</sup> NR – No Requirement

#### Properties acc. to Superpave

ORBITON 45/80-55 properties acc. to *Superpave* (tests conducted in 2009–2012).

- classification acc. to AASHTO MP 1: **PG 70-22**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (unaged bitumen)  $T_{crit} = 74.5^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 72.9^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\ 000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 17.7^\circ\text{C}$
- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa  $T(S)_{60} = -18.1^\circ\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -16.9^\circ\text{C}$
  - stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 242$  MPa

- results and classification based on the MSCR (discussion in Chapter 7)

| Temperatura   | 58°C        | 64°C        | 70°C      |
|---|-------------|-------------|-----------|
| J <sub>nr</sub> 0.1 kPa   | 0.170       | 0.406       | 0.943     |
| J <sub>nr</sub> 3.2 kPa   | 0.180       | 0.490       | 1.608     |
| J <sub>nr</sub> diff  | 6           | 20.8        | 70.5      |
| R 0.1 kPa   | 68.9        | 65.0        | 52.3      |
| R 3.2 kPa   | 67.5        | 59.5        | 34.6      |
| R diff  | 2           | 8           | 34        |
| Final classification of suitability for road traffic (at the test temperature) as per the most recent PG classification | E (Extreme) | E (Extreme) | H (Heavy) |

## Process temperatures

| At laboratory:  |           |
|---|-----------|
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press)   | 145–150°C |
| At mixing plant:  |           |
| Bitumen pumping temperature   | > 150°C   |
| Temperature of bitumen for bituminous mixture production  | 175–185°C |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 8 h)   | < 230°C   |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 4 h)   | < 240°C   |
| <b>Note:</b> it is recommended for MA production to use additives to reduce the process temperature (mixing with aggregate and placement) so that the mastic asphalt production takes place at a temperature below 200°C. |           |
| At site   |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)  | 155°C     |

## Viscosity dependence on temperature

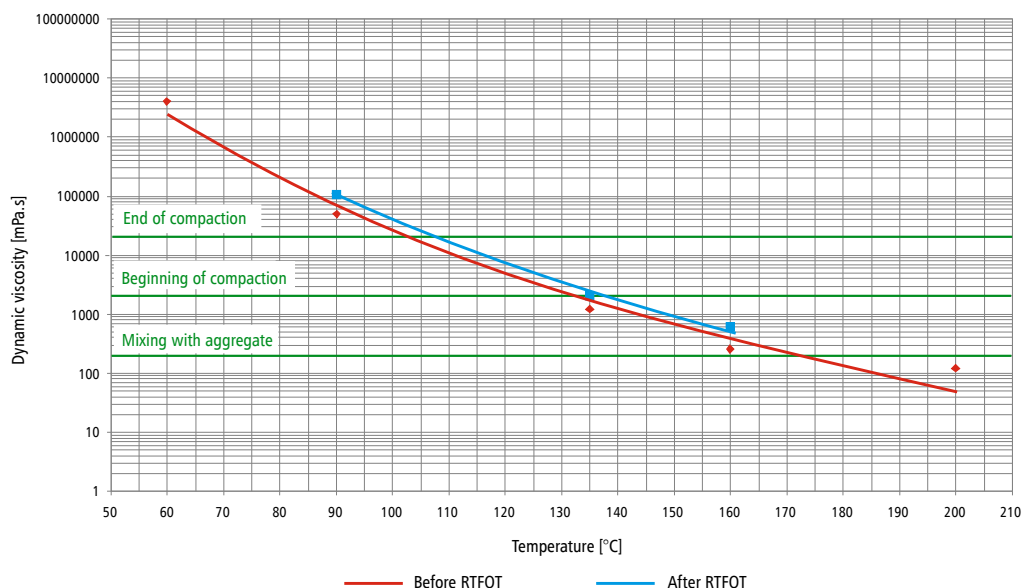


Figure 5.4. Viscosity dependence on temperature for modified bitumen ORBITON 45/80-55

**Table 5.7.** Example results of viscosity tests on bitumen ORBITON 45/80-55. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature  | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|------|-------------------|-----------------------------------|
| dynamic        | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle No 27        | Pa*s | 90°C              | 44.33                             |
|                |                              |                          |                      |      | 135°C             | 1.12                              |
|                |                              |                          |                      |      | 160°C             | 0.22                              |
|                |                              |                          | spindle No 27        | Pa*s | 90°C after RTFOT  | 103.00                            |
|                |                              |                          |                      |      | 135°C after RTFOT | 1.93                              |
|                |                              |                          |                      |      | 160°C after RTFOT | 0.55                              |

### Microstructure

- polymer dispersion code acc. to EN 13632: B/H/S/r or B/H/S/o

### Storage

#### Short-term storage at high temperature (up to 7 days)

- recommended bitumen storage temperature: 160–180°C
- guaranteed period of bitumen service life for asphalt production: 7 days

It is recommended to conduct basic inspection tests for modified bitumen properties after 5 days in order to make sure that the product has not lost its properties due to the stability loss of the bitumen-polymer combination caused by component separation. The test should be conducted after 5 days of storage and every subsequent 2 days (7th day, 9th day, etc.) or in other time intervals, depending on the actual needs:

- penetration at 25°C, EN 1426
- Softening Point R&B, EN 1427
- elastic recovery at 25°C, EN 13398

If the mixing plant is fitted with tanks with agitators, bitumen should be periodically mixed in the tank. Circulation can be used for that purpose.

#### Long-term storage at high temperature (over 7 days)

It is not recommended to store modified bitumen for more than 7 days. Where such storage is necessary, we recommend periodic testing of binder properties, e.g. every 2 days (scope of tests indicated above). It is desirable to mix bitumen in the tank for at least 6 hours per day. The recommended storage temperature is 150–160°C.

#### Long-term storage at low temperature (over 7 days)

Due to its significant hardness, it is not recommended to store this binder if it is cooled down to ambient temperature (e.g. over winter) since it is very difficult to melt it.

### 5.3.4. ORBITON PMB 45/80-55 EXP

#### Intended use

Modified bitumen ORBITON 45/80-55 EXP is a binder with similar properties to ORBITON 45/80-55, however its breaking point was lower by 3°C (before 2014), present properties – see table 5.6). It is intended for use in all bituminous mixtures for wearing courses (AC, SMA).

#### Properties as per EN 14023:2010

**Table 5.8.** Properties of ORBITON 45/80-55 EXP modified bitumen acc. to PN-EN 14023:2010

| Property  | Test method                    | Unit              | Requirement      |
|---|--------------------------------|-------------------|------------------|
| Penetration at 25°C                                     | EN 1426                        | 0.1 mm            | 45 – 80          |
| Softening Point R&B                                     | EN 1427                        | °C                | ≥ 55             |
| Elastic recovery at 25°C                                | EN 13398                       | %                 | ≥ 50             |
| Fraass Breaking Point                                   | EN 12593                       | °C                | ≤ -15            |
| Flash Point   | EN ISO 2592                    | °C                | ≥ 235            |
| Force ductility<br>(low tension rate)                   | EN 13589<br>EN 13703           | J/cm <sup>2</sup> | ≥ 2 at 5°C       |
| Change of mass after RTFOT                              | EN 12607-1                     | %                 | ≤ 0.5            |
| Increase in softening point after RTFOT                 | EN 1427                        | °C                | ≤ 8              |
| Retained penetration after RTFOT                        | EN 1426                        | %                 | ≥ 60             |
| Elastic recovery at 25°C after RTFOT                    | EN 12607-1,<br>EN 13398        | %                 | ≥ 50             |
| Storage stability:<br>Difference in softening point     | EN 13399,<br>EN 1427           | °C                | ≤ 5              |
| Storage stability:<br>Difference in penetration at 25°C | EN 13399,<br>EN 1427           | 0.1 mm            | ≤ 9              |
| Plasticity range  | EN 14023<br>Subsection 5.2.8.4 | °C                | TBR <sup>a</sup> |
| Drop in softening point after RTFOT                     | EN 12607-1,<br>EN 1427         | °C                | ≤ 2              |
| a) TBR – To Be Reported<br>b) NR – No Requirement       |                                |                   |                  |

#### Properties acc. to Superpave

ORBITON 45/80-55 EXP properties acc. to *Superpave* (tests conducted in 2013).

- classification acc. to AASHTO MP 1: **PG 70-22**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (unaged bitumen)
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)
  - $G^* \cdot \sin\delta = 5\,000$  kPa (bitumen after RTFOT and PAV)

$$T_{crit} = 74.7^{\circ}\text{C}$$

$$T_{crit} = 70.6^{\circ}\text{C}$$

$$T_{crit} = 14.4^{\circ}\text{C}$$

- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa
  - temperature at  $m(60) \geq 0.3$
  - stiffness at  $-16^\circ\text{C}$

$$T(S)_{60} = -17.9^\circ\text{C}$$

$$T(m)_{60} = -18.0^\circ\text{C}$$

$$S(T)_{-16} = 242.3 \text{ MPa}$$

- results and classification based on the MSCR (discussion in Chapter 7)

| Temperature   | 64°C              | 70°C         |
|---|-------------------|--------------|
| $J_{nr}$ 0.1 kPa  | 0.568             | 1.180        |
| $J_{nr}$ 3.2 kPa  | 0.682             | 1.729        |
| $J_{nr}$ diff   | 20.1              | 46.5         |
| R 0.1 kPa   | 62.6              | 57.2         |
| R 3.2 kPa   | 57.3              | 42.9         |
| R diff  | 10                | 30           |
| Final classification of suitability for road traffic (at the test temperature) as per the most recent PG classification | V<br>(Very Heavy) | H<br>(Heavy) |

### Process temperatures

| At laboratory:  |           |
|---|-----------|
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press)   | 145–150°C |
| At mixing plant:  |           |
| Bitumen pumping temperature   | > 150°C   |
| Temperature of bitumen for bituminous mixture production  | 175–185°C |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 8 h)   | < 230°C   |
| Mastic asphalt temperature in the mixer (asphalt storage time of up to 4 h)   | < 240°C   |
| <b>Note:</b> it is recommended for MA production to use additives to reduce the process temperature (mixing with aggregate and placement) so that the mastic asphalt production takes place at a temperature below 200°C. |           |
| At site   |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)  | 155°C     |

### Viscosity dependence on temperature

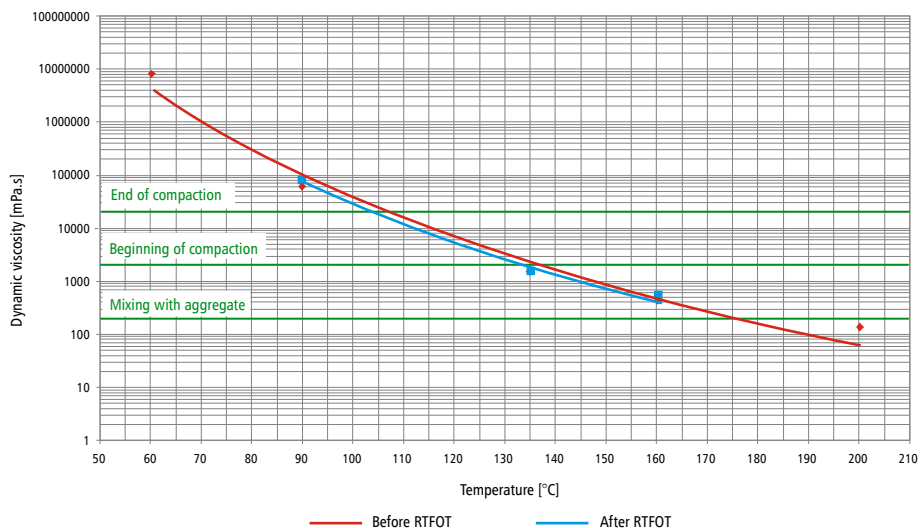


Figure 5.5. Viscosity dependence on temperature for modified bitumen ORBITON 45/80-55 EXP

**Table 5.9.** Example results of viscosity tests on bitumen ORBITON 45/80-55 EXP. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature  | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|------|-------------------|-----------------------------------|
| dynamic        | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle No 21        | Pa*s | 90°C              | 55.100                            |
|                |                              |                          |                      |      | 135°C             | 1.260                             |
|                |                              |                          |                      |      | 160°C             | 0.402                             |
|                |                              |                          | spindle No 21        | Pa*s | 90°C after RTFOT  | 70.917                            |
|                |                              |                          |                      |      | 135°C after RTFOT | 1.571                             |
|                |                              |                          |                      |      | 160°C after RTFOT | 0.453                             |

### Microstructure

- polymer dispersion code acc. to EN 13632: B/H/S/r or B/H/S/o

### Storage

#### Short-term storage at high temperature (up to 7 days)

- recommended bitumen storage temperature: 160–180°C
- guaranteed period of bitumen service life for MMA production: 7 days

It is recommended to conduct basic inspection tests for modified bitumen properties after 5 days in order to make sure that the product has not lost its properties due to the stability loss of the bitumen-polymer combination caused by component separation. The test should be conducted after 5 days of storage and every subsequent 2 days (7th day, 9th day, etc.) or in other time intervals, depending on the actual needs:

- penetration at 25°C, EN 1426
- Softening Point R&B, EN 1427
- elastic recovery at 25°C, EN 13398

If the mixing plant is fitted with tanks with agitators, bitumen should be periodically mixed in the tank. Circulation can be used for that purpose.

#### Long-term storage at high temperature (over 7 days)

It is not recommended to store modified bitumen for more than 7 days. Where such storage is necessary, we recommend periodic testing of binder properties, e.g. every 2 days (scope of tests indicated above). It is desirable to mix bitumen in the tank for at least 6 hours per day. The recommended storage temperature is 150–160°C.

#### Long-term storage at low temperature (over 7 days)

Due to its significant hardness, it is not recommended to store this binder if it is cooled down to ambient temperature (e.g. over winter) since it is very difficult to melt it.



### 5.3.5. ORBITON PMB 45/80-65

#### Intended use

Modified bitumen ORBITON 45/80-65 is intended for application in wearing courses and for special applications. It is marked by very high elasticity, high softening point and favourable low-temperature properties. Its high polymer content renders it troublesome for placement in adverse weather (quick stiffening, compaction problems). Its very high softening point and high modification rate render it suitable for application at locations where high tensile strength and fatigue resistance are necessary in combination with very good low-temperature properties. Modified bitumen ORBITON 45/80-65 is intended primarily for application in wearing courses, as well as for porous asphalt mixes.

#### Properties as per PN-EN 14023:2011

**Table 5.10.** Properties of ORBITON 45/80-65 modified bitumen acc. to PN-EN 14023:2011

| Property  | Test method                    | Unit              | Requirement      |
|---|--------------------------------|-------------------|------------------|
| Penetration at 25°C                                     | EN 1426                        | 0.1 mm            | 45 – 80          |
| Softening Point R&B                                     | EN 1427                        | °C                | ≥ 65             |
| Elastic recovery at 25°C                                | EN 13398                       | %                 | ≥ 80             |
| Fraass Breaking Point                                   | EN 12593                       | °C                | ≤ -15            |
| Flash Point   | EN ISO 2592                    | °C                | ≥ 235            |
| Force ductility (low tension rate)                      | EN 13589<br>EN 13703           | J/cm <sup>2</sup> | ≥ 2 at 10°C      |
| Change of mass after RTFOT                              | EN 12607-1                     | %                 | ≤ 0.5            |
| Increase in softening point after RTFOT                 | EN 1427                        | °C                | ≤ 8              |
| Retained penetration after RTFOT                        | EN 1426                        | %                 | ≥ 60             |
| Elastic recovery at 25°C after RTFOT                    | EN 12607-1,<br>EN 13398        | %                 | ≥ 60             |
| Storage stability:<br>Difference in softening point     | EN 13399,<br>EN 1427           | °C                | ≤ 5              |
| Storage stability:<br>Difference in penetration at 25°C | EN 13399,<br>EN 1427           | 0.1 mm            | NPD <sup>b</sup> |
| Plasticity range  | EN 14023<br>Subsection 5.2.8.4 | °C                | NR <sup>b</sup>  |
| Drop in softening point after RTFOT                     | EN 12607-1,<br>EN 1427         | °C                | TBR <sup>a</sup> |
| a) TBR – To Be Reported<br>b) NR – No Requirement       |                                |                   |                  |

#### Properties acc. to Superpave

ORBITON 45/80-65 properties acc. to *Superpave* (tests conducted in 2009–2012).

- classification as per AASHTO MP 1: **PG 76-22**
- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (unaged bitumen)  $T_{crit} = 83.2^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 77.7^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\ 000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 17.6^\circ\text{C}$

- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa  $T(S)_{60} = -18.3^{\circ}\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -14.3^{\circ}\text{C}$
  - stiffness at  $-16^{\circ}\text{C}$   $S(T)_{-16} = 235$  MPa
- results and classification based on the MSCR (discussion in Chapter 7)

| Temperature   | 64°C        | 70°C        |
|---|-------------|-------------|
| J <sub>nr</sub> 0.1 kPa   | 0.114       | 0.271       |
| J <sub>nr</sub> 3.2 kPa   | 0.135       | 0.377       |
| J <sub>nr</sub> diff  | 18.6        | 39.1        |
| R 0.1 kPa   | 84.4        | 75.4        |
| R 3.2 kPa   | 82.9        | 70.5        |
| R diff  | 2           | 6           |
| Final classification of suitability for road traffic (at the test temperature) as per the most recent PG classification | E (Extreme) | E (Extreme) |

### Process temperatures

|   |           |
|---|-----------|
| <b>At laboratory:</b>   |           |
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press) | 150–155°C |
| <b>At mixing plant:</b>   |           |
| Bitumen pumping temperature   | > 150°C   |
| Temperature of bitumen for bituminous mixture production                                | 175–185°C |
| <b>At site</b>  |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)              | 160°C     |

### Viscosity dependence on temperature

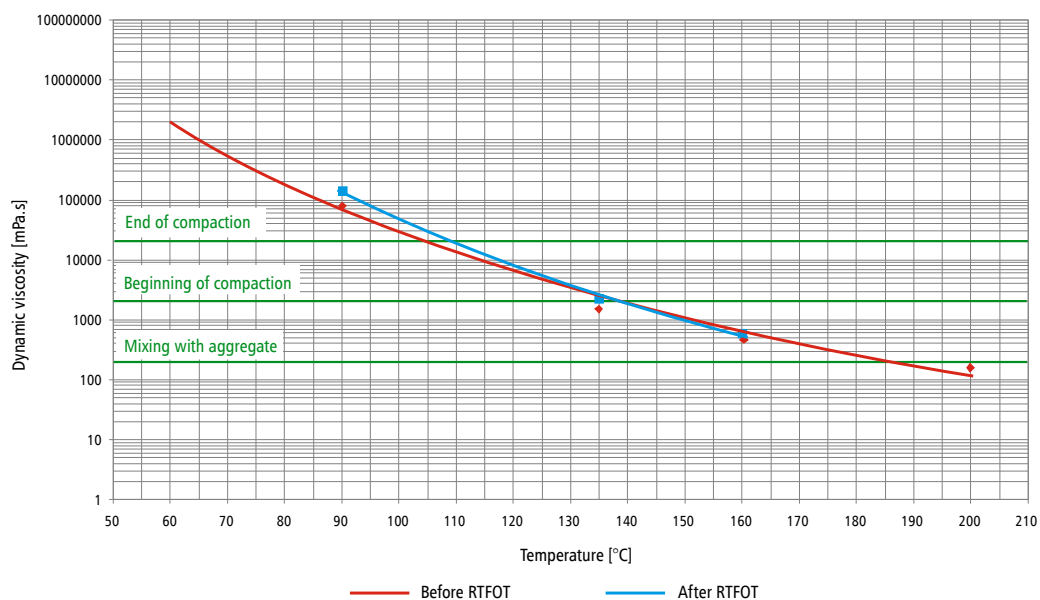


Figure 5.6. Viscosity dependence on temperature for modified bitumen ORBITON 45/80-65

**Table 5.11.** Example results of viscosity tests on bitumen ORBITON 45/80-65. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature  | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|------|-------------------|-----------------------------------|
| dynamic        | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle No 21        | Pa*s | 90°C              | 81.57                             |
|                |                              |                          |                      |      | 135°C             | 1.54                              |
|                |                              |                          |                      |      | 160°C             | 0.49                              |
|                |                              |                          | spindle No 27        | Pa*s | 90°C after RTFOT  | 128.00                            |
|                |                              |                          |                      |      | 135°C after RTFOT | 2.17                              |
|                |                              |                          |                      |      | 160°C after RTFOT | 0.58                              |

### Microstructure

- polymer dispersion code acc. to EN 13632: B/H/S/r or B/H/S/o

### Storage

#### Short-term storage at high temperature (up to 7 days)

- recommended bitumen storage temperature: 160–180°C
- guaranteed period of bitumen service life for asphalt production: 7 days

It is recommended to conduct basic inspection tests for modified bitumen properties after 5 days in order to make sure that the product has not lost its properties due to the stability loss of the bitumen-polymer combination caused by component separation. The test should be conducted after 5 days of storage and every subsequent 2 days (7th day, 9th day, etc.) or in other time intervals, depending on the actual needs:

- penetration at 25°C, EN 1426
- Softening Point R&B, EN 1427
- elastic recovery at 25°C, EN 13398

If the mixing plant is fitted with tanks with agitators, bitumen should be periodically mixed in the tank. Circulation can be used for that purpose.

#### Long-term storage at high temperature (over 7 days)

It is not recommended to store modified bitumen for more than 7 days. Where such storage is necessary, we recommend periodic testing of binder properties, e.g. every 2 days (scope of tests indicated above). It is desirable to mix bitumen in the tank for at least 6 hours per day. The recommended storage temperature is 150–160°C.

#### Long-term storage at low temperature (over 7 days)

Due to its significant hardness, it is not recommended to store this binder if it is cooled down to ambient temperature (e.g. over winter) since it is very difficult to melt it.

### 5.3.6. ORBITON PMB 65/105-60

#### Intended use

Modified bitumen ORBITON 65/105-60 is intended for application in thin-layered hot mix wearing courses, where the mix demonstrates good mineral skeleton. It is produced from a soft base bitumen with high polymer content, which provides a product with excellent low-temperature properties.

ORBITON 65/105-60 is marked by higher penetration at 25°C (from 65 to 105) than modified bitumen 45/80-65, and at the same time demonstrates high cohesion<sup>2</sup> and elasticity. The combination of those features renders the product a very good binder for thin-layered gap graded mixes. Such applications include porous asphalt, BBTM and AUTL mixes for thin wearing courses and SMA mixes. Those are primarily special wearing courses and wearing courses used at low-temperature locations. Another use for this binder is bridge deck mixes where excellent elasticity and cohesion of the binder is required.

#### Properties acc. to PN-EN 14023:2011

**Table 5.12.** Properties of ORBITON 65/105-60 modified bitumen acc. to PN-EN 14023:2011

| Property  | Test method                    | Unit              | Requirement      |
|---|--------------------------------|-------------------|------------------|
| Penetration at 25°C   | EN 1426                        | 0.1 mm            | 65–105           |
| Softening Point R&B   | EN 1427                        | °C                | ≥ 60             |
| Elastic recovery at 25°C  | EN 13398                       | %                 | ≥ 70             |
| Fraass Breaking Point   | EN 12593                       | °C                | ≤ -15            |
| Flash Point   | EN ISO 2592                    | °C                | ≥ 235            |
| Force ductility<br>(low tension rate)                                 | EN 13589<br>EN 13703           | J/cm <sup>2</sup> | ≥ 3 at 5°C       |
| Change of mass after RTFOT  | EN 12607-1                     | %                 | ≤ 0.5            |
| Increase in softening point after RTFOT                               | EN 1427                        | °C                | ≤ 10             |
| Retained penetration after RTFOT                                      | EN 1426                        | %                 | ≥ 60             |
| Elastic recovery at 25°C after RTFOT                                  | EN 12607-1,<br>EN 13398        | %                 | ≥ 60             |
| Storage stability:<br>Difference in softening point                   | EN 13399,<br>EN 1427           | °C                | ≤ 5              |
| Storage stability:<br>Difference in penetration at 25°C               | EN 13399,<br>EN 1427           | 0.1 mm            | NR <sup>b</sup>  |
| Plasticity range  | EN 14023<br>Subsection 5.2.8.4 | °C                | NR <sup>b</sup>  |
| Drop in softening point after RTFOT                                   | EN 12607-1,<br>EN 1427         | °C                | TBR <sup>a</sup> |
| <sup>a</sup> TBR – To Be Reported<br><sup>b</sup> NR – No Requirement |                                |                   |                  |

#### Properties acc. to Superpave

ORBITON 65/105-60 properties acc. to *Superpave* (on the basis of tests conducted in 2009–2012).

- classification acc. to AASHTO MP 1: **PG 64-28**

2) Here: the measure of internal bitumen strength to; the ductility method with force measurement acc. to PN-EN 13589 is used for polymer-modified bitumen.

- high critical temperatures (AASHTO T 315):
  - $G^*/\sin\delta = 1$  kPa (unaged bitumen)  $T_{crit} = 74.9^\circ\text{C}$
  - $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)  $T_{crit} = 69.2^\circ\text{C}$
  - $G^*\cdot\sin\delta = 5\,000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 13.6^\circ\text{C}$
- low critical temperatures (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) \leq 300$  MPa  $T(S)_{60} = -20.5^\circ\text{C}$
  - temperature at  $m(60) \geq 0.3$   $T(m)_{60} = -20.6^\circ\text{C}$
  - stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 172$  MPa
- results and classification based on the MSCR (discussion in Chapter 7)

| Temperature   | 64°C        |
|---|-------------|
| $J_{nr}$ 0.1 kPa  | 0.382       |
| $J_{nr}$ 3.2 kPa  | 0.469       |
| $J_{nr}$ diff   | 22.9        |
| R 0.1 kPa   | 79.3        |
| R 3.2 kPa   | 76.1        |
| R diff  | 4           |
| Final classification of suitability for road traffic (at the test temperature) as per the most recent PG classification | E (Extreme) |

### Process temperatures

| At laboratory:  |           |
|---|-----------|
| Sample compaction temperature (Marshall samples or samples compacted in gyratory press) | 145–150°C |
| At mixing plant:  |           |
| Bitumen pumping temperature   | >150°C    |
| Temperature of bitumen for bituminous mixture production                                | 175–185°C |
| At site   |           |
| Minimum temperature of the supplied bituminous mixture (spreader's hopper)              | 155°C     |

### Viscosity dependence on temperature

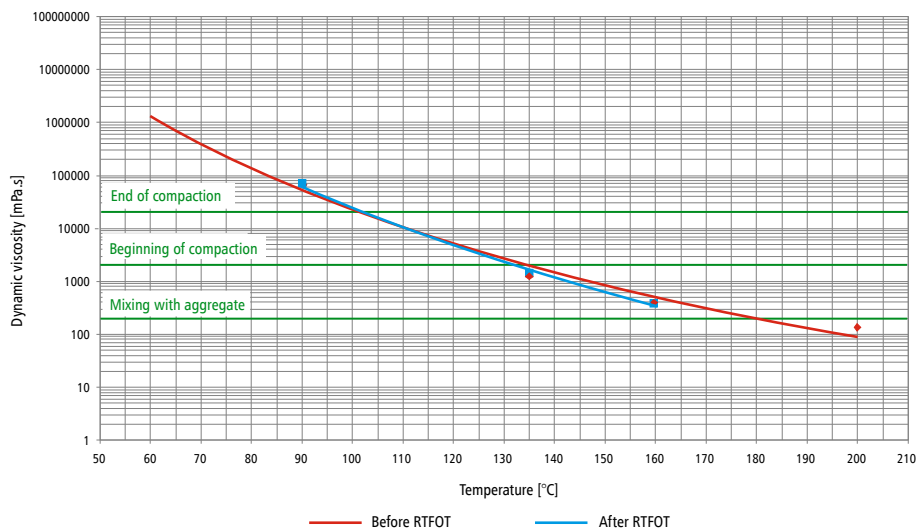


Figure 5.7. Viscosity dependence on temperature for modified bitumen ORBITON 65/105-60

**Table 5.13.** Example results of viscosity tests on bitumen ORBITON 65/105-60. Tests conducted by ORLEN Laboratorium sp. z o.o.

| Viscosity type | Test method                  | Reference document       | Equipment parameters | Unit | Test temperature  | Example test result for viscosity |
|----------------|------------------------------|--------------------------|----------------------|------|-------------------|-----------------------------------|
| dynamic        | Brookfield rotary viscometer | ASTM D4402<br>EN 13702-2 | spindle No 21, 29    | Pa*s | 90°C              | 70.00                             |
|                |                              |                          |                      |      | 135°C             | 1.23                              |
|                |                              |                          |                      |      | 160°C             | 0.39                              |
|                |                              |                          | spindle No 27        | Pa*s | 90°C after RTFOT  | 63.83                             |
|                |                              |                          |                      |      | 135°C after RTFOT | 1.36                              |
|                |                              |                          |                      |      | 160°C after RTFOT | 0.41                              |

### Microstructure

- polymer dispersion code acc. to EN 13632: B/H/S/r or B/H/S/o

### Storage

#### Short-term storage at high temperature (up to 7 days)

- recommended bitumen storage temperature: 160–180°C
- guaranteed period of bitumen service life for asphalt production: 7 days

It is recommended to conduct basic inspection tests for modified bitumen properties after 5 days in order to make sure that the product has not lost its properties due to the stability loss of the bitumen-polymer combination caused by component separation. The test should be conducted after 5 days of storage and every subsequent 2 days (7th day, 9th day, etc.) or in other time intervals, depending on the actual needs:

- penetration at 25°C, EN 1426
- Softening Point R&B, EN 1427
- elastic recovery at 25°C, EN 13398

If the mixing plant is fitted with tanks with agitators, bitumen should be periodically mixed in the tank. Circulation can be used for that purpose.

#### Long-term storage at high temperature (over 7 days)

It is not recommended to store modified bitumen for more than 7 days. Where such storage is necessary, we recommend periodic testing of binder properties, e.g. every 2 days (scope of tests indicated above). It is desirable to mix bitumen in the tank for at least 6 hours per day. The recommended storage temperature is 150–160°C.

#### Long-term storage at low temperature (over 7 days)

Due to its significant hardness, it is not recommended to store this binder if it is cooled down to ambient temperature (e.g. over winter) since it is very difficult to melt it.

## 5.4. Other properties of Polymer Modified Bitumens ORBITON

Among modified bitumen properties listed in EN 14023:2010 there are those which the manufacturer can provide to customers as additional data. They include e.g. bitumen density, process temperatures and polymer dispersion (microstructure observable with a microscope).

The data provided in Table 9.1 in Chapter 9 can be used for the design of bituminous mixtures and calculations acc. to EN 12697-8.

Process temperatures are provided in summary Table 8.2 in Chapter 8 (Section 8.6), while polymer-modified bitumen storage conditions in Section 8.2. There are also separated in present chapter 5.

Modified-polymer microstructure is presented in Table 5.1.4. The test was conducted according to EN 13632 *Bitumen and bituminous binders. Visualisation of polymer dispersion in polymer-modified bitumen.*

An optical microscope with an epifluorescence unit was used for the test. Observations were conducted in 100x magnification in reflected light, using an optical filter.

The structure of the tested polymer-modified bitumen was described using letter codes describing the dispersion combination of polymer and bitumen on the basis of Annex A.3 to EN 13632, see p. 2.3.5.

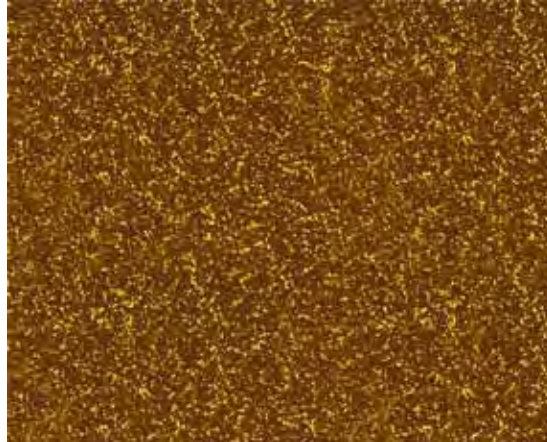
The following mixed designations are used in the description:

- H/I** – for samples with uniform dispersion of small polymer particles, with a markedly smaller quantity of medium particles with non-uniform dispersion
- S/M** – for samples with the largest share of small polymer particles, with a markedly smaller quantity of medium particles
- s/r** – for samples with particles in the form of elongated polymer clusters. The sample also contains cylindrical (round) particles and/or particles with a tendency to cluster

**Table 5.14.** Polymer-modified microstructure acc. to EN 13632

| Polymer-modified bitumen type | Phase description |                   |                  |                   |
|-------------------------------|-------------------|-------------------|------------------|-------------------|
|                               | Phase continuity  | Phase description | Size description | Shape description |
| 10/40-65                      | B                 | H                 | S                | r/o               |
| 25/55-60                      | B                 | H                 | S                | r/o               |
| 45/80-55 or EXP               | B                 | H                 | S                | r/o               |
| 45/80-65                      | B                 | H                 | S                | r/o               |
| 65/105-60                     | B                 | H                 | S                | r/o               |

Figure 5.8 shows an example image of polymer-modified bitumen microstructure captured in reflected light, 100x magnification (UV light).



**Figure 5.8.** Example image of polymer-modified microstructure with codes B-H-S-r





## Chapter 6

# HIGHLY MODIFIED BITUMENS ORBITON HIMA

### 6.1. Introduction

Research conducted by numerous academic centres over the last decades has corroborated the claim that higher polymer content in bitumen produces additional quality benefits, substantially contributing to the durability improvement of bituminous pavements in terms of cracking resistance, rutting and fatigue. Particularly encouraging was exceeding the limit of SBS polymer content (about 7–7.5% m/m), after which the polymer phase in the polymer-modified bitumen becomes continuous.

However, such a significant quantity of SBS for bitumen modification carried with it specific technical consequences for the production and application of modified bitumen in road engineering:

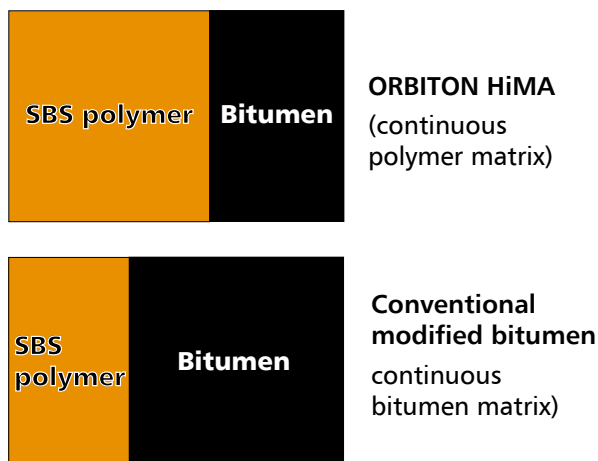
- stability problems in storage and transport of modified bitumen, as well as higher risk of polymer separation from the product,
- high viscosity, which means that such binders would have to be heated in the mixing plant to a much higher temperature than conventional polymer-modified bitumens,
- problems with compaction of the bituminous mixture containing highly viscous binders at the road construction site, causing rapid stiffening of the mixture and low compaction ratios.

The above limitations to the concept of highly-modified bitumen for road engineering uses represented a challenge not only for road binder manufacturers, but also for polymer suppliers. However, research work of the polymer industry have produced positive outcomes, resulting in the market availability, for a few years already, of a polymer which enables the production of highly-modified bitumen not having the downsides referred to above.

Bitumens of this type are referred to as **HiMA** – **H**ighly-**M**odified **A**sphalt.

### 6.2. Operating principle of HiMA

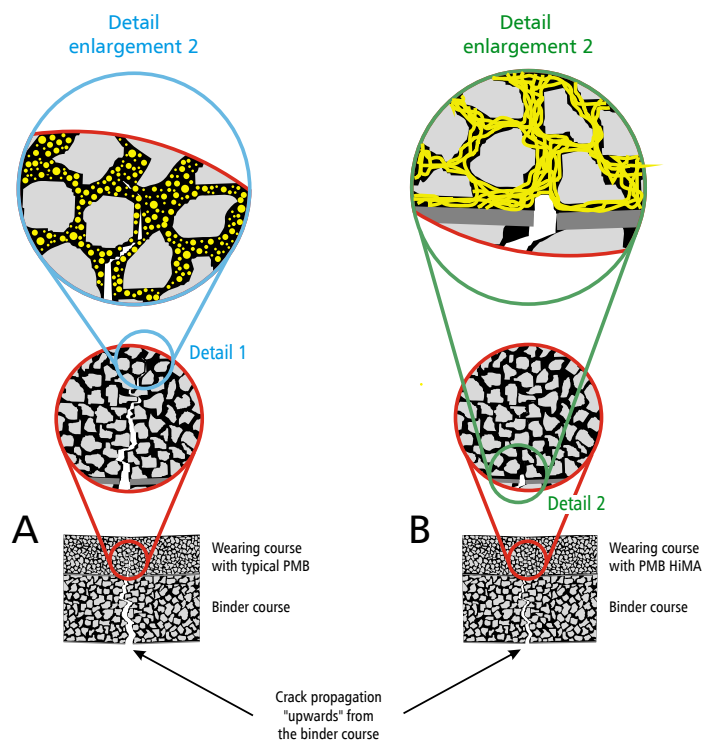
As already mentioned, the primary purpose behind highly-modified bitumen (asphalt) is to counteract pavement cracking, ruts, and to increase fatigue resistance. To achieve that, high polymer content is used, in excess of 7% m/m, which leads to phase reversal in the mixing of bitumen with the polymer (Figure 6.1).



**Figure 6.1.** Volumetric proportions between bitumen and polymer in conventional polymer-modified bitumen and highly-modified bitumen

The advantages of a continuous polymer network (polymer phase) in the binder, acting in the bituminous mix being as an elastic reinforcement, can be clearly demonstrated taking the example of limiting crack propagation by highly-modified binders. Figure 6.2 shows schematic representations of two hypothetical cases:

- Figure A: propagation of cracks reflected through the asphalt course with a conventional modified bitumen with non-continuous polymer network (marked with yellow dots) – here, the crack can pass through the course, finding weak spots in the binder between the polymer network sections,
- Figure B: propagation of cracks reflected through the asphalt course with HiMA with continuous polymer network (marked with yellow lines) – here, the crack's passing through the course is more difficult, because of the barrier formed by the polymer network in the binder.



**Figure 6.2.** Crack propagation through asphalt courses, a) with conventional polymer-modified bitumen, b) with highly-modified bitumen

### 6.3. ORBITON HiMA product family

Since 2011, the Technology, Research and Development Department of ORLEN Asphalt has been working on a new family of bituminous binders. Three new modified bitumen types have been developed as a result of laboratory work and production tests. These are:

- ORBITON 25/55-80 HiMA
- ORBITON 45/80-80 HiMA
- ORBITON 65/105-80 HiMA

All ORBITON HiMA types are classified according to the European system of EN 14023. Figure 6.3 presents a Pen<sub>25</sub>-T<sub>B&R</sub> chart showing how those products are positioned relative to paving-grade bitumen and (conventional) modified bitumen. A significant increase in the softening point range for all ORBITON HiMA types can be clearly seen, which is a direct effect of their high polymer content.

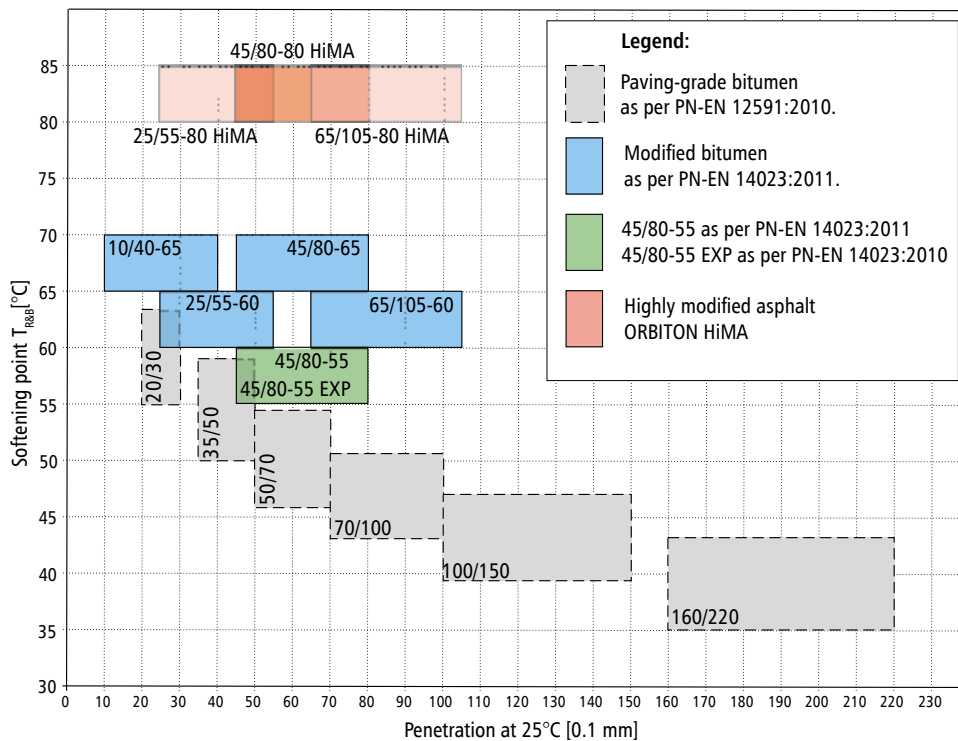


Figure 6.3. Positioning of ORBITON HiMA relative to paving-grade bitumen, and conventional modified bitumens

### 6.4. ORBITON HiMA test results

Highly-modified bitumens from the ORBITON HiMA family have been tested in the course of laboratory and process work. Below are test results of binders and asphalt mixtures containing those binders compared with other road binders manufactured by ORLEN Asphalt.

## Properties acc. to EN 14023

**Table 6.1.** Selected properties of ORBITON HiMA acc. to EN 14023

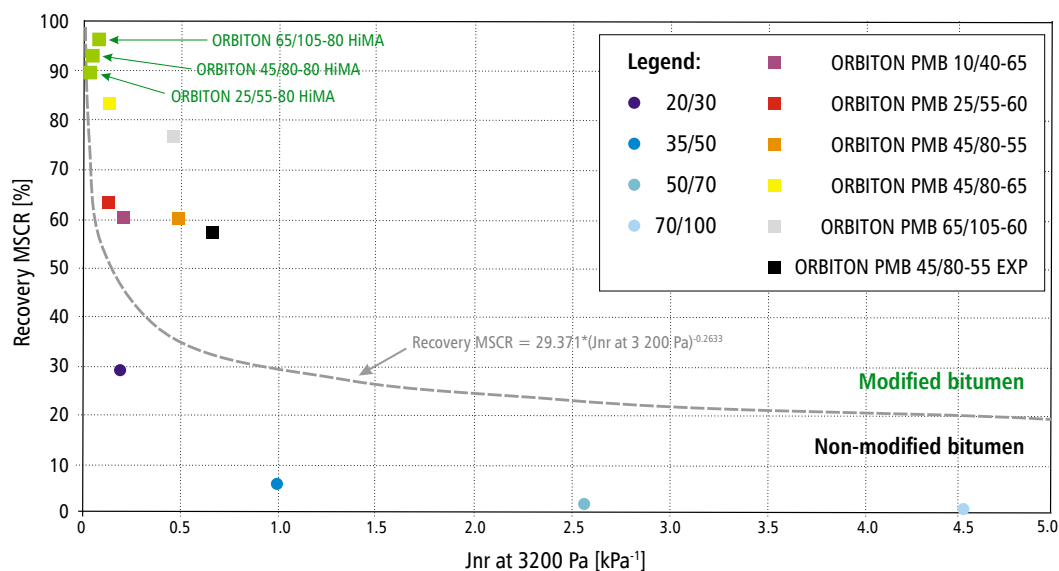
| Property   | Test method                     | Unit   | ORBITON HiMA |          |           |
|--|---------------------------------|--------|--------------|----------|-----------|
|  |                                 |        | 25/55-80     | 45/80-80 | 65/105-80 |
| Penetration at 2°C                               | EN 1426                         | 0.1 mm | 25 ÷ 55      | 45 ÷ 80  | 65 ÷ 105  |
| Softening point R&B                              | EN 1427                         | °C     | ≥80          | ≥80      | ≥80       |
| Fraass breaking point                            | EN 12593                        | °C     | ≤-15         | ≤-18     | ≤-18      |
| Elastic recovery at 25°C                         | EN 13398                        | %      | ≥80          | ≥80      | ≥80       |
| Storage stability:<br>Softening point difference | EN 13399,<br>EN 1427            | °C     | ≤5           | ≤5       | ≤5        |
| Plasticity range                                 | EN 14023<br>Subsection 5.2.8.4. | °C     | ≥95          | ≥95      | ≥100      |

## Properties acc. to Superpave

**Table 6.2.** Selected properties of ORBITON HiMA acc. to Superpave (PG system)

| Property   | Test method | ORBITON HiMA   |                |                |
|--|-------------|----------------|----------------|----------------|
|  |             | 25/55-80       | 45/80-80       | 65/105-80      |
| PG classification  | AASHTO MP 1 | 94-22          | 82-28          | 76-28          |
| Classification based<br>on the MSCR (for traffic category) at 64°C | ASTM D 7405 | E<br>(Extreme) | E<br>(Extreme) | E<br>(Extreme) |
| Classification based<br>on the MSCR (for traffic category) at 70°C | ASTM D 7405 | E<br>(Extreme) | E<br>(Extreme) | E<br>(Extreme) |

The summary chart in Figure 6.4 presents the results of ORBITON HiMA against the remaining road construction binders. What is particularly striking is excellent elasticity of HiMA binders at high service temperatures.



**Figure 6.4.** Test results of ORBITON HiMA on the MSCR chart, parameter Jnr at 3.2 kPa, 64°C

### Low-temperature pavement properties (cracking resistance)

Next to the testing of ORBITON HiMA binders, tests have also been conducted on asphalt mixtures containing those binders. Asphalt concrete AC 16S was used for the tests (comparative mixture as in Chapter 7) having the same gradation size and varying binder types (for comparison). The results of the tests (TSRST method acc. to EN 12697-46) is shown in Figure 6.5. To note, ORBITON HiMA perform better in comparison with other binders having similar hardness.

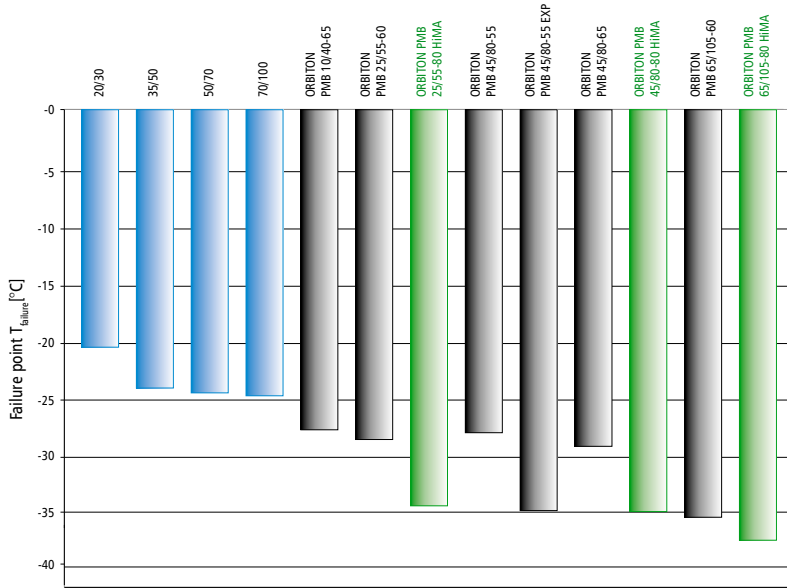


Figure 6.5. Pavement cracking resistance test results, TSRST method EN 12697-46, AC 16S mix

### High-temperature pavement properties (rutting resistance)

High-temperature properties of HiMA (rutting resistance) have also been tested. The same bituminous mixture was used for the tests (AC 16S) conducted acc. to EN 12697-22 in a small apparatus (method B), in the air, at 60°C, with 10 000 loading cycles. The results are shown in Figure 6.6.

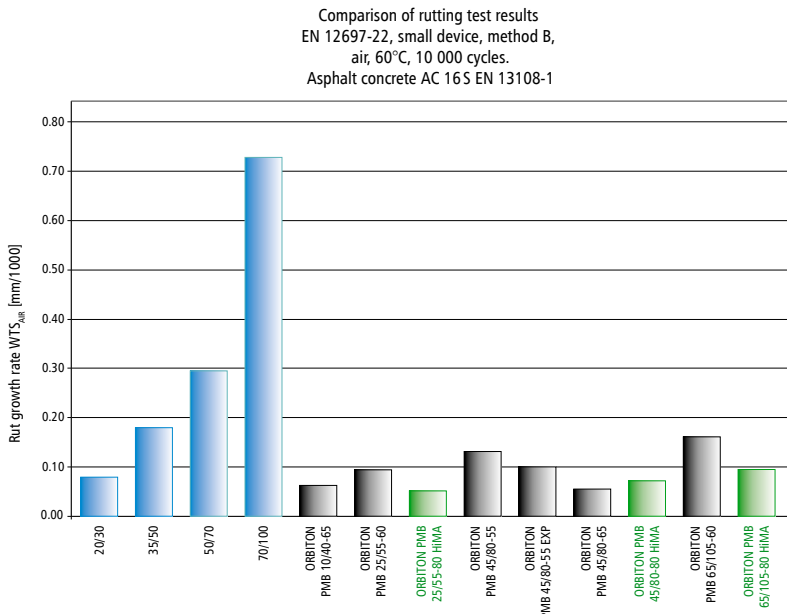


Figure 6.6. Pavement rutting resistance test results, parameter  $WTS_{AIR}$ , method EN 12697-22, small wheel tracker (method B) in the air, at 60°C, with 10 000 loading cycles, AC 16S mix

## 6.5. Experimental section in Poland

In Autumn 2013, an experimental section of road pavement with ORBITON 65/105-80 HiMA was completed in Poland. This was the 6th section with HiMA in Europe.

Two wearing course sections were placed, one made of AC 11 (layer thickness of 4 cm), and the other of a special SMA 5 DSH mix (silent pavement, 2 cm thick layer). The experimental section provided a lot of process data and proved that the production and compaction properties of the bituminous mixture with highly-modified, HiMA-type binder are close to those demonstrated by SBS-modified bitumen types.

Figure 6.7 shows a photograph from the process of section placement.



Figure 6.7. Placement of experimental section with ORBITON 65/105-80 HiMA in 2013.

## 6.6. Conclusion

Several years of research work to develop and launch for production a new group of highly-modified SBS binders referred to as ORBITON HiMA ended in 2013 with the placement of an experimental section in Poland. Having analysed the results from the binder and asphalt mixture tests, as well as conclusions from the placement process, we are confident that binders of this type will soon become an important part of ORLEN Asphalt's offering.

## Chapter 7

# TEST RESULTS OF BITUMEN IN ASPHALT MIXTURES

### 7.1. Introduction

Chapters 4, 5 and 6 of this Handbook discuss the properties of all paving-grade, modified and highly-modified bitumen types manufactured by ORLEN Asphalt. For obvious reasons, those properties have to meet the requirements of the relevant reference documents on the basis of which they are produced.

The discussed binder properties enable their tentative classification in terms of resistance to external factors they are exposed to in the various bituminous mixtures (AC, SMA, MA, etc.) and the courses they are incorporated into (e.g. wearing course, binder course, base).

From the perspective of road administrators, pavement designers and contractors, it is vital to know the behaviour of bitumen in the pavement exposed to the actual service conditions. Knowing that testing binders alone is important, but does not provide road engineers with comprehensive knowledge they expect, ORLEN Asphalt has long been testing the behaviour of bitumen in asphalt mixtures, examining their resistance to damage at both high and low temperatures. This chapter presents test results for high and low-temperature rutting and cracking of various asphalt mixtures incorporating ORLEN Asphalt's binders.

### 7.2. Rutting resistance tests

One of the key service parameters of asphalt mixtures is rutting resistance. The contribution of bituminous binders of any type is never decisive in ensuring pavement resistance to rutting; however, careful selection of the bitumen type can support the mineral aggregate skeleton of the pavement structure. This chapter presents comparative test results for asphalt mixture rutting resistance, conducted by ORLEN Asphalt in 2008–2013. The tests were conducted according to PN-EN 12697-22.

Particular test conditions included:

- simulation of rutting with the so-called small device,
- testing in the air, procedure B,
- test temperature of 60°C,
- number of sample loading cycles – 10 000,
- thickness of the plate for rutting – 60 mm,
- AC 16 mixture, binder content of 5.6% m/m (fixed content in all tests, regardless of bitumen type).

A slightly higher-than-usual binder content (5.6%) was used in the AC16 mixture in order to enable the comparison of the effects of a broad range of binders – from very hard 20/30 to very soft 70/100. The application of a drier AC 16 mix would cluster the results for all hard binders into a very narrow  $WTS_{AIR}$  range – from 0.01 to 0.05 mm/1 000 cycles, which would frustrate the comparison effort in practical terms.

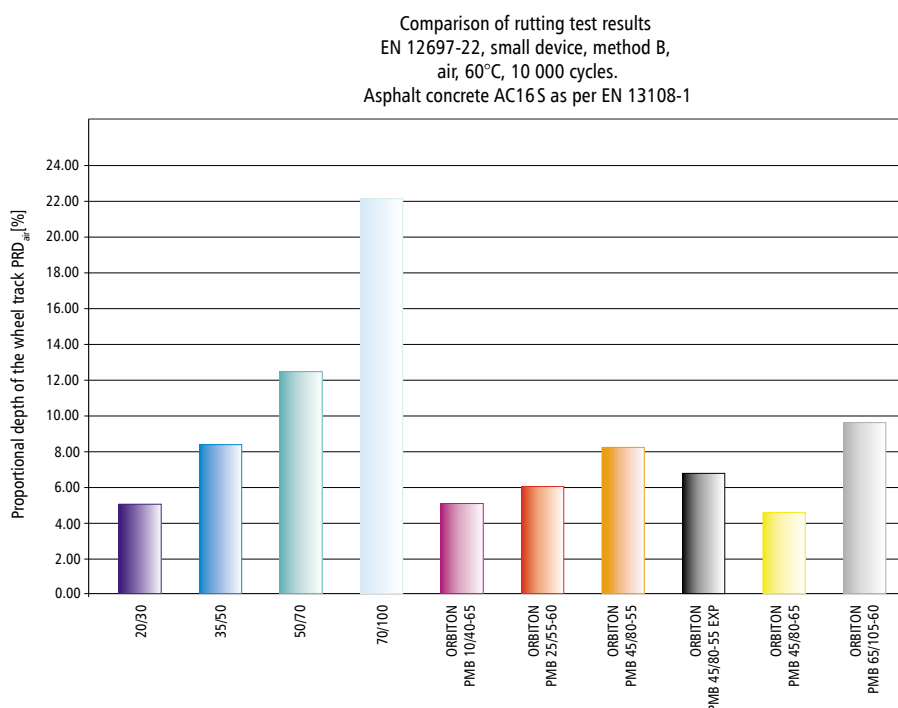
Test results presented in this chapter apply to all types of bituminous binders. Since the axle load limit in Poland is 115 kN (11.5 tonnes), a small device for rutting was used for the tests, in accordance with the guidelines of PN-EN 13108. Most Polish guidelines on asphalt mixtures provide the requirement for rutting resistance only for  $WTS_{AIR}$ , whereas for  $PRD_{AIR}$ , "report the result" option is used.

### 7.2.1. Comparative testing of all binders with AC 16S

The same asphalt concrete mixture AC16 acc. to PN-EN 13108-1 was used for the 2012 and 2013 comparative testing of all ORLEN Asphalt-manufactured road binders for hot asphalt mixes. The following were tested:

- paving-grade bitumen: 20/30, 35/50, 50/70, 70/100,
- modified bitumen ORBITON: 10/40-65, 25/55-60, 45/80-55, 45/80-55 EXP, 45/80-65, 65/105-60.

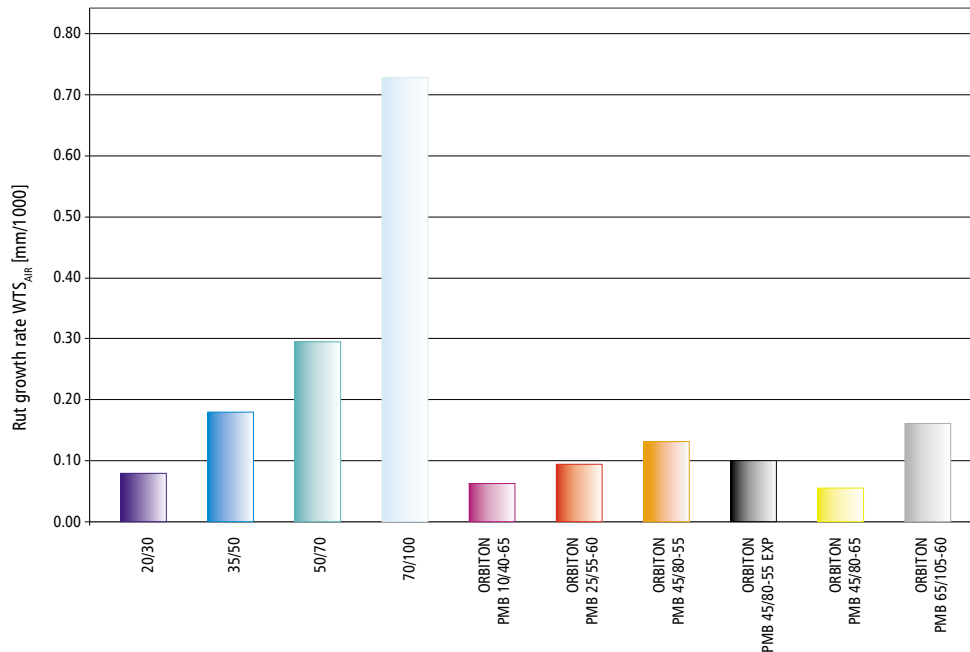
Ten bituminous binders were compared for their contribution to asphalt concrete resistance to rutting. The results are shown on Figures 7.1 and 7.2 as  $PRD_{AIR}$  and  $WST_{AIR}$ . Importantly, the results are presented in the charts only for comparison between the binders themselves, so  $WTS$  and  $PRD$  values should not be interpreted as the ultimate measure.



**Figure 7.1.**  $PRD_{AIR}$  comparative tests for 10 bituminous binders manufactured by ORLEN Asphalt in 2012 and 2013. AC 16 asphalt mixture



Comparison of rutting test results  
 EN 12697-22, small device, method B,  
 air, 60°C, 10 000 cycles.  
 Asphalt concrete AC16S as per EN 13108-1



**Figure 7.2.** WTS<sub>AIR</sub> comparative tests for 10 bituminous binders manufactured by ORLEN Asphalt in 2012. AC 16 asphalt mixture

What transpires is the dependence of rutting resistance on binder hardness and viscosity. Obviously, good rutting resistance is achieved with very hard binders, such as paving-grade bitumen 20/30 or modified bitumen ORBITON 10/40-65. In turn, softer binders showing relatively high viscosity perform much better than paving-grade bitumen types, which applies in particular to modified bitumen types.

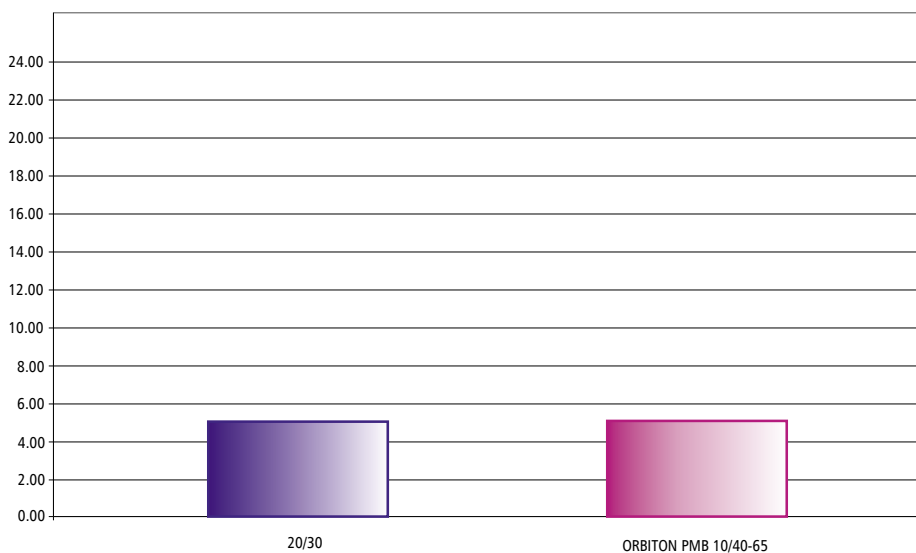
## 7.2.2. Comparative tests between individual binders

Comparative tests directly between paving-grade and modified bitumens were conducted in 2008–2013. In analysing the results it must be remembered that they pertain to specific asphalt mixtures and would not necessarily be repeated for the mixtures with other aggregates, different gradation, different binder content and other volumetric properties. The purpose of the tests was primarily to examine differences in the various bitumen types' contribution to rutting resistance.

### 7.2.2.1. ORBITON 10/40-65

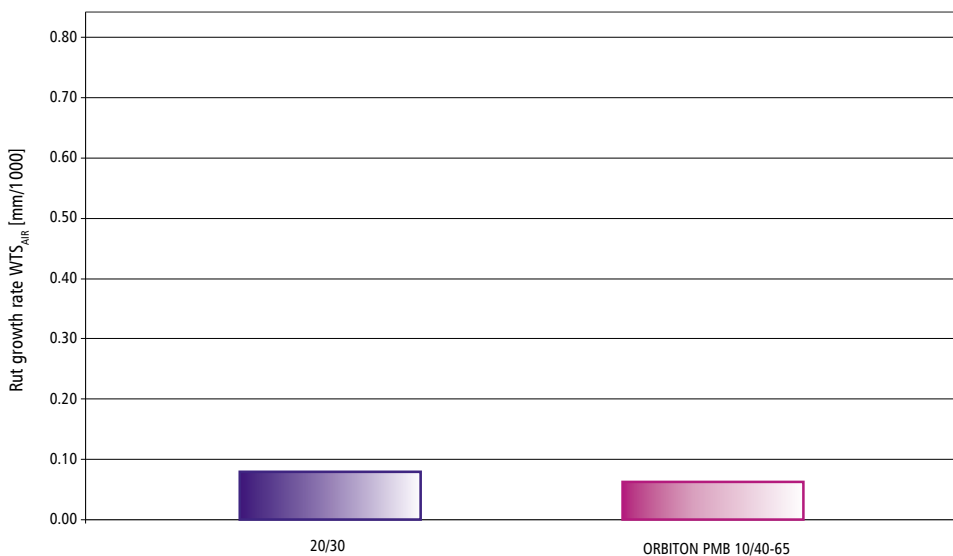
Figure 7.3 and 7.4 show comparative test results for AC 16, with paving-grade bitumen 20/30 and polymer-modified bitumen 10/40-65.

Comparison of rutting test results  
 EN 12697-22, small device, method B,  
 air, 60 °C, 10 000 cycles.  
 Asphalt concrete AC16S as per EN 13108-1



**Figure 7.3.** Results of comparative tests for rutting resistance ( $PRD_{AIR}$ ) of AC 16S between paving-grade bitumen 20/30 and modified bitumen ORBITON 10/40-65

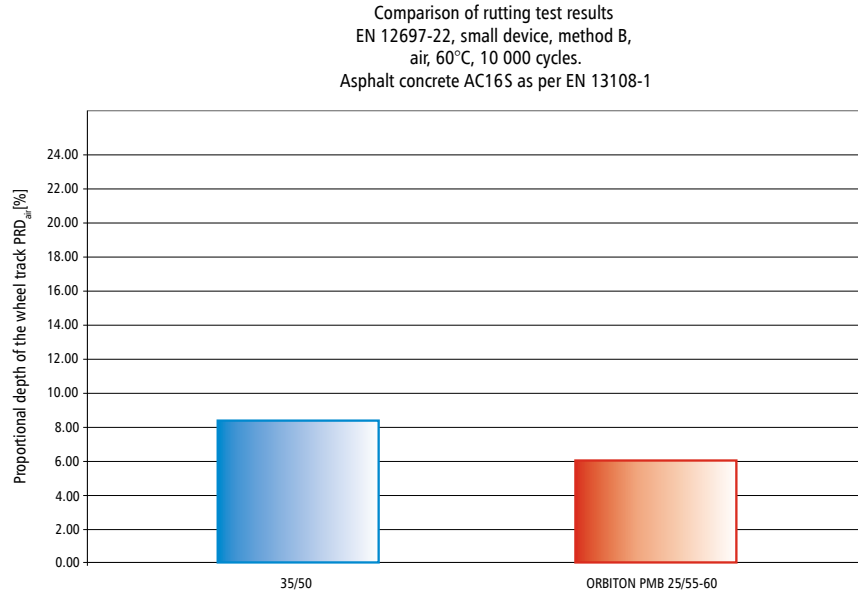
Comparison of rutting test results  
 EN 12697-22, small device, method B,  
 air, 60°C, 10 000 cycles.  
 Asphalt concrete AC16S as per EN 13108-1



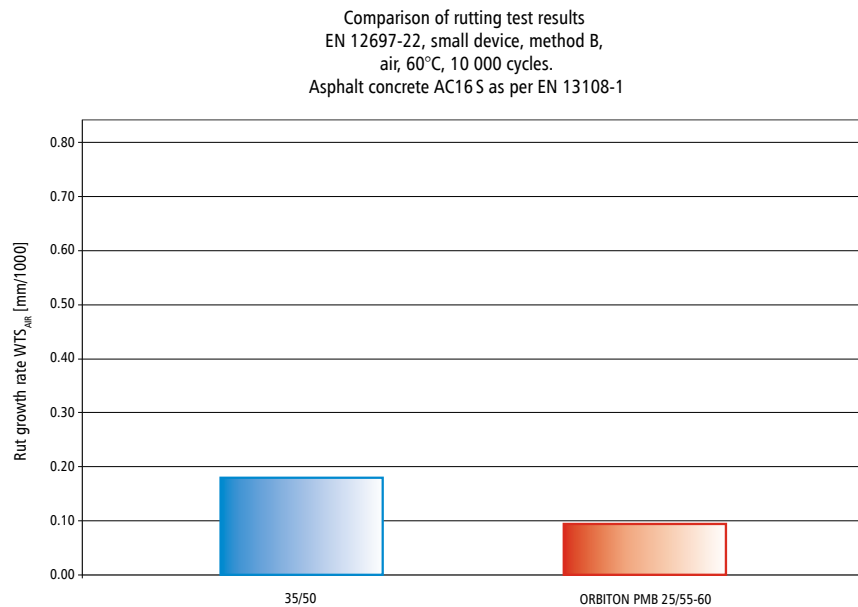
**Figure 7.4.** Results of comparative tests for rutting resistance ( $WTS_{AIR}$ ) of AC 16S between paving-grade bitumen 20/30 and modified bitumen ORBITON 10/40-65

**7.2.2.2. ORBITON 25/55-60**

Figures 7.5 and 7.6 show comparative test results for AC 16, binder course mixture (W), with paving-grade bitumen 35/50 and polymer-modified bitumen ORBITON 25/55-60. For both key rutting parameters ORBITON 25/55-60 demonstrates very good properties as compared with similar-hardness paving-grade bitumen (35/50).



**Figure 7.5.** Results of comparative tests for rutting resistance (PRD<sub>AIR</sub>) of AC 16S between paving-grade bitumen 35/50 and modified bitumen ORBITON 25/55-60

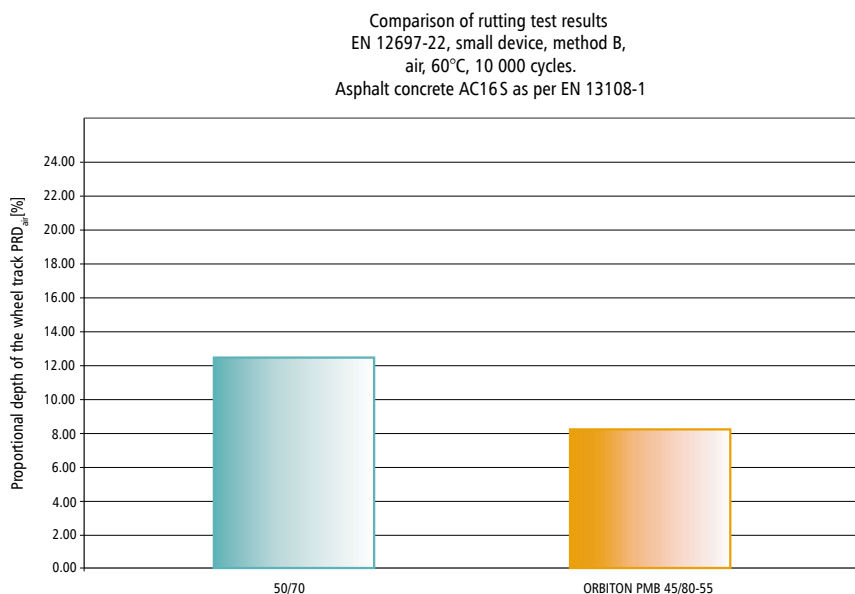


**Figure 7.6.** Results of comparative tests for rutting resistance (WTS<sub>AIR</sub>) of AC 16S between paving-grade bitumen 35/50 and modified bitumen ORBITON 25/55-60

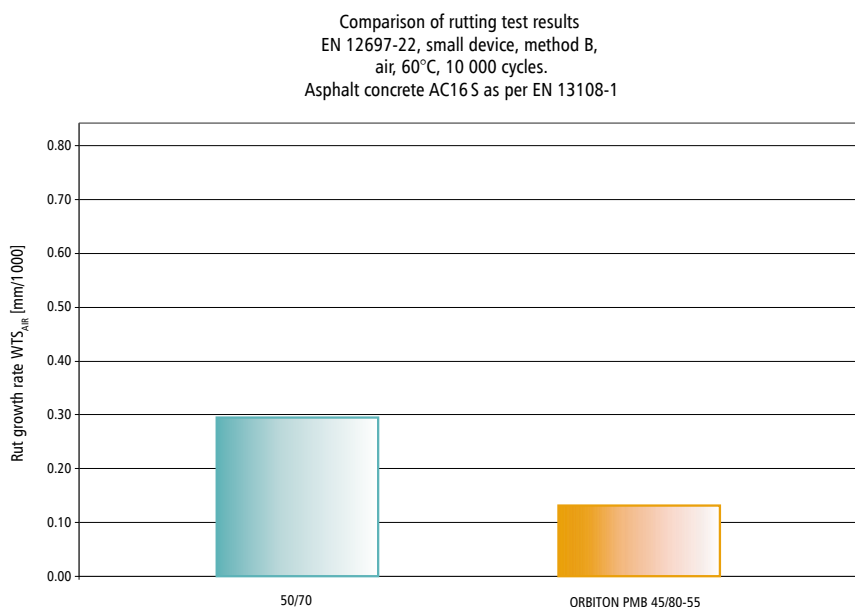
### 7.2.2.3. ORBITON 45/80-55

ORBITON 45/80-55 is one of the most popular modified bitumen types used in Poland for wearing courses. It is marked by good service properties and easy placement.

Figures 7.7 and 7.8 show comparative test results for AC 16S with paving-grade bitumen 50/70 and polymer-modified bitumen ORBITON 45/80-55. In terms of both key rutting parameters, ORBITON 45/80-55 demonstrates incomparably better properties than similar-hardness paving-grade bitumen (50/70).



**Figure 7.7.** Results of comparative tests for rutting resistance ( $PRD_{AIR}$ ) of AC 16S between paving-grade bitumen 50/70 and modified bitumen ORBITON 45/80-55

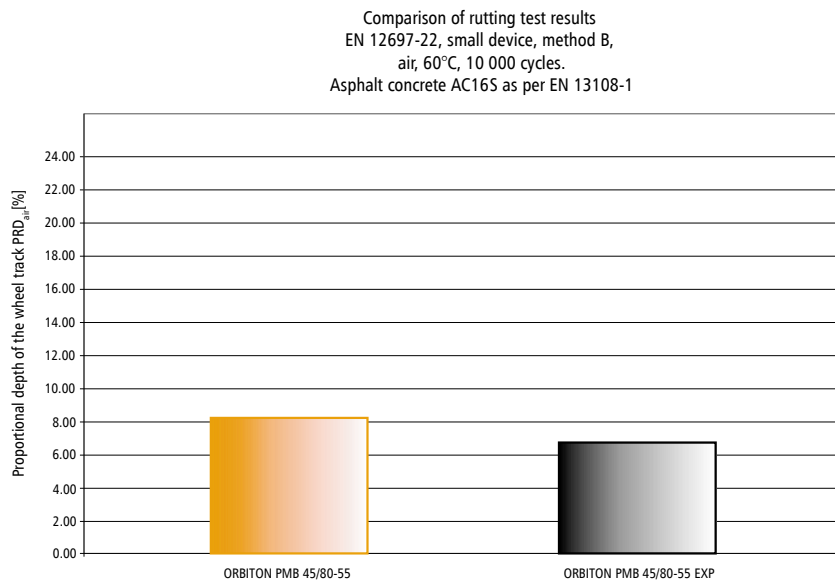


**Figure 7.8.** Results of comparative tests for rutting resistance ( $WTS_{AIR}$ ) of AC 16S between paving-grade bitumen 50/70 and modified bitumen ORBITON 45/80-55

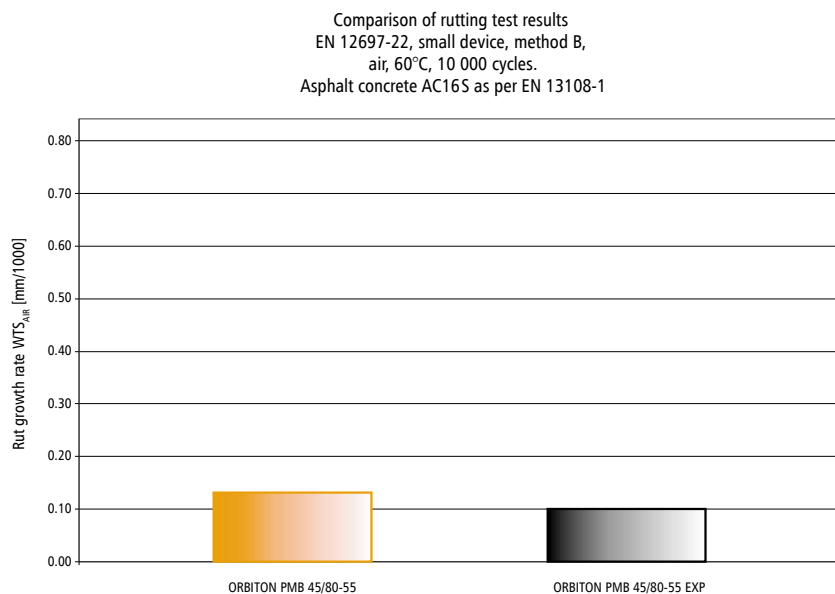
### 7.2.2.4. ORBITON 45/80-55 EXP

The production of ORBITON 45/80-55 EXP for the Baltic States' markets (Lithuania, Latvia and Estonia) was launched in 2013. This binder differs from the conventional ORBITON 45/80-55 as it has higher polymer content and demonstrates better low-temperature performance (Fraass  $\leq -15^{\circ}\text{C}$ ). Excellent properties of ORBITON 45/80-55 EXP have led to its launch also on the Polish market, and gradual replacement of ORBITON 45/80-55.

Figures 7.9 and 7.10 show comparative test results for AC 16S with polymer-modified bitumen ORBITON 45/80-55 and polymer-modified bitumen ORBITON 45/80-55 EXP. For both key rutting parameters ORBITON 45/80-55 EXP demonstrates excellent properties as compared with similar-hardness ORBITON 45/80-55.



**Figure 7.9.** Results of comparative tests for rutting resistance (PRD<sub>AIR</sub>) of AC 16S between modified bitumen ORBITON 45/80-55 and modified bitumen ORBITON 45/80-55 EXP

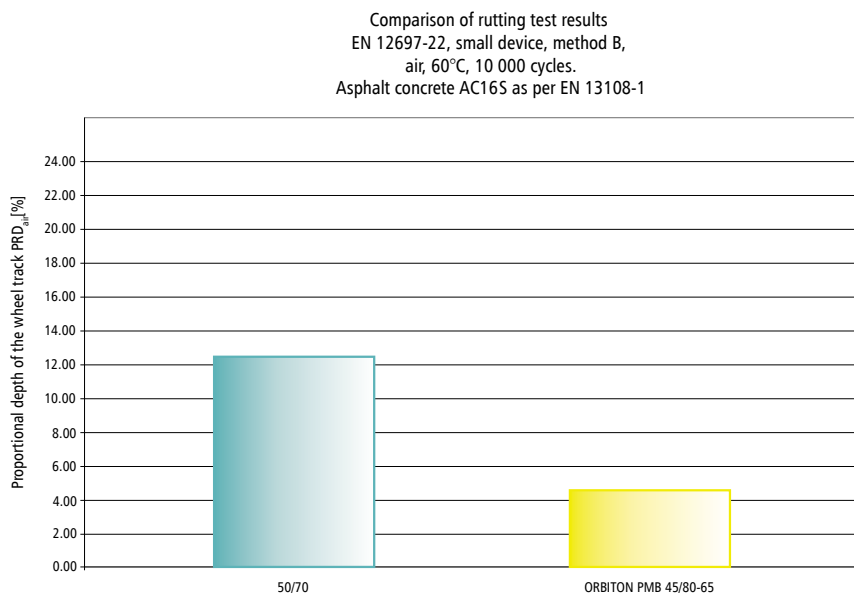


**Figure 7.10.** Results of comparative tests for rutting resistance (WTS<sub>AIR</sub>) of AC 16S between modified bitumen ORBITON 45/80-55 and modified bitumen ORBITON 45/80-55 EXP

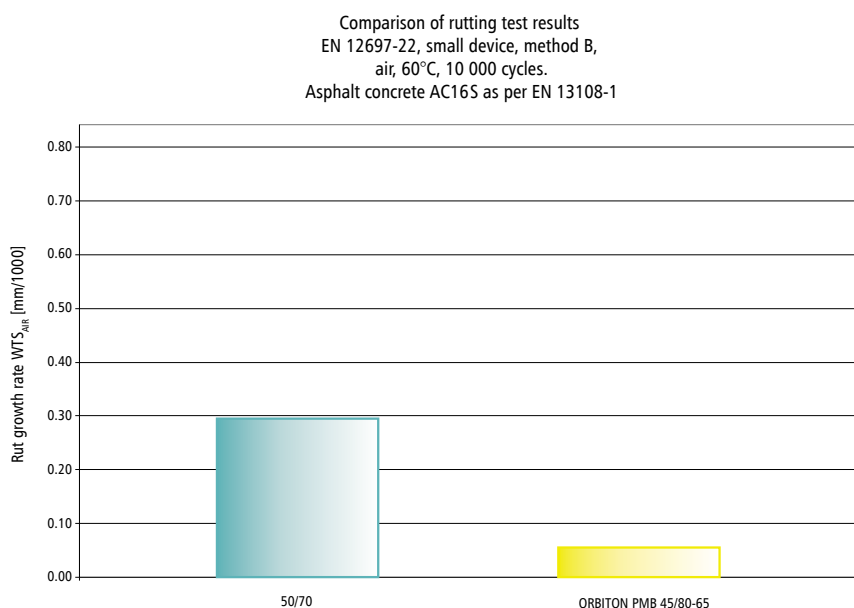
### 7.2.2.5. ORBITON 45/80-65

ORBITON 45/80-65 is a modified bitumen type for wearing courses. It is marked by very good service properties, such as high softening point and elasticity.

Figures 7.11 and 7.12 show comparative test results for AC 16S with paving-grade bitumen 50/70 and polymer-modified bitumen ORBITON 45/80-65. In terms of both key rutting parameters, ORBITON 45/80-65 demonstrates markedly better properties than similar-hardness paving-grade bitumen (50/70).



**Figure 7.11.** Results of comparative tests for rutting resistance (PRD<sub>AIR</sub>) of AC 16S between paving-grade bitumen 50/70 and modified bitumen ORBITON 45/80-65

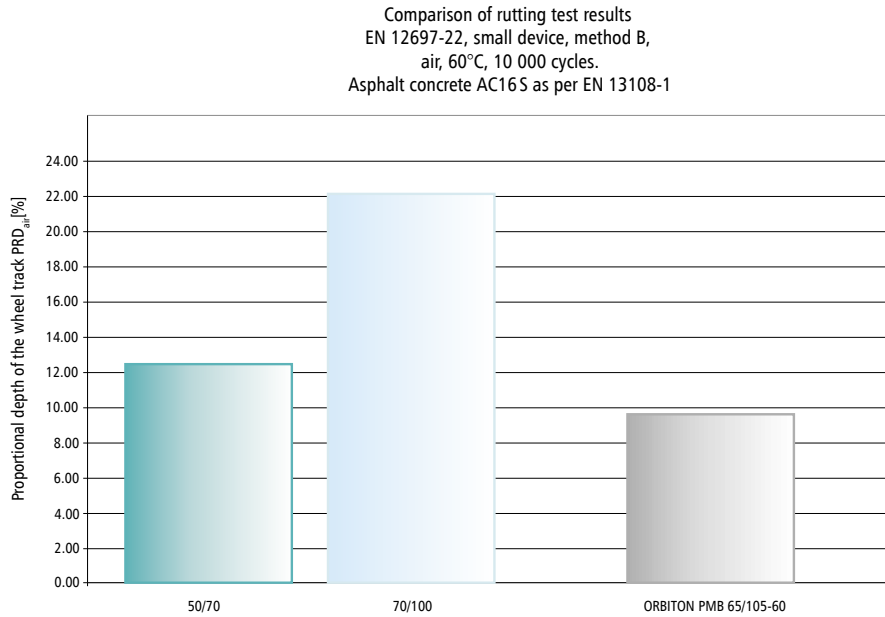


**Figure 7.12.** Results of comparative tests for rutting resistance (WTS<sub>AIR</sub>) of AC 16S between paving-grade bitumen 50/70 and modified bitumen ORBITON 45/80-65

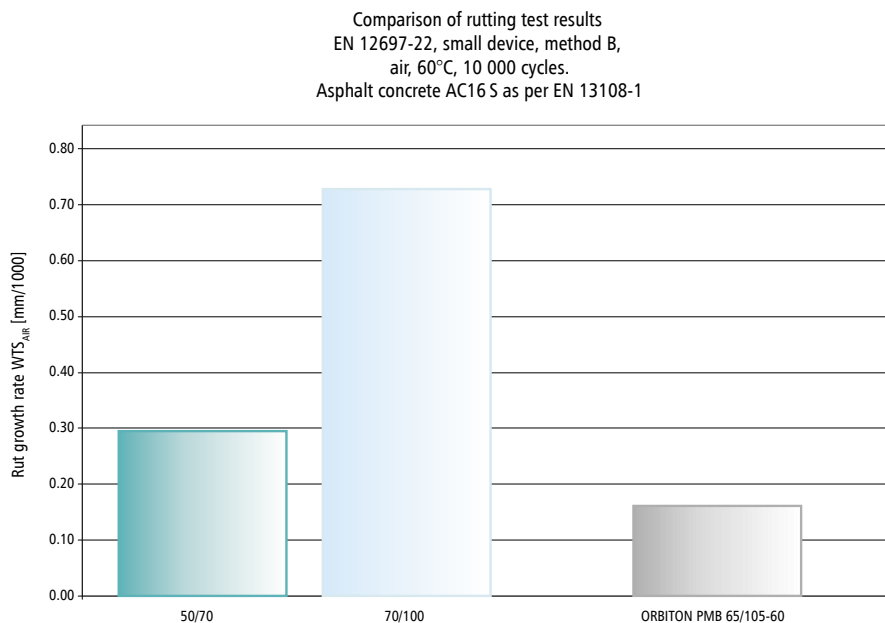
### 7.2.2.6. ORBITON 65/105-60

ORBITON 65/105-60 is a soft modified bitumen type for wearing courses. It is marked by very good ductility and elasticity and excellent low-temperature properties.

Figures 7.13 and 7.14 show comparative test results for AC 16S with paving-grade bitumens 50/70 and 70/100 and polymer-modified bitumen ORBITON 65/105-60. In terms of key rutting parameters, ORBITON 65/105-60 demonstrates markedly better properties than similar-hardness paving-grade bitumen types (50/70 and 70/100).



**Figure 7.13.** Results of comparative tests for rutting resistance (PRD<sub>AIR</sub>) of AC 16S between paving-grade bitumens 50/70 and 70/100 and modified bitumen ORBITON 65/105-60



**Figure 7.14.** Results of comparative tests for rutting resistance (WTS<sub>AIR</sub>) of AC 16S between paving-grade bitumens 50/70 and 70/100 and modified bitumen ORBITON 65/105-60

## 7.3. Low-temperature cracking resistance tests

Rutting resistance tests verify the behaviour of the bituminous mixture at a relatively high temperature (60°C). We know, however, that the pavement is also damaged in winter when temperatures drop below 0°C. Low-temperature shrinkage cracks, which may emerge in winter, markedly reduce the pavement's service life and generate extra cost of repair and sealing.

### 7.3.1. TSRST method

Various test methods were used in the past to answer the question to what extent the asphalt mixture resists cracking. A new standard was made available in 2012, describing a number of useful testing methods: PN-EN 12697-46 *Bituminous mixtures – Test methods for hot mix asphalt – Part 46: Low temperature cracking and properties by uniaxial tension tests*. From among the testing methods indicated in the standard, ORLEN Asphalt selected TSRST (*Thermal Stress Restrained Specimen Test*). The TSRST method is standardised in the US as AASHTO TP 10.

The TSRST test involves the preparation of the sample as a cylinder or rectangular prism from the compacted asphalt mixture (samples are typically cut out from a plate). Samples ends are attached to a rigid, non-deformable frame and placed in a cryostasis chamber. During the TSRST temperature in the chamber is reduced at the pre-agreed rate of  $dT = -10$  K/h. As a result of the temperature drop the sample shrinks; however, fastening of the sample to the frame prevents sample deformation, which causes internal tension stress to appear in the sample, leading to shrinkage cracking. The following values are obtained from the test: failure point  $T_{\text{Failure}}$  and failure stress  $\sigma_{\text{failure}}$ . Tension stress is also recorded as a function of temperature:  $\sigma_{\text{cri}}(T)$ .

### 7.3.2. Comparative testing of all binders with AC 16S

Figures 7.15 and 7.16 show the effect of bituminous binders on asphalt concrete resistance to low-temperature cracking. The results, taking the form of the failure point  $T_{\text{failure}}$  and failure stress  $\sigma_{\text{failure}}$  are shown on separate charts. The tests were conducted on the same AC 16S asphalt mixture that was used for rutting tests.

Obviously, failure points provided on Chart 7.15. are not expected to be precisely replicated in the actual pavement. It must be remembered that the TSRST gives a **conventional** failure point under specific testing conditions. It very rarely takes place in reality that a temperature drop reaches 10°C/hour. It can therefore be assumed that the results pertain to extreme conditions, and the actual failure point would be smaller, in particular with the contribution of a smaller temperature drop gradient. An additional aspect of the cracking test evaluation is the content of the binder in the tested AC 16S. The mixture contains relatively high binder content (5.6%), and therefore it cannot be said to be dry. On the other hand, however, it is not true that the more binder we have, the smaller the failure point will be. As with the majority of asphalt mixtures, the optimum quantity of the binder in the mix also exists for this case. For practical reasons, certain assumptions for the comparison were adopted under ORLEN Asphalt's long-term research programme, owing to which we can compare binders in terms of their sensitivity to cracking under specific conditions. Let us therefore look at the bar sizes and compare them rather than analyse the failure point.



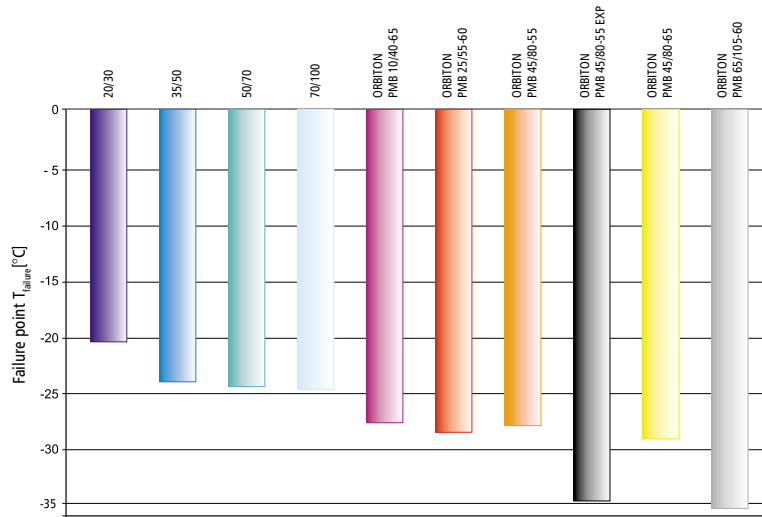


Figure 7.15. Results of comparative tests for the failure point  $T_{failure}$  for AC 16S with 10 bituminous binders manufactured by ORLEN Asfalt

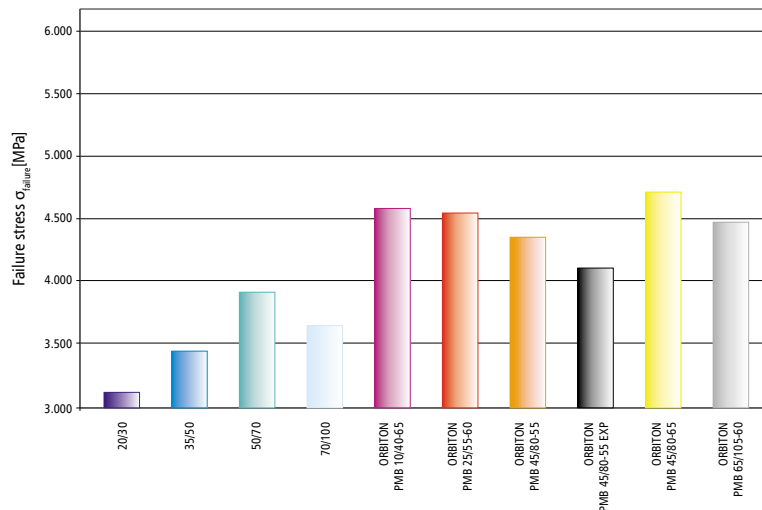


Figure 7.16. Results of comparative tests for the failure stress  $\sigma_{failure}$  for AC 16S with 10 bituminous binders manufactured by ORLEN Asfalt

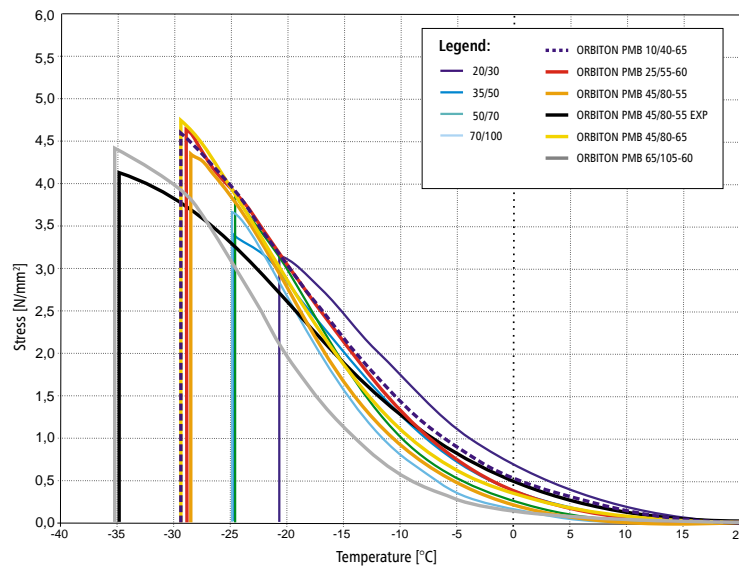


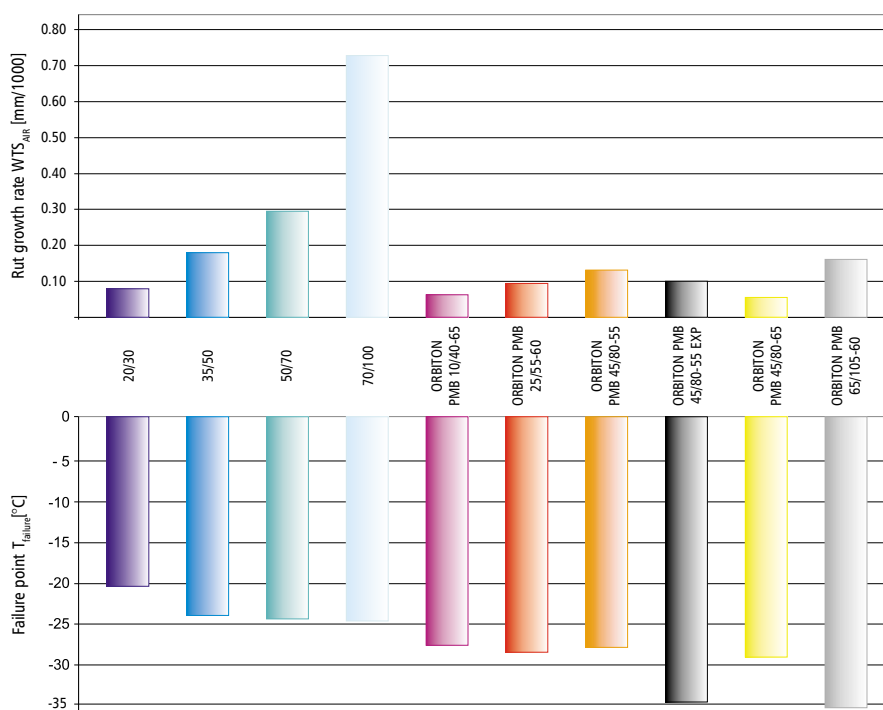
Figure 7.17. Results of comparative tests for the difference in the tension stress in the course of the TSRST depending on temperature  $\sigma_{cry}(T)$  for AC 16S with 10 bituminous binders manufactured by ORLEN Asfalt

## 7.4. Conclusions

This chapter presented separate comparative test results for rutting and low-temperature cracking. Some of the presented tests were covered by the research programme examining performance properties of binders manufactured by ORLEN Asphalt.

Already at the time that road engineers started analysing high- and low-temperature properties of bituminous binders and their effect on pavement behaviour, it was known that it would be very difficult to come up with a universal binder. Figure 7.18 shows the problem in detail. Binders with a very good contribution to pavement resistance to rutting are not the best solution if we expected cold winters. Importantly, however, we can influence pavement cracking to a smaller degree than is the case for rutting, and therefore it is worth applying softer binders with good low-temperature resistance – e.g. ORBITON 65/105-60. However, it requires hard work on the development of the mineral skeleton in the mix to ensuring the required level of resistance to lasting deformations in summer.

Figure 7.18 presents a summary comparison of all 10 binders covered by the research programme of ORLEN Asphalt in terms of their rutting resistance (parameter  $WTS_{AIR}$ ) and failure resistance (failure point  $T_{failure}$ ).



**Figure 7.18.** Comparative test results of bituminous binders at high and low temperatures. Comparison of parameter  $WTS_{AIR}$  and failure point  $T_{failure}$ . Bituminous mixture AC 16 S



## Chapter 8

# BITUMEN PROPERTIES ACCORDING TO SUPERPAVE

Test methods and requirements used in European standards on road binders deal primarily with the so-called commercial quality. Performance (service) quality is a much broader phenomenon and comprises a wider range of parameters. One of the systems describing the performance aspect of bituminous binders is the Performance Grade (PG) System developed in the US.

In 2006–2013, the Technology, Research and Development Department of ORLEN Asphalt tested bitumen properties using testing methods based on the American *Superpave* methodology (AASHTO MP1).

The tests covered selected road bitumen and polymer-modified bitumen types. Experimental binders and new binder concepts were also examined, although they are beyond the scope of this Handbook (except for ORBITON HiMA).

### 8.1. Superpave

A major research programme was launched in the United States in 1987, referred to as the Strategic Highway Research Program (SHRP). One of its objectives was to develop a new classification system for road pavements focusing on the binders' performance of specific functions in the pavement. The entire system, including a method for designing asphalt mixtures, was named *Superpave* (*SUPERior PERforming Asphalt PAVEMENTs*).

Following the introduction of the *Superpave* system, "classical" parameters, such as penetration, softening point, etc. were abandoned in the US. The basis for the binder classification system according to *Superpave* is the range of bitumen service temperatures, referred to as PG – **Performance Grade**.

Binder performance grade is designated as **PG X-Y**, where:

- x is the maximum pavement temperature ("high PG"),
- y is the minimum pavement temperature ("low PG"),

at which a given bitumen type is able to operate as required. It can therefore be argued that the performance grade is primarily determined by the weather in the area where the road is constructed. High and low PG are selected from the list, with intervals of 6°C, on the basis of binder test results (Table 8.1)

**Table 8.1.** Performance grade series

| High temperature ("high PG") | Low temperature ("low PG")        |
|------------------------------|-----------------------------------|
| PG 46-                       | -34, -40, -46                     |
| PG 52-                       | -10, -16, -22, -28, -34, -40, -46 |
| PG 58-                       | -16, -22, -28, -34, -40           |
| PG 64-                       | -10, -16, -22, -28, -34, -40      |
| PG 70-                       | -10, -16, -22, -28, -34, -40      |
| PG 76-                       | -10, -16, -22, -28, -34           |
| PG 82-                       | -10, -16, -22, -28, -34           |

The bituminous binder testing system for PG determination involves tests at various temperatures, since bitumen is a thermoplastic material, and therefore its properties change as the temperature changes. The change in those properties translates into specific pavement damage types:

- high bitumen temperature → viscoplastic deformations,
- low bitumen temperature → low-temperature cracking,
- intermediate bitumen temperature → fatigue damage from vehicle traffic,

considering the impact of short-term and long-term ageing on bitumen properties. Temperatures of specific tests depend on the expected pavement temperature at the placement location.

New testing equipment was also developed as a result of the work on the *Superpave* system, now used to examine bitumen properties. Table 8.2 lists those equipment items and their intended use.

**Table 8.2.** New testing equipment as per Superpave

| Equipment item                      | Intended use  |
|-------------------------------------|---|
| Dynamic Shear Rheometer (DSR)       | Determination of binder properties at medium and high temperatures                |
| Rotational Viscometer (RV)          | Determination of binder properties at the asphalt mixture production temperatures |
| Bending Beam Rheometer (BBR)        | Determination of binder properties at low temperatures                            |
| Direct Tension Test (DTT)           |   |
| Rolling Thin Film Oven Test (RTFOT) | Short-term ageing simulation  |
| Pressure Ageing Vessel (PAV)        | Long-term ageing simulation, the test is usually preceded by RTFOT                |

## 8.2. Low-temperature properties testing

The Fraass breaking point test used in Europe has a number of weaknesses and is criticised by many. In the US, the bending beam rheometer (BBR) is primarily used to test bitumen behaviour at low temperatures.

In analysing the BBR test results, we evaluate the degree of bitumen stiffness at a low temperature. Too-high bitumen stiffness at low temperatures is disadvantageous as it leads to cracking. The *Superpave* system assumes that creep stiffness  $S(t)$  may not exceed 300 MPa, which should ensure the appropriate cracking resistance (no binder over-stiffness). The value of parameter  $m$  should in turn be greater than 0.300, which is related to the fact that bitumens with a high parameter  $m$  value demonstrate a more effective relaxation of stresses present in the binder when temperatures drop [15].

Table 8.3 presents low-temperature property testing results for ORLEN Asphalt binders, with the test carried out by the Bending Beam Rheometer (BBR), and the samples aged in RTFOT and PAV. Test parameters:

- Testing at four temperatures: -10, -16, -22, -28°C.
- Sample temperature control time: 60 min.
- Values recorded after 60 s of loading:  $S(60s)$  MPa,  $m(60s)$

**Table 8.3.** Results of the BRR low-temperature test after ageing (RTFOT+PAV) (example critical temperatures at  $S(60) = 300$  MPa,  $m(60) = 0.3$  and bitumen stiffness  $S$  at  $-16^{\circ}\text{C}$ )

| Bitumen type                  | Dolna temperatura krytyczna  |  | Bitumen stiffness at $-16^{\circ}\text{C}$<br>$S(T)_{-16}$ [MPa] |
|-------------------------------|--|--|--|
|                               | Critical temperature at<br>$S(60) = 300$ MPa<br>$T(S)_{60}$ [ $^{\circ}\text{C}$ ] | Critical temperature at<br>$m(60) = 0,3$<br>$T(m)_{60}$ [ $^{\circ}\text{C}$ ] |  |
|                               | EN 14771<br>AASHTO PP 42   | EN 14771<br>AASHTO PP 42   | EN 14771<br>AASHTO PP 42   |
|                               | less = better  | less = better  | less = better  |
| Paving-grade 20/30            | -14.7  | -8.1   | 370.5  |
| Paving-grade 35/50            | -15.4  | -11.5  | 338.5  |
| Paving-grade 50/70            | -16.6  | -15.0  | 294.0  |
| Paving-grade 70/100           | -16.9  | -16.2  | 285.0  |
| Modified ORBITON 10/40-65     | -17.2  | -8.6   | 271.5  |
| Modified ORBITON 25/55-60     | -16.9  | -13.8  | 278.0  |
| Modified ORBITON 45/80-55     | -18.1  | -16.9  | 242.0  |
| Modified ORBITON 45/80-55 EXP | -17.9  | -18.0  | 242.3  |
| Modified ORBITON 45/80-65     | -18.3  | -14.3  | 235.0  |
| Modified ORBITON 65/105-60    | -20.5  | -20.6  | 172.3  |

Certain types of road binders demonstrate too high creep stiffness values  $S(t)$  or too small parameter  $m$  during the BBR test, and yet are resistant to low-temperature cracking. Therefore, the BBR test alone is not the ultimate measure of binder suitability at low temperatures. To determine that, the Direct Tension Test set was developed, determining the binder's capacity for elongation. No laboratory in Poland possesses that piece of equipment. It is also rarely found in Europe.

### 8.3. Testing of properties at high and intermediate temperatures

This type of test is to determine the binder's capacity to counteract viscoplastic deformations in the pavement. The DSR (dynamic shear rheometer) is used for the test, along with the method AASHTO M 320 or ASTM D7175.

The DSR test specifies high-temperature resistance:

- complex stiffness modulus  $G^*$  and angle phase  $\delta$  of the bitumen prior to RTFOT,
- complex stiffness module  $G^*$  and angle phase  $\delta$  of the bitumen after RTFOT.

It is required that bitumens demonstrate specific parameters tested in the DSR at its specific, maximum pavement service temperature (so-called high PG).

- $G^*/\sin\delta \geq 1.00$  kPa (bitumen before RTFOT)
- $G^*/\sin\delta \geq 2.20$  kPa (bitumen after RTFOT)

Another pavement damage mechanism is fatigue. This test, which checks binder resistance to fatigue cracks, also uses the DSR. The test is conducted at an intermediate temperature (depending on the PG type) and is intended to verify whether the complex modulus  $G^*$  is not too high, and therefore whether the pavement is not too stiff. The requirements put a limit on stiffness  $G^* \cdot \sin\delta$ , with the maximum value being 5 000 kPa (the newer version of the PG system raises this requirement to 6 000 kPa – see Section 8.4).

Table 8.4 presents the DSR test results for the relevant properties. Test parameters:

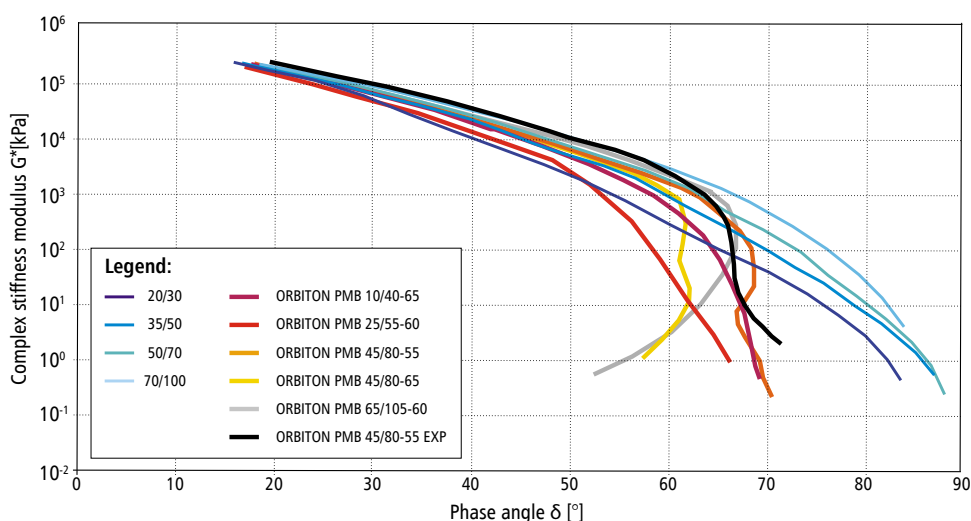
- complex stiffness modulus  $G^*$  and angle phase  $\delta$  of the bitumen prior to RTFOT to determine critical temperature at  $G^*/\sin\delta=1$  kPa,
- complex stiffness modulus  $G^*$  and angle phase  $\delta$  of the bitumen after RTFOT to determine critical temperature at  $G^*/\sin\delta=2.2$  kPa,
- complex stiffness modulus  $G^*$  and angle phase  $\delta$  of the bitumen after RTFOT+PAV to determine critical temperature at  $G^* \cdot \sin\delta=5\ 000$  kPa,

**Table 8.4.** DSR test results for the relevant properties. Bitumen testing values from 2009–2013

| Type of paving-grade bitumen type | Critical temperature at $G^*/\sin\delta=1$ kPa bitumen before ageing | Critical temperature at $G^*/\sin\delta=2.2$ kPa bitumen after RTFOT | Critical temperature at $G^* \cdot \sin\delta=5\ 000$ kPa bitumen after RTFOT+PAV |
|-----------------------------------|--|--|---|
|                                   | AASHTO T 315   | AASHTO T 315   | AASHTO T 315  |
|                                   | more = better  | more = better  | less = better   |
| Paving-grade 20/30                | 83.7   | 84.7   | 26.0  |
| Paving-grade 35/50                | 73.2   | 74.2   | 23.1  |
| Paving-grade 50/70                | 67.7   | 67.8   | 20.5  |
| Paving-grade 70/100               | 63.4   | 63.6   | 19.1  |
| Modified ORBITON 10/40-65         | 88.5   | 83.8   | 19.5  |
| Modified ORBITON 25/55-60         | 83.1   | 80.5   | 22.0  |
| Modified ORBITON 45/80-55         | 74.5   | 72.9   | 17.7  |
| Modified ORBITON 45/80-55 EXP     | 74.7   | 70.6   | 14.4  |
| Modified ORBITON 45/80-65         | 83.2   | 77.7   | 17.6  |
| Modified ORBITON 65/105-60        | 74.9   | 69.2   | 13.6  |

Figure 8.1 shows the Black curves for the tested bitumen types. They are used to evaluate the dependence of the binder's complex stiffness modulus  $G^*$  on angle phase  $\delta$ , and in particular to check the behaviour of the binder in the range  $\delta > 60^\circ$  that is the range of viscous material behaviour.

Figures 8.2–8.3 present master curves of the complex stiffness modulus  $G^*$  and angle phase  $\delta$  depending on frequency. The test was conducted in the frequency range of 0.1–10 Hz for -10, 0, 10, 25, 40, 60, 70°C, and then, using the temperature and frequency superposition, master curves for 25°C were obtained.



**Figure 8.1.** Black curve for bitumen before ageing

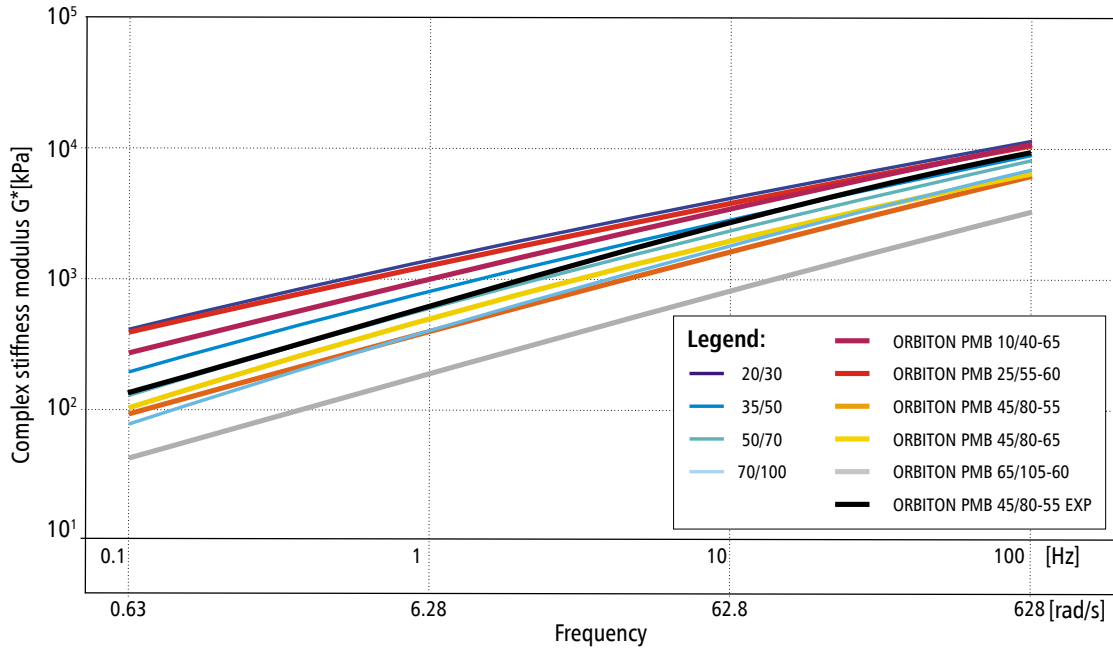


Figure 8.2. Master curve of the complex modulus  $G^*$  depending on frequency for bitumen before ageing. Sweep in the frequency range from 0.1 to 10 Hz, superposition to 25°C

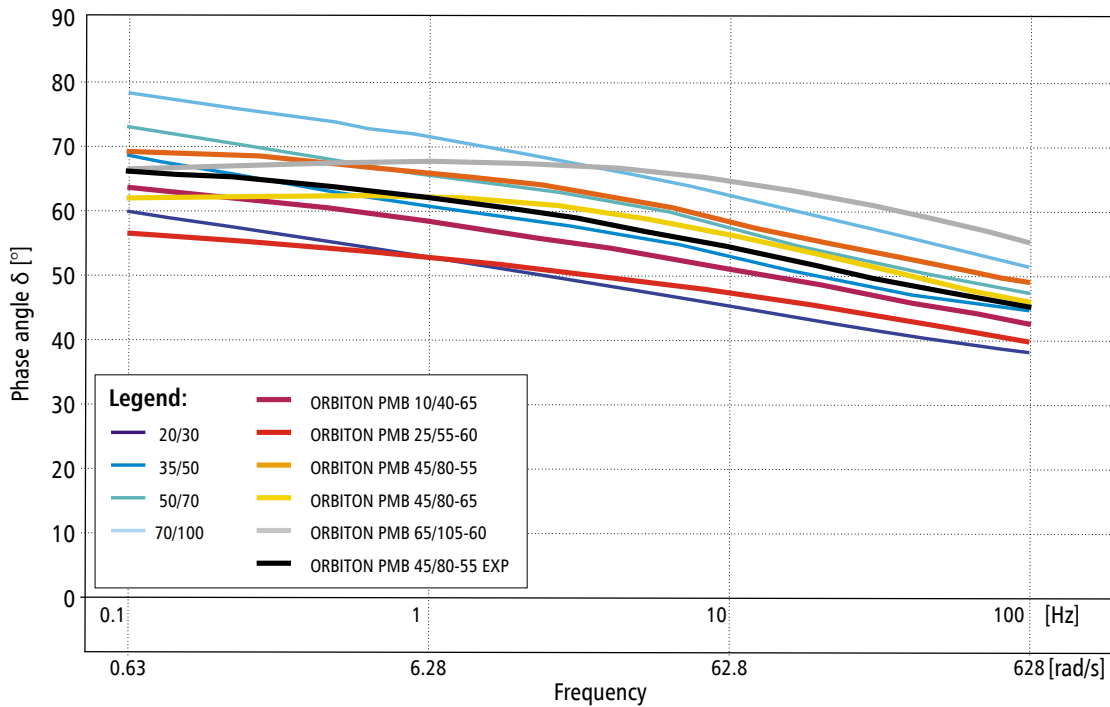


Figure 8.3. Master curve for phase angle  $\delta$  as a function of frequency for bitumen before ageing. Sweep in the frequency range from 0.1 to 10 Hz, superposition to 25°C

## 8.4. PG system adjustment – MSCR test

As indicated in Section 8.3., the results of critical temperature tests with parameter  $G^*/\sin\delta \geq 1$  kPa for bitumen before ageing and  $G^*/\sin\delta \geq 2.2$  kPa for bitumen after RTFOT are to indicate bitumen rutting resistance. Currently, however, this relationship has been challenged and the PG system is adjusted based on the MSCR test, gradually coming into use in the US since 2010.

The **MSCR (Multiple Stress Creep Recovery test)** involves the measurement of certain binder properties in order to determine (among other things) the resistance of asphalt with the binder to rutting.

The MSCR test is conducted according to the following standards: AASHTO TP 70 *Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)* and ASTM D7405 *Standard Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer*.

The test is to replace additional tests of modified bitumen specified in the so-called PG “plus”: elastic recovery, force ductility, toughness and tenacity.

The following mechanisms are examined in the course of the MSCR:

- binder sample creep mechanism – during the 1-second stress application,
- binder sample recovery mechanism – during the 9-second rest period (after the stress is removed).

The test is conducted for two stress values: 0.1 kPa and 3.2 kPa, typically at high limit temperatures at which the pavement with the tested binder is to operate. In effect, two pairs of results are obtained: non-recoverable creep compliance  $J_{nr}$  [ $\text{kPa}^{-1}$ ] and the average percentage deformation  $R$  [%] for two stress values (0.1 kPa and 3.2 kPa).

Of those parameters,  $J_{nr,3.2}$  kPa is crucial for binder classification, as it is the measure of binder resistance to deformation – the smaller  $J_{nr,3.2}$  kPa, the greater rutting resistance.  $R_{3.2}$  recovery, in turn, indicates the effectiveness of binder modification (if any).

Two indicators are calculated from the results of  $J_{nr,0.1}$  kPa,  $J_{nr,3.2}$  kPa,  $R_{0.1}$  and  $R_{3.2}$ :

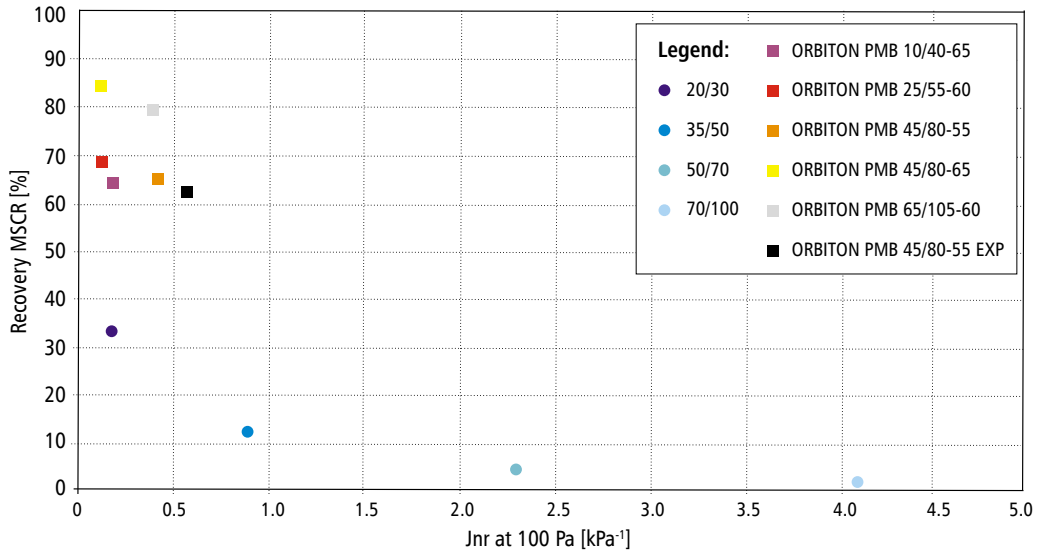
- $J_{nr,diff}$  – percentage indicator of the difference in  $J_{nr}$  after the change (increase) in the stress from 0.1 to 3.2 kPa – it is a measure of binder sensitivity to load increase; the increase must not be greater than 75%,
- $R_{diff}$  – percentage indicator of the difference in elastic recovery after the change (increase) in the stress from 0.1 to 3.2 kPa – it is a measure of binder elasticity under load increase conditions.

The American tests have specified experimentally the line separating modified bitumens from non-modified ones or, in other words – effectively modified bitumens from non-modified bitumens. The line is shown in Figure 8.5.

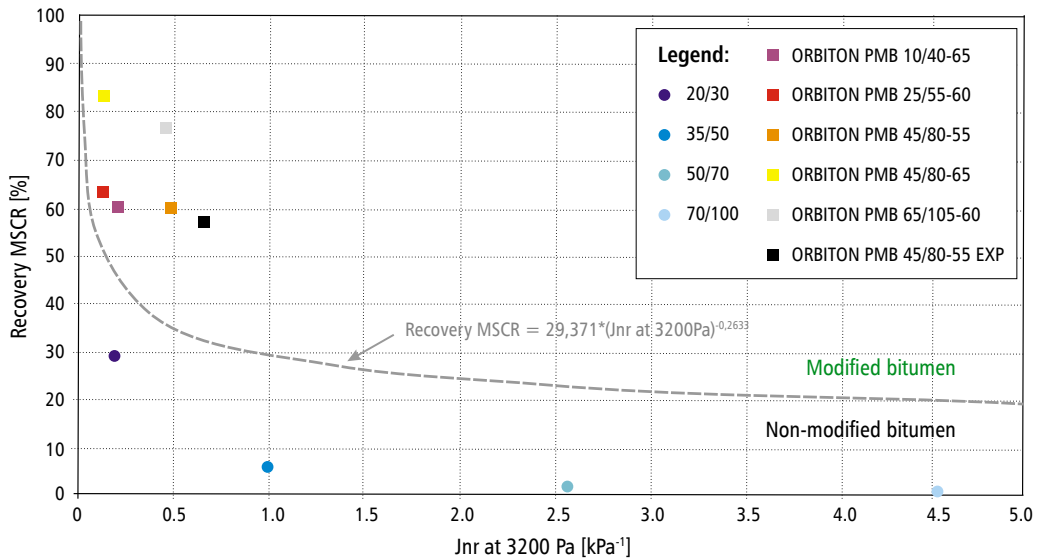
In planning binder testing using the MSCR, it was assumed that maximum pavement temperatures in Poland do not exceed 55–60°C, and therefore all binders were tested at 64°C. Some binders were also tested at 58 and 70°C in order to examine how their behaviour changes with the change of temperature.

Figures 8.4 and 8.5 present test results for various bitumens manufactured by ORLEN Asphalt and tested by MSCR at 64°C. Figure 8.5 shows the line separating modified bitumens (e.g. binders which meet the requirements for modified bitumens in terms of recovery  $R_{3.2}$  correlated with  $J_{nr,3.2}$  kPa range).





**Figure 8.4.** Presentation of bitumen results on the MSCR chart: elastic deformation R as a function of  $J_{nr}$  at the load of 0.1 kPa, at 64°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder)



**Figure 8.5.** Presentation of bitumen results on the MSCR chart: elastic deformation R as a function of  $J_{nr}$  at the load of 3.2 kPa, at 64°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder)

In the classical PG system, the results of parameter  $G^*\sin\delta \leq 5\,000$  kPa critical temperature refer to pavement (with the tested binder) fatigue resistance after RTFOT + PAV. In the new "PG System" specification, the value is increased to 6 000 kPa.

Since the very determination of the high PG limit does not guarantee that the pavement will not rut, the latest PG system, based on the MSCR, introduces additional binder values, depending on the road traffic volume (Table 8.5) on a given pavement. Suitability for a given traffic volume is assessed on the basis of  $J_{Nr3.2}$ .

**Table 8.5.** Binder designations and requirements relative to traffic volume and characteristics

| Traffic designation   | Loading (number of standard equivalent axles and traffic conditions) | Required for the binder at the high PG temperature |   |
|---|--|--|---|
|   |  | Requirement for $J_{nr,32}$                        | Stress sensitivity parameter for $J_{nr, diff}$ (*) |
| S – standard  | < 10 million axles and standard traffic                              | $\leq 4.0$   | $\leq 75\%$   |
| H – heavy   | 10-30 million axles or slow traffic                                  | $\leq 2.0$   |   |
| V – very heavy  | > 30 million axles or vehicle parking                                | $\leq 1.0$   |   |
| E – extreme   | > 30 million axles and vehicle parking                               | $\leq 0.5$   |   |
| $J_{nr, diff} = \frac{J_{nr,3.2 kPa} - J_{nr,0.1 kPa}}{J_{nr,0.1 kPa}} \cdot 100$ |  |  |   |
| *) bitumen sensitivity to stress change   |  |  |   |

## 8.5. Classification of ORLEN Asphalt bitumens acc. to PG System

Paving-grade bitumen grades acc. to the Superpave system are presented in table 8.6.

**Table 8.6.** Paving-grade bitumen classification acc. to Superpave (testing of bitumen samples from 2010–2012)

| Bitumen type                  | Actual PG grade (direct test results) | PG grade AASHTO MP 1 |
|-------------------------------|---------------------------------------|----------------------|
| Paving-grade 20/30            | 84-18                                 | 82-16                |
| Paving-grade 35/50            | 74-21                                 | 70-16                |
| Paving-grade 50/70            | 67-25                                 | 64-22                |
| Paving-grade 70/100           | 63-26                                 | 58-22                |
| Modified ORBITON 10/40-65     | 83-18                                 | 82-16                |
| Modified ORBITON 25/55-60     | 80-23                                 | 76-22                |
| Modified ORBITON 45/80-55     | 72-26                                 | 70-22                |
| Modified ORBITON 45/80-55 EXP | 70-27                                 | 70-22                |
| Modified ORBITON 45/80-65     | 77-24                                 | 76-22                |
| Modified ORBITON 65/105-60    | 69-30                                 | 64-28                |

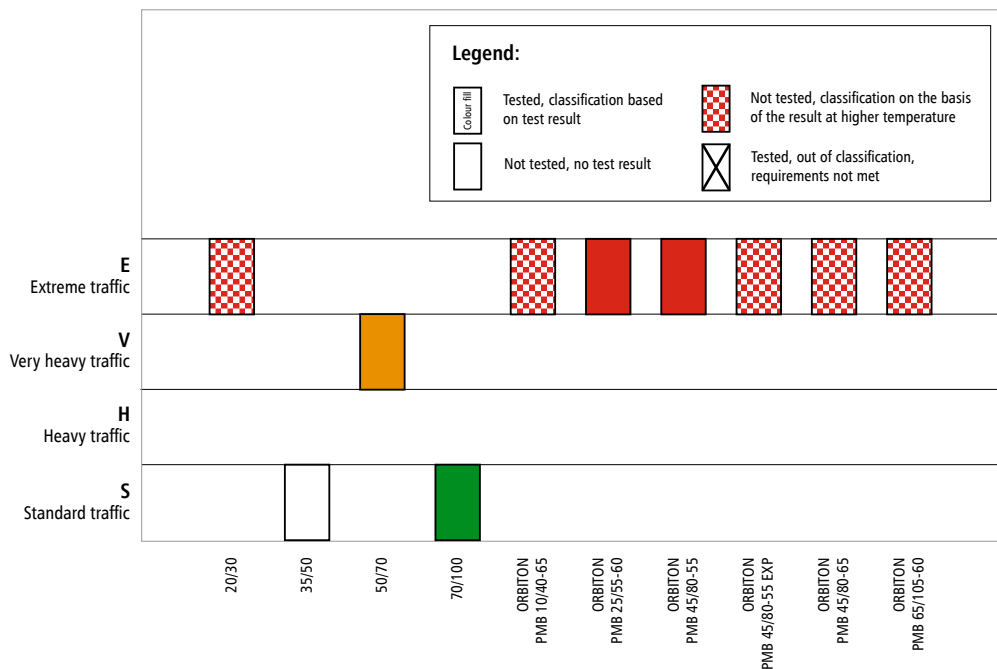
Table 8.7 shows classification results of all bitumens with reference to traffic load at the “high PG” temperature: 58°C and 64°C on the basis of  $J_{nr,3.2}$  kPa with MSCR.

**Table 8.7.** Binder classification after the MSCR test by traffic load (testing of bitumens from 2012) on the basis of ranges from Table 8.5

| Bitumen type                  | Traffic classification for temperature |      |
|-------------------------------|--|------|
|                               | 58°C                                   | 64°C |
| Paving-grade 20/30            | (E)                                    | E    |
| Paving-grade 35/50            | No data                                | V    |
| Paving-grade 50/70            | V                                      | S    |
| Paving-grade 70/100           | S                                      | *    |
| Modified ORBITON 10/40-65     | (E)                                    | E    |
| Modified ORBITON 25/55-60     | E                                      | E    |
| Modified ORBITON 45/80-55     | E                                      | E    |
| Modified ORBITON 45/80-55 EXP | (E)                                    | V    |
| Modified ORBITON 45/80-65     | (E)                                    | E    |
| Modified ORBITON 65/105-60    | (E)                                    | E    |

classification in parentheses on the basis of the result at a higher test temperature  
 \* outside classification, requirements not met  
 S – standard  
 H – heavy  
 V – very heavy  
 E – extreme

The results should be analysed with a proviso that they are from example tests and do not represent typical values achieved over the entire (and each) production season. Obviously, the values are not guaranteed by ORLEN Asphalt sp. z o.o.



**Figure 8.6.** Schematic presentation of binder classification by traffic load at 58°C, on the basis of details from Table 8.7

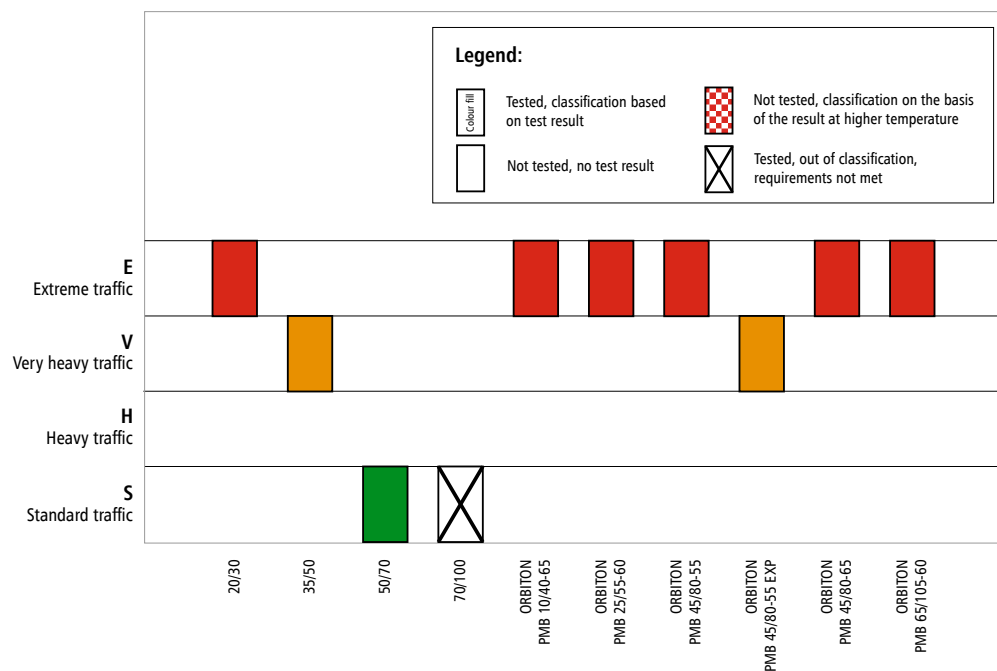


Figure 8.7. Schematic presentation of binder classification by traffic load at 64°C, on the basis of details from Table 8.7



## Chapter 9

# BITUMEN APPLICATION TECHNOLOGY

The application of bituminous binders requires, most of all, expertise on optimum process temperatures and particular criteria for bitumen sample handling. Subsequent sections provide details which may be of interest to laboratory staff and process/technology divisions of road engineering companies.

Table 9.2 summarises all key information about process temperatures for the application of bitumen manufactured by ORLEN Asfalt.

### 9.1. Laboratory guidelines

#### 9.1.1. Determination of process temperatures

Bitumen types differ from one another in terms of their characteristic viscosities within 60–165°C temperature brackets (most commonly tested temperature range). Viscosity values for refinery-produced, non-aged bitumen will always differ from viscosity values after ageing. After ageing, bitumen hardens and its viscosity increases. The simulation of short-term ageing under laboratory conditions takes place in the RTFOT device, and in the PAV vessel for long-term ageing simulation.

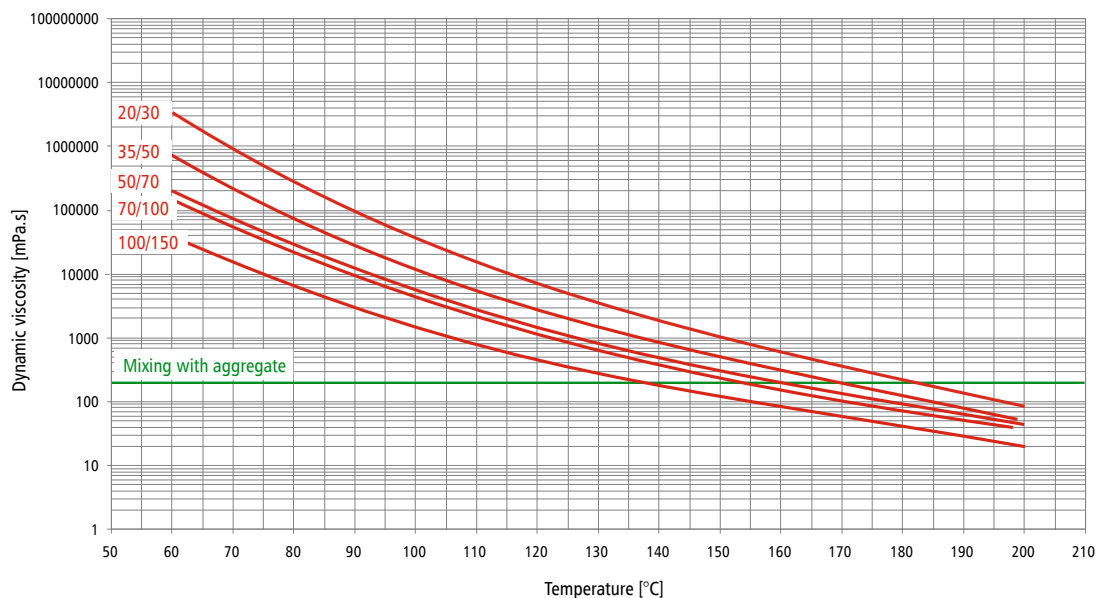
Understandably, post-RTFOT viscosity-temperature relation curve does not overlap with the characteristic curve for non-aged bitumen and is shifted towards higher viscosity ranges. This means that process temperatures should be determined on the basis of bitumen viscosity testing, both before and after RTFOT.

Optimum viscosity, or viscosity range, for the majority of key processes is already known, and serves as the basis for the determination of optimum process temperatures.

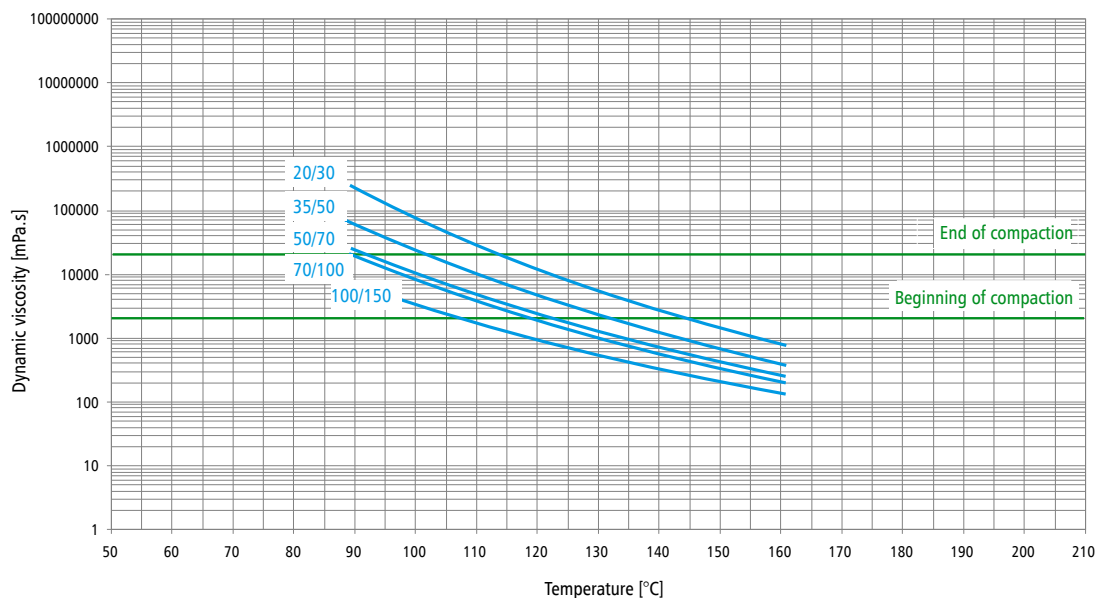
In order to determine the pumping and aggregate mixing temperatures, bitumen test results *before ageing* are used, since those processes occur before the contact of a thin binder layer with hot aggregate surface (before the main short-term ageing process starts). In order to determine the start and end temperature of onsite asphalt mixture compaction, viscosity values obtained *after ageing* should rather be used (RTFOT method). In the actual asphalt mixture production process, “wet” mixing of components (aggregate and bitumen) is followed by hot mix storage in the silo and its transport to the construction site. This stage typically lasts from a few dozen minutes to a few hours. Over that time, bitumen spreads over hot aggregate and ages – lighter components evaporate and, in effect, the bitumen hardens. Concurrently, its penetration drops, its softening point and viscosity rises, and the breaking point deteriorates. When the mixture spreading and compaction starts, the binder in the mix has already undergone short-term ageing. Therefore we suggest taking the viscosity value measured after RTFOT to determine the compaction start and end temperatures.

Figure 9.1 shows paving-grade bitumen viscosity curves before ageing which help to determine pumping and aggregate and binder mixing temperatures. Figure 9.2 presents paving-grade bitumen post-ageing viscosity curves which are useful for the determination of compaction start and end temperatures. Those curves are provided in bitumen type sheets included in Chapters 4 and 5.

Since bitumen viscosities largely depend on raw material properties (crude oil distillation vacuum residue), it should be assumed that the process temperature to be determined may fluctuate over the production season, depending on the actual raw material properties.



**Figure 9.1.** Paving-grade bitumen viscosity curves before ageing (on the basis of test results obtained by ORLEN Asphalt sp. z o.o.)



**Figure 9.2.** Paving-grade bitumen viscosity curves after RTFOT (on the basis of test results obtained by ORLEN Asphalt sp. z o.o.)

In examination of equivalent temperature values, particular attention should be paid to the appropriate selection of sample compaction temperatures at the laboratory (according to the method chosen from EN 13108-20). Asphalt mixture sample preparation temperatures should correspond to the actual conditions on the construction site. If the temperature adopted at the lab is too high, the volumetric density of the bituminous mixture in the samples will be high and the void content will be underrated. If the conditions on the site markedly

differ from those adopted at the lab, i.e. the asphalt mixture temperature in the course of layer compaction is significantly lower, this will in practical terms prevent the achievement of the required layer compaction index. Conversely, if the temperature adopted by the lab is too low, compaction indexes in excess of 100% will be achieved on the site, and void content in the layer will be too low, which will increase the risk of rutting. That is why adopting the appropriate sample compaction temperature at the laboratory mix design stage is so important.

### 9.1.2. Bitumen samples at the lab

The laboratory receives bituminous binder samples from ORLEN Asphalt in metal packaging (closed cans) or, in exceptional cases, in small cardboard containers lined with aluminium foil (volume of about 1 litre).

The way such bitumen is handled has a major effect on the test results of both bitumen and asphalt mixtures. It should be remembered that a bitumen sample which is heated and/or overheated in the oven multiple times may significantly harden.

Multiple heating of bitumen samples should therefore be avoided. We suggest using a greater number of small samples (for one-off use) rather than a single, large bitumen-holding container.

If it is necessary to use bitumen from one, large container, heating the container for the first time is recommended, its homogenisation through mixing, and subsequently pouring into a few smaller containers to be used later.

The handling of bitumen samples for tests is specified in EN 12594 *Bitumen and bituminous binders. Preparation of test samples*.

Sample heating at the laboratory according to the standard procedure:

- the container must not be tightly closed,
- under no circumstances should the samples be heated at the temperature exceeding 200°C,
- **containers with up to 1 litre in volume**, heating time of up to 2 hours, oven heating temperature: not more than bitumen softening point +100°C,
- **containers with 1–2 litres in volume**, heating time of up to 3 hours, oven heating temperature: not more than bitumen softening point +100°C,
- **containers with 2–3 litres in volume**, heating time of up to 3.5 hours, oven heating temperature: not more than bitumen softening point +100°C,
- **containers with 3–5 litres in volume**, heating time of up to 4 hours, oven heating temperature: not more than bitumen softening point +100°C,
- **containers with more than 5 litres in volume**, heating time of up to 12 hours, oven heating temperature: not more than bitumen softening point +50°C,

After samples are heated in the containers, they should be homogenised by mixing. The process should be careful not to introduce air bubbles into the sample. The maximum mixing (homogenisation) time is 10 minutes.

Bitumen samples obtained from the extraction of bituminous mixtures as per EN EN 12697-1, EN 12697-2, and EN 12697-4 should be tested promptly upon extraction in order to avoid reheating.

### 9.1.3. Bitumen adhesion to mineral aggregates

Bitumen adhesion to aggregate grain surfaces depends on a number of factors, including the type of rock used to produce the aggregate. In general terms, “acidic” and “alkaline” aggregates are used in road engineering, depending on high or low content of SiO<sub>2</sub> (silica) in the rock. The general rule is that “acidic” aggregates bear little affinity to bitumen and require the application of additives that improve bitumen adhesion. “Alkaline” aggregates, such as limestone, are marked by better adhesion. Before the application of adhesion promoters, however, they should be tested at the lab, because certain chemical agents cause bitumen ad aggregate bonding (adhesion) to deteriorate.

The current standards provide tools for the testing of bitumen adhesion to aggregate and, more broadly, bituminous mixture resistance to water and frost:

- PN-EN 12697-11 *Bituminous mixtures – Test methods for hot mix asphalt – Part 11: Determination of the affinity between aggregate and bitumen,*
- PN-EN 12697-12 *Bituminous mixtures – Test methods for hot mix asphalt – Part 12: Determination of the water sensitivity of bituminous specimens.*

In the case of poor affinity between bitumen and aggregate, adhesion promoters (agents) are used. Adhesion evaluation can follow e.g. the test described in EN 12697-11 on the selected aggregate mixture fraction.

Adhesion agents available on the market and their content in bitumen should be selected for a specific bitumen and aggregate from the mineral mixture, and it should be remembered that rarely do we encounter all-purpose products that perform well with each bitumen-aggregate combination.

The final check of the bituminous mixture resistance to water and frost is the ITSr test acc. to EN 12697-12.

## 9.2. Bitumen storage

Bituminous binders should be stored in tanks designed specifically for that purpose. Bitumen in the working tank should be heated indirectly, using the temperature control system, to ensure that the specified temperature with  $\pm 5^{\circ}\text{C}$  tolerance is retained. This means that the tank should be fitted with precision instrumentation systems with local or remote temperature reading, placed in the heating coil area and outside it, and be easily removable for regular cleaning. The requirements of the standard on the Factory Production Control of asphalt mixtures, namely EN 13108-21 *Bituminous mixtures – Requirements – Part 21: Factory Production Control*) provide that bitumen temperature should be recorded once per day.

Long-term storage of bitumen batches at temperatures close to the maximum storage temperature may cause deposit buildup at the bottom of the tanks after some time, formed by precipitation of the heaviest bitumen fractions (so-called coke). The harder the bitumen, the more likely it is that coke will build up, and therefore the tank should be periodically monitored for deposit buildup if paving-grade bitumen 20/30 and 35/50 is stored. If the tank is not cleaned, over time the deposit may get into the pipes, and block filters and pumps.





**Figure 9.3.** Modified-bitumen tanks, Płock site (photo by ORLEN Asphalt sp. z o.o.)

Paving-grade bitumen storage in a tank may also entail ageing caused by bitumen oxidation and evaporation of its lighter components. Bitumen ageing process in the tank is slow, because the contact area between bitumen and air is small. Nevertheless, storing a small quantity of bitumen in tanks at high temperatures may overheat the bitumen layer on the silo walls or heating coils. This causes an additional coke buildup at the bottom of the tank and deterioration of binder properties.

**Table 9.1.** Bitumen ageing in storage silos

| Reasons for bitumen ageing in the silo         | Ageing prevention factors   |
|--|---|
| Long-term bitumen storage at high temperatures | Bitumen storage at high temperatures over prolonged periods of time should be avoided. In the course of asphalt mixture production downtimes, it is recommended to reduce bitumen temperature in the tank down to the level that enables subsequent heating.  |
| Bitumen circulation                            | Bitumen circulation is commonly used to homogenise it in the silo. If bitumen is stored over a long time, circulation should be limited, or activated periodically only. Circulation is particularly useful for the storage of modified bitumen. Its application helps to achieve better binder homogeneity after a prolonged period of storage. The circulating bitumen return pipe inlet into the tank should be located below the upper surface of the liquid that the binder forms in the tank. |
| Tank structure                                 | The most desirable situation is when the ratio of bitumen surface and its volume in the tank is small, and that is why storage tanks for bitumen should be vertical, because then the silo height-to-diameter ratio is high.  |

### Storage of paving-grade bitumen

If it is necessary to store paving-grade bitumen 35/50 in the silo at a high temperature for over 10 days, either penetration at 25°C (EN 1426) or R&B softening point (EN 1427) should be determined to control the ageing rate.

## Storage of polymer modified bitumen

Direct use of ORBITON modified bitumen after delivery is recommended, without long-term storage in the silo. Long-term storage of modified bitumen in the silo at a high temperature should be avoided. If longer storage (in excess of 7 days) is required, the bitumen should be homogenised by closed-cycle mixing in one or multiple tanks. At least one of the silos should be fitted with an agitator. More details in this respect are provided in Chapter 5.

### Other recommendations

Before changing the type or grade of bitumen in the tank, it should be ensured that the tank is empty.

Different bitumen types should not be mixed, such as paving-grade bitumens with polymer-modified bitumens. The mixing would markedly downgrade the binder and pavement performance.

The mixing of bitumen of the same type, but different grades, such as 50/70 with 70/100 is at the sole responsibility of the contractor. The process requires an effective mixing system in the tank and laboratory control. Binders from different manufacturers should not be mixed.

Multiple heating and cooling cycles for both ORBITON modified bitumen and paving-grade bitumen are not recommended.

If bitumen is to be kept in the mixing plant tank over winter, the temperature in the tank should be reduced to ambient temperature. Bitumen can be stored for several months under such conditions. It should be remembered, however, that the heating of a few dozen tonnes of bitumen may be lengthy in spring and depend on the efficiency and structure of the tank heating system. Reheating polymer-modified bitumen may be even more troublesome. Binder properties must be tested in each case after heating.

Bitumen temperature in the course of storage should not exceed values indicated in Table 9.2.

## 9.3. Asphalt mixture production

The viscosity of bitumen supplied to the hot mix plant should be low enough to enable its unloading from the road tanker. Since bitumen viscosity is strictly related to its temperature, bitumen temperature in the road tanker should be monitored in transport in cooler seasons. It is assumed that the minimum pumping temperature is achieved if bitumen viscosity is about 2 Pa·s.

In the course of bitumen mixing with aggregate, ageing processes accelerate rapidly (a very thin layer of bitumen over aggregate, very high temperature and oxygen supply), therefore “wet” mixing time should be carefully selected.

The application for production of a binder which is too hot may have other adverse effects, notably in the case of SMA mix or PA production, which will run a higher risk of binder draindown. In such cases it is required to increase the content of the stabiliser (e.g. cellulose fibres) and check drainage using the Schellenberg method for increased production temperatures (description provided in EN 12697-18 *Bituminous mixtures. Test methods for hot mix asphalt – Part 18: Binder drainage*).

Overheating the asphalt mixture during production at the mixing plant will result in significant short-term ageing of the bitumen, and in consequence downgrade the performance of the bituminous pavement. For this reason, the maximum production temperature should never be exceeded, even to improve the workability and compatibility on the construction site.

Temperatures provided in Table 9.2 do not apply to asphalt mixtures supplemented by an agent for production and placement temperature reduction.

The storage period in the tank of a fresh mixture should not cool it down excessively, and depends on the following factors:

- mixture production temperature,
- mixture type and binder content and type (paving-grade bitumen, multigrade or modified bitumen),
- presence of additives such as stabilisers, modifiers or adhesion agents,
- tank condition and equipment (thermal insulation, heating systems),
- asphalt mixture quantity in the tank.

## 9.4. Asphalt mixture transport

Particular attention should be paid to whether the cargo compartment of the vehicles carrying the mixture to the construction site is clean (no residue of formerly carried mixture). Internal parts of the cargo compartment should be sprayed (not excessively) with a special agent to protect its walls and bottom against adhesion of the mixture. The only anti-adhesion agents to be used are those that do not produce an adverse effect on the bituminous binder. **Diesel oil or any other mineral oils or solvents must not be used for the cargo compartment spraying.**

Cargo compartments must be always covered by tarpaulin in the course of asphalt mixture transport. Whenever temperatures are low or other adverse weather factors operate, it is recommended to use vehicles with isolated cargo compartments. If it is necessary to work under adverse weather conditions (low temperature, strong wind, long travel distance), the use of intermediate equipment with an additional mixer and mixture heating (MTV, shuttle buggy) between the paver and the mixture unloading vehicle should be considered. The transport work should be arranged so as to ensure the continuity of deliveries to the construction site (no paver stops).

Upon loading of the asphalt mixture on the vehicle, its temperature should be inspected and visual assessment performed. The following points should be considered [4]:

- **blue smoke** – raising over the mixture – indicates its excessive overheating in the course of mixing with aggregate (over 200°C). The mixture is essentially destroyed (overburnt) and will ravel after placement and fail to demonstrate resistance to water and frost,
- **mixture flows** in the cargo compartment – possible reasons:
  - a. bitumen feeder damage or too high bitumen content,
  - b. incorrect content of the mineral mix – either fraction missing, even if the bitumen content is correct,
  - c. incorrect recipe of the asphalt mixtures – the laboratory design originally envisaged too much bitumen,
  - d. adhesion agent overdose,
- **after loading, the mixture forms a sharp cone, is matt and shows no gloss** – this may testify to the mixture's temperature being too low, or to bitumen content being too small; in effect, the mixture may not have the required workability and compatibility on the site; typically the mixture should form a dome-like shape after loading,

- aggregate is not entirely covered in bitumen – possible reasons:
  - a. too little bitumen in the mix (design flaw),
  - b. bitumen feeder damage,
  - c. too low bitumen temperature in the course of mixing with aggregate,
  - d. “wet” mixing time in the mixing plant too short.

## 9.5. Placement

Asphalt concrete mixes with high stiffness modulus (AC WMS or AC EME, depending on the country) combined with hard bitumens should have the thickest-permitted, in process and design terms, layer placed. This will improve the temperature aspect for compaction.

When mixtures are placed on the base having an increased temperature (just-placed courses), the temperature at mid-thickness of the placed layer should be carefully controlled. Non-contact thermometers are not recommended, unlike thermometers with a steel spindle allowing for immersion into the layer. If the temperature of the placed mix is very high (mixture cools down very slowly), rolling should not commence until the temperature drops to the point enabling the compaction to proceed. A similar procedure applies if the mixture is placed on a hot base (previous course still hot). The above guidelines do not apply to the *Kompaktasphalt* technology.

Mastic asphalt mix should not be placed manually due to its high viscosity. Mechanical equipment for mixture placement is recommended, along with additives to reduce the placement temperature.

## 9.6. Roadbase preparation

One of the preconditions for stable bonding of bituminous layers is good preparation of the roadbase: cleaning and bitumen spraying (typically emulsified bitumen). If there are oil stains on the surface of the course they should be removed with sorbent.

By their very name, sorbents are substances capable of absorbing other substances. They have found their application in road engineering, helping to clean oil or fuel spills on the surface of a course/subgrade. It is important to remember that quick removal of such stains from the road is one of the preconditions for future stability of the bituminous pavement. Oil-based substances dissolve bitumen and penetrate into deeper courses, causing permanent damage.

Sand or sawdust was formerly used to remove oil-based substances from the surface. Although sawdust (cellulose sorbents: wood, paper) sorbents can be used to absorb oil spills, their disadvantage is low density – they are relatively light, so their application is limited to windless weather due to their sensitivity to wind gusts. Importantly, sorbents of this type also absorb water. There are also processed cellulose sorbents on the market which no longer absorb water.

The following factor in the selection of a specific solvent:

- quick absorption and high absorption rate,
- no negative effect on the bituminous pavement,
- versatility,
- all-weather applicability:
  - hydrophobic properties (rain, snow)
  - sufficient weight (wind insensitive)
- easy to remove after application (no slime is formed),
- antislip properties where possible.

It must also be remembered that waste is generated after sorbent application to remove oil or fuel spills from the road, which must be handled in accordance with the regulations on waste. This involves both transport and proper recovery or disposal of a given waste type, depending on the type of sorbent used and the absorbed substance.

## 9.7. Process temperatures

**Table 9.2.** Minimum and maximum temperature of bitumen and bituminous mixtures depending on bitumen type

| Bitumen type  | Paving-grade bitumen |                    |                    | Polymer-modified bitumen |                  |                  |                      |                   |                   |
|---|----------------------|--------------------|--------------------|--------------------------|------------------|------------------|----------------------|-------------------|-------------------|
|   | EN 12591, NA         |                    |                    | EN 14023:2011, NA        |                  |                  | EN 14023:2010        | EN 14023:2011, NA |                   |
|   | Bitumen 20/30        | Bitumen 35/50      | Bitumen 50/70      | ORBITON 10/40-65         | ORBITON 25/55-60 | ORBITON 45/80-55 | ORBITON 45/80-55 EXP | ORBITON 45/80-65  | ORBITON 65/105-60 |
|   | Temperature [°C]     |                    |                    |                          |                  |                  |                      |                   |                   |
| <b>Laboratory</b>   |                      |                    |                    |                          |                  |                  |                      |                   |                   |
| Marshall sample compaction temperature/gyratory press   | 155-160              | 140-145            | 135-140            | 150-155                  | 145-150          | 145-150          | 150-155              | 150-155           | 145-150           |
| <b>Component temperature at the mixing plant</b>  |                      |                    |                    |                          |                  |                  |                      |                   |                   |
| Bitumen pumping   | over 140°C           | over 130°C         | over 130°C         | over 150°C               | over 150°C       | over 150°C       | over 150°C           | over 50°C         | over 150°C        |
| Short-term bitumen storage at the mixing plant  | up to 185            | up to 185 (200***) | up to 185 (200***) | up to 185                | up to 185        | up to 185        | up to 185            | up to 185         | up to 185         |
| Aggregate temperature during MMA production (above MMA production temperature)  | max. 30              | max. 30            | max. 30            | max. 30                  | max. 30          | max. 30          | max. 30              | max. 30           | max. 30           |
| <b>Ready bituminous mixture temperature in the mixing plant's mixer:</b>  |                      |                    |                    |                          |                  |                  |                      |                   |                   |
| Asphalt concrete  | max. 185             | max. 180           | max. 175           | max. 185                 | max. 185         | max. 185         | max. 185             | max. 185          | max. 185          |
| SMA   | –                    | –                  | max. 175           | –                        | max. 185         | max. 185         | max. 185             | max. 185          | max. 185          |
| Porous concrete   | –                    | –                  | –                  | –                        | –                | max. 185         | max. 185             | max. 185          | max. 185          |
| Mastic asphalt  | <230*)               | <230*)             | –                  | <230**)                  | <230**)          | –                | –                    | –                 | –                 |
| <b>Temperature on site</b>  |                      |                    |                    |                          |                  |                  |                      |                   |                   |
| Minimum temperature of the supplied bituminous mixture in the spreader's hopper   | 150                  | 145                | 140                | 160                      | 150              | 150              | 160                  | 155               | 150               |
| End of effective compaction temperature   | >120                 | >115               | >110               | >125                     | >120             | >120             | >120                 | >125              | >120              |
| *) mastic asphalt residence time in the bitumen boiler of up to 12 h at the specified temperature; higher temperature of mastic asphalt, up to 250°C, is permitted if boiler residence time does not exceed 5 h<br>**) mastic asphalt residence time in the bitumen boiler of up to 8 h, at the specified temperature; higher temperature of mastic asphalt, up to 250°C, is permitted if boiler residence time does not exceed 4 h<br>***) maximum temperature in the silo equal to 200°C only in exceptional cases of the delivery of bitumen at such temperatures from the refinery. |                      |                    |                    |                          |                  |                  |                      |                   |                   |

## Chapter 10

### OTHER BITUMEN PROPERTIES

#### 10.1. Bitumen density

Bitumen density is determined according to EN 15326 or EN ISO 3838.

Laboratories cooperating with ORLEN Asphalt typically determine density of all bitumens at 15°C two times per year. Current results are available from the Technology, Research and Development Department (contact details on page four of this Handbook's cover) and published on the website [www.orlden-asfalt.pl](http://www.orlden-asfalt.pl), tab Technical Information/For laboratories.

Binder densities as indicated in Table 10.1 can be adopted for the design of asphalt mixtures.

**Table 10.1.** Results of bitumen density tests at 15°C in 2013

| Bitumen type                  | Density at 15°C<br>as per EN ISO 3838 or EN 15326<br>[Mg/m <sup>3</sup> ] |
|-------------------------------|---|
| Paving-grade 20/30            | 1.035   |
| Paving-grade 35/50            | 1.030   |
| Paving-grade 50/70            | 1.024   |
| Paving-grade 70/100           | 1.010   |
| Paving-grade 100/150          | 1.022   |
| Paving-grade 160/220          | 1.019   |
| Modified ORBITON 10/40-65     | 1.034   |
| Modified ORBITON 25/55-60     | 1.022   |
| Modified ORBITON 45/80-55     | 1.024   |
| Modified ORBITON 45/80-55 EXP | 1.024   |
| Modified ORBITON 45/80-65     | 1.030   |

Bitumen densities provided in Table 10.1, column 2, apply to tests at 15°C. If bitumens are to be used at other temperatures, the indicated density at 15°C should be converted into the density at the application temperature using the following equation:

$$\rho_x = \rho_{15} - (0,00061 \cdot \Delta t)$$

where:

$\rho_x$  – density at the application temperature X

$\rho_{15}$  – density at 15°C in Mg/m<sup>3</sup>

$\Delta t$  – temperature difference (X – 15), X ∈ {15,16...200}

## 10.2. Bitumen solubility

Various solvents can be used for the extraction of the asphalt mixture sample as per EN 12697-1. Table 10.2 provides bituminous binder solubility tests results as per EN 12592 for binders manufactured by ORLEN Asphalt. The results according to EN 12592 can be used to calculate "T" referred to in A.4, EN 12697-1.

**Table 10.2.** Bituminous binder solubility test results according to EN 12592 for binders manufactured by ORLEN Asphalt

| Bitumen type                   |              | Solubility in xylene<br>% m/m | Solubility<br>in tetrachloroethylene<br>% m/m | Solubility in toluene<br>% m/m |
|--------------------------------|--------------|-------------------------------|---|--------------------------------|
| Paving-grade<br>bitumen        | 35/50        | 99.8                          | 99.80   | 99.95                          |
|                                | 50/70        | 99.90                         | 99.85   | 99.90                          |
|                                | 70/100       | 99.95                         | 99.95   | 99.95                          |
| Modified<br>bitumen<br>ORBITON | 10/40-65     | 99.65                         | 99.60   | 99.90                          |
|                                | 25/55-60     | 99.85                         | 99.80   | 99.75                          |
|                                | 45/80-55     | 99.90                         | 99.90   | 99.90                          |
|                                | 45/80-55 EXP | 99.80                         | 99.90   | 99.90                          |
|                                | 45/80-65     | 99.65                         | 99.70   | 99.70                          |
|                                | 65/105-60    | 99.75                         | 99.65   | 99.70                          |

Samples tested by Soxhlet method demonstrated solubility at 100% m/m.

## 10.3. Data for mechanistic pavement design

Certain software packages for asphalt pavement dimensioning use primary binder parameters in order to calculate their stiffness modulus and then the modulus for the asphalt mix. Table 9.3 provides data about mean values of the primary parameters of bituminous binders manufactured by ORLEN Asphalt in 2012.

**Table 10.3.** Data for mechanistic pavement design

| Binder           | Penetration at 25°C<br>[0,1 mm] | R&B softening point<br>[°C] |
|------------------|---------------------------------|-----------------------------|
| ORBITON 25/55-60 | 40                              | 62                          |
| ORBITON 45/80-55 | 60                              | 57                          |
| 35/50            | 45                              | 54                          |
| 50/70            | 65                              | 48                          |

## Chapter 11

# QUALITY CONTROL OF BITUMEN DELIVERIES ACC. TO EN ISO 4259

### 11.1. Quality acceptance rules for deliveries acc. to EN ISO 4259

It does happen at times that there is a dispute between the supplier and recipient over the quality of the delivered bitumen. Let us consider the requirement for the Fraass breaking point " $T_{\text{Fraass}} \leq -18^{\circ}\text{C}$ ". Does it really mean that, when we receive a result from the recipient's laboratory showing that  $T_{\text{Fraass}} = -17^{\circ}\text{C}$ , the delivered product does not comply with the requirements? This question can be answered by the standard EN ISO 4259 "Petroleum products – Determination and application of precision data in relation to methods of test", designed specifically for that purpose. EN ISO 4259 is referenced in each bitumen standard – namely EN 12591, EN 14023 and EN 13924.

Below in this chapter we provide the explanation for the problem posed by the example from [4].

### 11.2. Determination of requirements

Typically, the specification provides a requirement such as "not less than (min)", or "not more than (max)", setting the limit for the tested property. We can say that there are two types of limits:

- **double limit** (upper and lower) – for instance penetration at  $25^{\circ}\text{C}$  from 10 to 50 [0.1 mm] or softening point  $T_{\text{PIK}}$  from 48 to  $52^{\circ}\text{C}$ ;
- **single limit** (upper or lower) – e.g. paraffin content not higher than 2.2% m/m; Importantly, there is sometimes an additional, apparent limit, e.g. in the case of solubility test with a single limit "not less than 99.0%", there will be a consequent, additional limit of 100% – in this case the single limit is transformed into a double limit.

The upper limit in EN ISO 4259 is designated as  $A_1$ , and the lower limit as  $A_2$ .

The standard also establishes a principle that, for the requirement in the Specification to be reasonable, it must consider reproducibility of the adopted property testing method.

Reproducibility (**R**) is the precision of an analytical method, whose measure is the consistency of results obtained by various contractors at various laboratories testing the same product using the same method. In other words, reproducibility helps to ascertain whether a given method produces the same results from tests of identical products, carried out at different laboratories.

Reproducibility is provided in each standard describing a specific test. For example, reproducibility for the softening point  $T_{\text{R&B}}$  of paving-grade bitumens as per EN 1427 is  $R=2.0^{\circ}\text{C}$ , and  $R=3.5^{\circ}\text{C}$  for modified bitumens.

Therefore, a requirement from the Specification is reasonable if:

- a specific range for a double limit ( $A_1$  and  $A_2$ ) is not smaller than the quadruple reproducibility **R**:

$$(A_1 - A_2) \geq 4 \cdot R$$



- a specific range for a single limit ( $A_1$  or  $A_2$ ) is not smaller than the double reproducibility  $R$ :

$$A_1 \geq 2 \cdot R \text{ or } A_2 \geq 2 \cdot R$$

If the Specification provides for requirements which fail to meet those conditions, the results will be uncertain and their significance in determining a sample's compliance will be doubtful.

If the condition  $(A_1 - A_2) \geq 4 \cdot R$  is not met, then the limits of the requirement should be extended or a testing method with higher precision should be selected.

### 11.3. Product evaluation by bitumen recipient

EN ISO 4259 describes the procedure for quality acceptance of bitumen deliveries. The actions to be carried out by the bitumen recipient are as follows. We are considering a case where the recipient received a single test result from its own laboratory. The recipient may argue that the delivered product **does not comply** with the requirement with 95% certainty only if the test result (here designated as  $Y$ ) is:

- for the single upper limit  $A_1$ :

$$Y > A_1 + 0.59 \cdot R$$

- for the single lower limit  $A_2$ :

$$Y < A_2 - 0.59 \cdot R$$

- for a double limit – one of the requirement should be met (one, because a result beyond the lower OR upper limit of the requirement range is typically challenged).

Example:

The supplier manufactures paving-grade bitumen 35/50 and delivers it to the recipient. The latter tests the delivery and obtains the result  $Pen_{25}=34$  [01 mm] (below designated as  $W_2$ ). Can the recipient consider the 35/50 bitumen compliant with EN 12591, or complain about the product ("penetration too low")? Standardised limits for 35/50 bitumen are  $A_2=35$  [0.1 mm] and  $A_1=50$  [0.1 mm], hence  $W_2$  is below the standard lower limit. The bitumen penetration test standard (EN 1426) specifies the method's reproducibility as <sup>1</sup>  $R=3$  [0.1 mm]. Has the supplier delivered bitumen compliant with the standard? Let us make the calculations:

$$35 - 0.59 \cdot 3 < W_2 < 50 + 0.59 \cdot 3$$

$$33.2 < W_2 < 51.8$$

In this case,  $W_2=34$  [0.1 mm] is within the limits of the specification, extended by the penetration test uncertainty. To reject the delivery, the recipient would have to argue that the result is smaller than 33.2 [0.1 mm] or greater than 51.8 [0.1 mm].

The bitumen recipient should be aware of receiving the product marked by an extended range (Figure 11.1), meaning a certain tolerance applies, resulting from the test method reproducibility. Therefore it is important to remember about the precision of the applied testing methods.

1) Reproducibility  $R=3$  [0.1 mm] is for bitumens with penetration  $<50$  [0.1 mm]

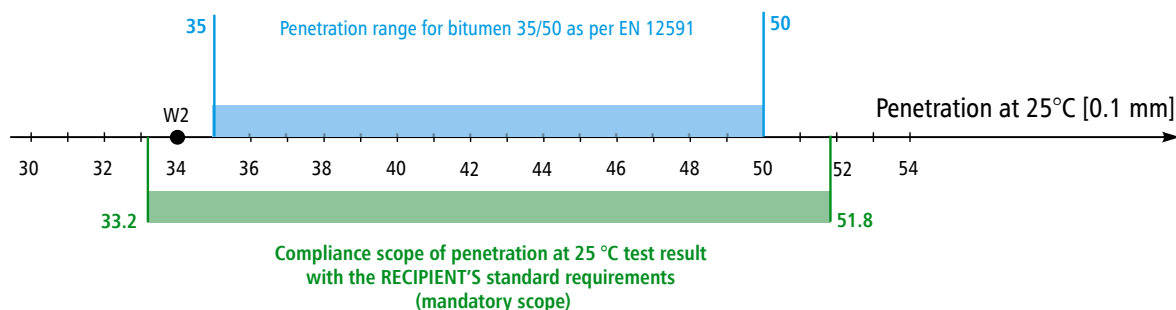


Figure 11.1. Example case – ranges of result compliance with the requirements at the supplier and recipient [4]

## 11.4. Disputes

If the recipient and the supplier are unable to agree on the quality of the delivered product, the procedure for accepting and rejecting results in the case of a dispute should be applied. This procedure is also described in EN ISO 4259 but will not be quoted here due to its considerable length.

## 11.5. Breaking point example

Let us return to the Fraass breaking point test, mentioned earlier in this chapter. The EN 12593 norm describing the testing for this bitumen property, sets its reproducibility at 6°C. If the upper limit is set at not higher than -18°C, then, applying the equation for the single limit, only results (W3) from the following range:

$$W3 > -18 + 0.59 \cdot 6$$

$$W3 > -14.5^{\circ}\text{C}$$

can be rejected. In other words: -15; -16; -17°C (below -14.5°C) should also be accepted as complying with the requirement  $\leq -18^{\circ}\text{C}$ . Surprising as this may appear, we have to remember that the Fraass test is a very low-precision test, hence the outcome.

## 11.6. Reproducibility values for selected bitumen properties

Table 11.1. provides reproducibility values for selected properties of bitumen and their determination methods.

Table 11.1. Reproducibility values for selected properties of bitumen and their determination methods

| Property                      | Test method   | Reproducibility R  |
|-------------------------------|---------------|--|
| Penetration at 25°C           | EN 1427:2007  | for penetration < 50 [0.1 mm] R=3<br>for penetration $\geq 50$ [0.1 mm] R=6% from the mean value |
| Ring and ball softening point | EN 1427:2007  | paving-grade bitumens R=2.0°C<br>modified bitumens R=3.5°C                                       |
| Fraass breaking point         | EN 12593:2007 | R=6°C  |



## Chapter 12

# OCCUPATIONAL SAFETY

### 12.1. Introduction

General aspects of occupational health, safety and environment discussed below apply to petroleum-derived bitumens used in road construction, manufactured by ORLEN Asphalt, whose standardised properties are elaborated on in Chapters 4 and 5 of this Guide.

Extensive ecological and toxicological information as well as hazard identification, conduct in the case of fire or inadvertent environmental release are all provided in MSDSs available from ORLEN Asphalt for all products.

Although bitumen is not classified as a hazardous substance, MSDSs for bitumen are broadly available for bitumen users in order to ensure maximum application safety and full product information.

The format and content of the MSDSs complies with EU regulations, namely the REACH regulation (*Registration Evaluation and Authorisation of Chemicals*) and the CLP regulation (*Classification, Labelling, Packaging*). Valid MSDSs for all bitumens manufactured by ORLEN Asphalt can be found on the company's website. This chapter discusses only some aspects of broadly understood HSE as applicable to working with bitumens. Comprehensive details are provided in the MSDSs referred to above.

Bitumen transport is governed by international rules on hazardous substance transport. **Bitumens are classified as hazardous due to their high temperature in transport.** The vast majority of ORLEN Asphalt's products is transported by road tankers. Road transport of dangerous goods in Europe is governed by international agreement abbreviated as ADR (*L'Accord européen relatif au transport international des marchandises Dangereuses par Route*), which introduces, among other things, specific marking for vehicles carrying bitumens.

It must be emphasised that the mixing of paving-grade bitumens with other substances or additives (outside the bitumen manufacturer's plant) should be considered for the identification of hazards and risk assessment. Such mixtures may generate additional hazards. However, it is the manufacturer of those mixtures that bears responsibility for the changes that may cause bitumen to become a substance which is dangerous for human health or environment.

Potential health hazards in the production, storage, transport and application of paving-grade bitumens are discussed below in this chapter.

### 12.2. Burns (skin, eye contact)

Paving-grade bitumen working temperature typically exceeds 100°C. Therefore, an important hazard which may occur while working with bitumens is thermal burns (up to and including third-degree burns). Burns may be sustained in various situations: in regular work (e.g. sampling, tanker unloading, maintenance work, etc.) as well as in emergencies, e.g. during an uncontrolled spill of hot bitumen as a result of tank integrity loss, or if shut-off valves work defectively.

Personal protective equipment must be used at all times when working with hot bitumen, such as e.g.:

- hard hat with face shield and neck protection. It should be remembered that safety glasses protect the eyes only!
- work clothing and footwear appropriate for high temperatures,
- heat-resistant safety gloves (it must be ensured that hot bitumen cannot get into the gloves!)



**Figure 12.1.** Hard hat with face shield – example  
(photo by H. Peciakowski)



**Figure 12.2.** Heat-resistant gloves with safety cuffs – example  
(photo by H. Peciakowski)

Procedure to be followed if burns are sustained:

- the burn should be immediately cooled down with cold, running water for at least 10 minutes,
- do not attempt to remove bitumen from the burn,
- medical assistance should be immediately sought in each case of heavy burns.

### 12.3. Fire

Paving-grade bitumens should not be stored at temperatures in excess of 220°C. Any handling should proceed at temperatures at least 30°C below the flash point. Importantly, flash point (Cleveland open cup method) of paving-grade bitumens discussed in this Handbook is over 300°C. The current bitumen standards do not require Pensky-Martens closed cup flash point testing, but it can be assumed to be lower than the open cup flash point.

If bitumen in the tank is overheated, flammable decomposition products are likely to occur, which increases the risk of fire, or even explosion. According to the chemical safety report prepared by CONCAWE (*Conservation Of Clean Air And Water In Europe*), bitumens as such are not considered explosive on the basis of structural considerations and oxygen balance [5]. In order to minimise the production of vapours, bitumen overheating should be avoided, because then bitumen loses the manufacturer-declared product properties. An important consideration for the operation of tanks is that deposits capable of self-combustion may build up on the walls and decks of tanks, and self-combust if oxygen is present.

The primary rule in the case of any fire is to use appropriate fire extinguishing agents. **Compact water streams directed at the surface of liquid bitumen must not be used for extinguishing bitumen fire** as it generates a hazard of abrupt splatters of hot bitumen. Water can only be used for cooling down hot surfaces.

Appropriate extinguishing agents include carbon dioxide, dry chemical, foam, and sand.

**Procedure to be followed in the case of bitumen fire:**

- immediately call the Fire Brigade,
- if there is no hazard to personal safety:
  - turn off bitumen heating,
  - shut off circulating pumps, etc.,
  - shut off the valves, which may contribute to limiting fire spread.

## 12.4. Foaming in the presence of water

Hot bitumen foams in contact with water as a result of an abrupt increase in volume (water turning into steam). It generates a real hazard of bitumen boiling over the tank or tanker. Bitumen foaming may be accompanied by hot bitumen splatters.

An important consideration for the loading procedure is to check whether the tanker contains water, and for the unloading operation – whether hoses do not contain water or moisture.

Bitumen storage tank should be dry at all times. An empty and cold tank should be initially filled with a small quantity of bitumen, so that to enable any potential moisture in the tank to evaporate slowly. Quick and careless filling of a cold, long-unused tank, as to which there is no certainty that it is dry, may cause bitumen to foam abruptly.

## 12.5. Bitumen vapours (bitumen mist, smoke)

Hot bitumen may emit vapours. For many years, the bitumen industry has been supporting and organising scientific research on the potential occupational hazards resulting from worker exposure to bitumen vapours. Research and production process monitoring still continues in Europe. If the process temperatures are strictly controlled so as to minimise bitumen vapour emission, and bitumen work site is open or well ventilated (working conditions control), it has not been found beyond a doubt that bitumen vapours represent a hazard for worker health (no sufficient evidence to confirm that).

It is recommended, in hot bitumen work, to avoid contact with the vapours and avoid inhaling vapours or mist from the hot product. Long-term exposure to high concentrations of vapours/smoke from hot bitumen may irritate the respiratory track or eyes, or even cause breathing problems or nausea. Therefore, emission of bitumen vapours should be minimised.

Worker exposure to bitumen vapours/smoke should be minimised through the application of the so-called best practices [11]:

- keep process temperatures as low as possible,
- work in well-ventilated areas,
- job rotation around the work site,
- use personal protective equipment, notably in confined spaces.

Whenever there are breathing problems caused by excessive inhalation of bitumen vapours:

- take the person suffering from breathing problems from the hazard area to fresh air,
- seek medical attention if problems with breathing persist.

### 12.5.1. Hydrogen sulphide

The elemental composition of bitumens varies depending on the chemical properties of petroleum used for their production and on the production methods [7]. For the majority of bitumens, however, the elemental composition also includes a small quantity of sulphur. Therefore, if hot bitumen is stored in closed tanks over a long time, hydrogen sulphide ( $H_2S$ ) may be released, whose concentration may reach dangerous levels. Opening of tanks with bitumen or entering empty bitumen tanks should be subject to particular safety procedures, complying with national and site regulations.

Conduct in the presence of hydrogen sulphide is governed by national safety regulations.

## LITERATURE

- [1] Anderson R.M.; Walker D.E.; Turner P.A. Low-temperature evaluation of Kentucky performance-graded 70-22 asphalt binders. Annual Meeting of the Transportation Research Board No 78 (01/1999) 1999, No 1661 pp. 69–74.
- [2] Bahia H. U. et al. Characterization of Modified Asphalt Binders in Superpave Mix Design. National Cooperative Highway Research Program 2001. REPORT 459. ISBN 0-309-06707-3
- [3] Bahia, H.U., and D.I. Hanson. "Survey Report of Modified Asphalt Binder Users, Producers, and Researchers," Project NCHRP 9-10 (Superpave Protocols for Modified Asphalt Binders), prepared for the National Cooperative Highway Research Program, Transportation Research Board, National Research Council, Washington, D.C. (February 1997).
- [4] Błażejowski K., Styk S. Technologia warstw asfaltowych (Bituminous course technology). Wydawnictwa Komunikacji i Łączności, 2004.
- [5] CONCAWE: Chemical Safety Report. Part B. "Bitumen" and "Oxidized Asphalt".
- [6] Development in Asphalt Binder Specifications. Transportation Research Circular E-C147. Transportation Research Board, December 2010, ISSN 0097-8515, pp. 39–40.
- [7] Encyklopedia fizyki (Encyclopedia of Physics). Państwowe Wydawnictwo Naukowe. 1972.
- [8] Gawel I., Kalabińska M., Piłat J. Asfalty drogowe (Paving-grade bitumens). Wydawnictwa Komunikacji i Łączności, 2001.
- [9] Gust J., Pawłowski K. Systemy oceny zgodności wyrobów budowlanych. Kryteria dobor systemu. (Construction product conformity assessment systems. System selection criteria) Warszawa-Miedzeszyn 2003.
- [10] <http://pl.wikipedia.org>
- [11] <http://www.eurobitume.eu/hse>
- [12] [http://www.ornen-asfalt.pl/informacje\\_technczne.php](http://www.ornen-asfalt.pl/informacje_technczne.php)
- [13] Kossowicz L., Polskie asfalty naftowe (Polish petroleum-based bitumens), Kraków 1968, Zjednoczenie Przemysłu Rafinerii Nafty.
- [14] Noor Zainab Habib, Ibrahim Kamaruddin, Madzlan Napiah and Isa Mohd Tan. Effect of thermoplastic copolymers on microstructure and viscoelastic behaviour of bitumen. Proceeding of Malaysian Universities Transportation Research Forum and Conferences 2010 (MUTRFC2010), 21 December 2010, Universiti Tenaga Nasional. ISBN 978-967-5770-08-1.
- [15] Physical differentiation between air-rectified and oxidised bitumens. Technical Committee Task Force. Eurobitume, 15.04.2011.
- [16] Pośniak M., Makhniashvili I., Kowalska J. (2000), Wielopierścieniowe węglowodory aromatyczne w procesach stosowania asfaltów (Polycyclic aromatic hydrocarbons in bitumen application processes), "BEZPIECZEŃSTWO PRACY nauka i praktyka" 7-8/2000, Centralny Instytut Ochrony Pracy.
- [17] Chemical Safety Report by CONCAWE. Part B. "Oxidised bitumen" and "Distilled bitumen".
- [18] Regulation of the Minister of Labour and Social Policy of 29 November 2002 on maximum permitted concentrations and rates of agents harmful to health in work environments.
- [19] Schramm G.: "Reologia. Podstawy i zastosowania" (Reology. Fundamentals and applications), Ośrodek Wydawnictw Naukowych, Poznań 1998.
- [20] Szadkowska-Stańczyk I., Analiza wyników badań epidemiologicznych dotyczących ryzyka nowotworowego u pracowników branży asfaltowej i przygotowanie opracowania uwzględniającego opublikowane w 2009 roku przez Międzynarodową Agencję Badań nad Rakiem (IARC) wyniki badań w zakresie tej problematyki (Analysis of epidemiological tests on cancer risk for bitumen industry workers and development of a study considering the related test results published by the International Agency for Research on Cancer in 2009), Instytut Medycyny Pracy, Łódź 2010.
- [21] Tymczasowe Wytyczne Techniczne TWT-PAD. (Interim Technical Guidelines on Polymer-Modified Paving-Grade Bitumens) Instytut Badawczy Dróg i Mostów 1993-1997-2003.
- [22] West R.C., Watson D.E., Turner P.A., Casola J.R. Mixing and Compaction Temperatures of Asphalt Binders in Hot-Mix Asphalt. NCHRP Report 648. Transportation Research Board. 2010.
- [23] Wymagania Techniczne WT-2 2010. Nawierzchnie Asfaltowe na drogach krajowych. (Technical Requirements WT-2 2010. Bituminous pavements on national roads) GDDKiA 2010.

## AUTHORS OF THE BITUMEN HANDBOOK



**Krzysztof Błażejowski (PhD Eng.)**

Graduate of the Civil Engineering Faculty at the Warsaw University of Technology (1992). Author of numerous publications on bituminous binders and pavements. Director for Technology, Research and Development at ORLEN Asphalt. Member of the Polish Academy of Sciences' Construction Materials Engineering Section. Road Pavement Expert of the Association of Polish Transport Engineers and Technicians (SITK RP).



**Jacek Olszacki (PhD Eng.)**

Graduate of the Construction, Architecture and Environmental Engineering Faculty at the Łódź University of Technology (2000). Author of numerous publications on porous asphalt and noise-reducing pavements. Involved in bituminous pavement rheology research, including DSR. Staff member of the Technology, Research and Development at ORLEN Asphalt.



**Hubert Peciakowski (M.Sc. Eng.)**

Graduate of the Construction, Mechanical and Petrochemical Engineering Faculty at the Warsaw University of Technology (2003). Specialised in bituminous binder research and production processes. His additional research focus is on the impact of raw materials on the quality of finished products. Staff member of the Technology, Research and Development at ORLEN Asphalt.

### Technology, Research and Development Department (TRDD)

Company department at ORLEN Asphalt within the production division. Active from the company's foundation in 2003. The TRDD deals with production technology, tests and development research on bituminous pavements, technical marketing and new product development. It also offers technical consultancy to customers on the application of bituminous binders manufactured by the company.

The TRDD achievements include patent applications, gold medal at the International Invention Exhibition IWIS 2007, and the prize awarded by the Polish Minister of Science and Higher Education for achievements in the area of inventions.

Technical consultancy is available for the company's customers at: [technology@orlen-asfalt.pl](mailto:technology@orlen-asfalt.pl).



This **Bitumen Handbook 2014**, prepared by the Technology, Research and Development Department of ORLEN Asphalt, contains a collection of technical information about bitumens, their testing methods and test results.

Part One provides information about bitumen testing methods. Part Two offers an overview of bitumens manufactured by ORLEN Asphalt and their properties according to EN standards and the American Performance Grade standard. Part Three provides insight into bitumen technology, which may come in useful for each road laboratory.

Bitumen Handbook 2014 is prepared for customers of ORLEN Asphalt and all those interested in the application of bitumen for road construction.

