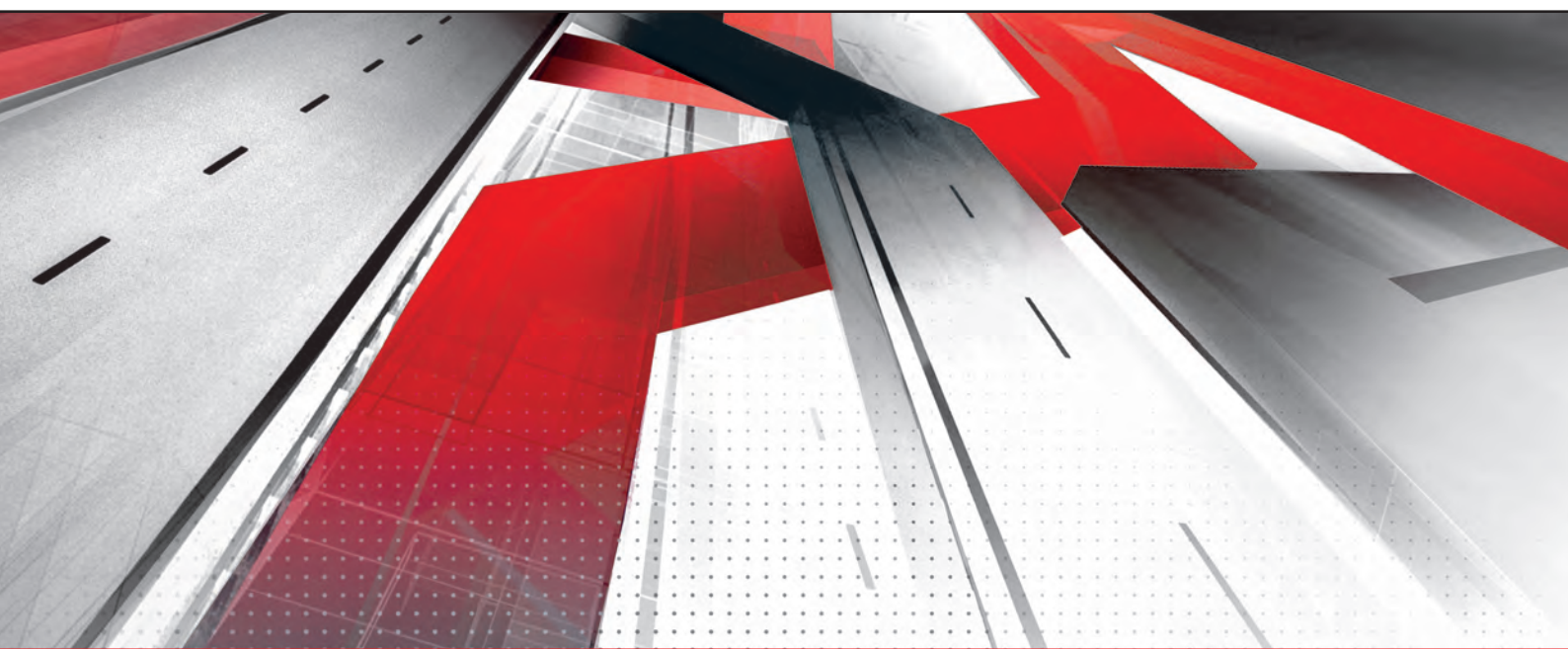


# 2017

## BITUMEN HANDBOOK

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Bitumen Handbook 2017  
ORLEN Asfalt Sp. z o.o., Poland

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## ABOUT ORLEN ASFALT

ORLEN Asphalt was divested in 2003 from the framework of PKN ORLEN, a Polish liquid fuel corporation, and it soon became one of the major Polish bitumen manufacturers and sellers. Since then the company has supplied its products for all major road building projects, thus, building its brand of a supplier of top-quality bitumen and a reliable business partner. ORLEN Asphalt intends to increase its sales to European markets, to build its image as a notable supplier in Europe, and to maintain its leading position on the Polish market.

Romania remains one of the major export markets, where our company has gained the status of the leading supplier. The growing demand for bitumen in this region was one of the main reasons for opening a division of ORLEN Asphalt in Bucharest and for commissioning a railway terminal, which was built to facilitate the reloading of bitumen and to expedite Poland – Romania transports. The terminal has been equipped with three tanks of the combined volume of 5000 m<sup>3</sup>, which are dedicated to the storage of 50/70 and 70/100 bitumen.

Since 2012, ORLEN Asphalt is the sole shareholder of a Czech company ORLEN Asphalt Česká republika s.r.o., formed as a result of the acquisition of all shares in Paramo Asphalt, selling bitumen manufactured in Czech refineries in Pardubice and Litvinov.

We are a transparent company, therefore any operations are carried out according to regulations and corporate governance of the PKN ORLEN S.A. Capital Group, bearing in mind at the same time the development of our employees and the preservation of the natural environment. Since 2005, the company operates in compliance with an integrated Management System, on the basis of standards ISO9001, ISO14001, OHSAS18001.

High quality of products on offer from ORLEN Asphalt is corroborated 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 Minister of Economy and the Polish Agency for Enterprise Development. ORBITON 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 Transportation Engineers and Technicians for outstanding achievements and products. In 2011, ORLEN Asphalt was awarded with the QI Golden Emblem for top product quality awarded in the programme implemented under the auspices of the Minister of Regional Development, the Polish Agency for Enterprise Development and the Polish ISO 9000 Forum Club. The Company was also twice awarded 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. In 2014, ORBITON HiMA binders highly modified with polymers were awarded the gold medal during the 20th International Road Construction Fair Autostrada-Polska, and received the prize of Leader of Innovation for 2015 in the Diamonds of Polish Infrastructure competition organised by the Executive Club.

The 2017 Bitumen Handbook has been developed by the employees of the Department of Research and Development of ORLEN Asphalt. It includes a series of technical data on bituminous binders and the current results of research carried out by ORLEN Asphalt

### Our products

The current product range of our Company includes paving grade bitumens, ORBITON modified bitumens, ORBITON HIMA highly-modified binders and oxidised bitumens. Following the consolidation of the bitumen segment, we have further extended our product offering to include bitumens from the Czech Republic (Pardubice and Litvínov production centres) and Lithuania (Mažeikiai production centre).

Production plants and major commercial markets of the ORLEN Asphalt Capital Group.



#### Bitumen products by production location:

Plock	Trzebinia	Litvinov	Pardubice	Mazeikiai
<b>Paving-grade bitumens</b>	<b>Paving-grade bitumens</b>	<b>Paving-grade bitumens</b>	<b>Paving-grade bitumens</b>	<b>Paving-grade bitumens</b>
20/30	20/30	50/70	20/30	20/30
35/50	35/50	70/100	30/45	35/50
50/70	50/70	160/220	35/50	50/70
70/100	70/100		50/70	70/100
100/150	100/150		70/100	100/150
160/220	160/220		160/220	160/220
<b>Modified bitumen</b>	<b>Modified bitumen</b>		<b>Multigrade bitumen</b>	Special Bitumen
ORBITON 10/40-65	ORBITON 10/40-65		VMT 25	BNK 40/180
ORBITON 25/55-55 EXP	ORBITON 25/55-60		VMT 45	
ORBITON 25/55-60	ORBITON 25/55-60 EXP		VMT 65	
ORBITON 45/80-55	ORBITON 25/55-65 EXP			
ORBITON 45/80-65	ORBITON 45/80-55			
ORBITON 65/105-60	ORBITON 45/80-65			
	ORBITON 65/105-60			
<b>Highly-modified bitumen</b>	<b>Highly-modified bitumen</b>		<b>Hard paving-grade bitumens</b>	
ORBITON 25/55-80 HiMA	ORBITON 25/55-60 HiMA		AP 15 (10/20)	
ORBITON 45/80-80 HiMA	ORBITON 45/80-80 HiMA		AP 25 (20/30)	
ORBITON 65/105-80 HiMA	ORBITON 65/105-80 HiMA			
			<b>Oxidised bitumen</b>	
	<b>Oxidised bitumen</b>		85/15	
	80/15		85/25	
	95/35		85/40	
			95/35	
			105/15	

## Chapter 1

### BITUMEN MANUFACTURING PROCESS

ORLEN Asphalt is a manufacturer of several paving grade bitumen types. The two manufacturing systems at the Płock (PKN ORLEN S.A.) and Trzebinia (ORLEN Południe S.A.) refineries produce the following types of bitumen: paving grade bitumen, ORBITON modified bitumen, ORBITON HiMA highly-modified bitumen and oxidised (industrial) bitumen.

In accordance with the Regulation No 305/2011 of the European Parliament and of the Council, which defines the conditions of marketing of construction products, ORLEN Asphalt has implemented a Factory Production Control (FPC) system, while production sites in Płock (PKN ORLEN) and Trzebinia have the appropriate FPC certificates (Table 1.1.).

Table 1.1. List of FPC Certificates for the Production Sites in Płock and Trzebinia.

	No. of FPC Certificate for the Płock Production Site (PKN ORLEN S.A.)	No. of FPC Certificate for the Trzebinia Production Site
Paving Grade Bitumen	1434-CPR-0183	1434-CPR-0185
ORBITON polymer-modified bitumen	1434-CPR-0184	1434-CPR-0186
ORBITON HiMA bitumen highly modified with polymer	1434-CPR-0184	1434-CPR-0186

#### 1.1. BITUMEN PRODUCTION TECHNOLOGIES AT ORLEN ASFALT

Bituminous binders from ORLEN Asphalt are manufactured from conventional raw material sources, and specifically from vacuum residue obtained as a result of crude oil processing. A general diagram of the process of manufacturing of bitumen has been shown below.

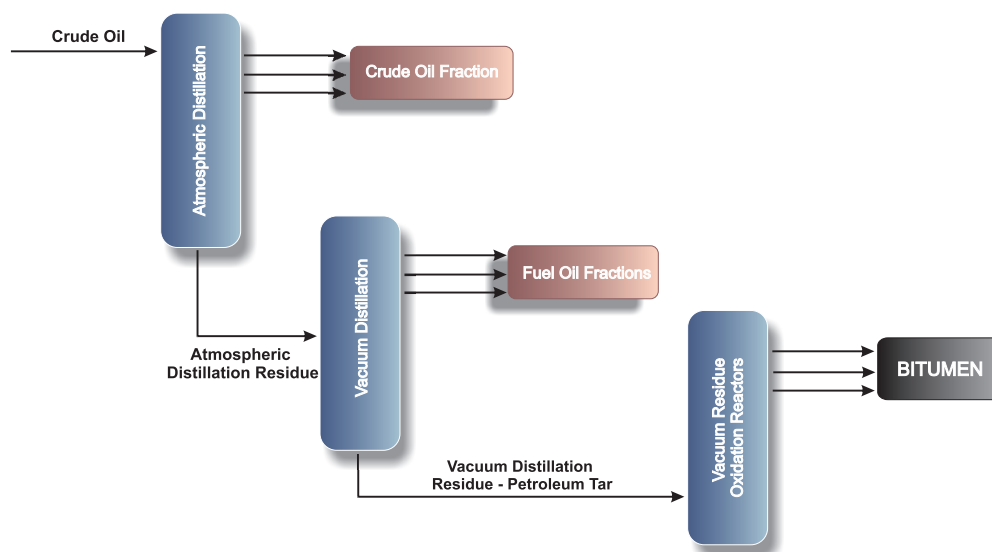


Fig. 1.1. Bitumen Manufacturing Diagram



The first phase of the bitumen production process consists of the two-stage distillation of crude oil. The distillation is initially performed under atmospheric pressure, with the separation of lighter components of crude oil. The remaining content is then introduced into the vacuum column, where it is subject to fractionation in low pressure. Vacuum residue is collected from the vacuum column, and subsequently subjected to oxidation. Process conditions, such as pressure and temperature, depend on the type of processed raw material and on the required properties of the end product. 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 also taking place concurrently produce oxygen compounds, and the associated dehydrogenative condensation of hydrocarbons leads to the creation of C-C (carbon-carbon) bonds. The result of this process is the production of resins and asphaltenes at the expense of naphthenic aromatic hydrocarbons. The mechanism of this reaction is strictly dependant on the process 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 as a result of ongoing reactions.

The process of oxidation may be conducted in a continuous or periodic manner. Bitumen obtained as described above belongs to the *semi-blown* or *air-rectified* type group of bituminous binders.

### 1.1.1. BITUROX® technology vacuum residue continuous oxidation system

The continuous oxidation system can be used for the production of paving grade bitumen and for the production of bases for modified and highly-modified bitumen. Production technology is licensed from Pörner of Austria. The process involves continuous, uninterrupted feedstock batching to the reactors and continuous discharge of products to storage tanks. The process is also marked by optimum utilisation of oxygen for oxidation and excellent hydrodynamic properties of the reaction. Process continuity ensures product homogeneity, i.e. uniform properties of the entire end product structure.

Biturox® reactors are at the heart of the manufacturing system. 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 (fig. 1.2).

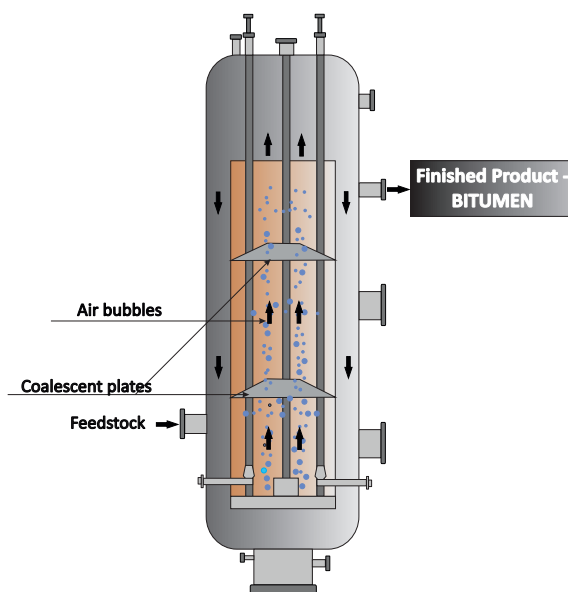


Fig. 1.2. Diagram of BITUROX® type reactor

Air forming large bubbles moves upwards inside the cylinder, where it is collected at two levels by coalescing plates and broken into smaller bubbles by the agitator's turbines. This ensures constant renovation of the reaction surfaces. The process takes place in the entire volume, using less air and over a shorter time of residence of raw materials in the reactor. The air flow is selected so that the quantity of oxygen in the off gas is 2–5 % (v/v). 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 down in bitumen coolers. Subsequently, it is sent to storage tanks, where it is mixed and tested for quality. Bitumen distribution to road and rail tankers takes place at sealed loading stations. The entire process is controlled by the DCS computer system.



Fig. 1.3. Continuous oxidation system – Biturox® reactors (photo by ORLEN Asphalt)

### 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 the production of other types of non-modified bitumen.

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

### Modification of bitumen

The purpose of bitumen modification with polymers is to expand the temperature range (the so-called Plasticity Range) in which bitumen demonstrates viscoelastic properties. The primary feedstock for polymer-modified bitumen production at ORLEN Asphalt comprises special bitumens, so-called base bitumens of adequate properties, which

are compatible with the applied polymers. 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*.

### 1.1.3. Bitumen polymer modification system

Manufacturing systems at ORLEN Asphalt implement the physical method for the production of modified bitumen, which involves the mechanical mixing of bitumen and polymer, with potential addition of crosslinkers.

SMS polymer is introduced into the hot base bitumen, and then the obtained mixture is fed to a high-shear mill, where the mixture is subject to milling, final mixing and homogenisation. The end product is quality tested in certified laboratories of the ORLEN Capital Group.

The technology of production of polymer bitumen has been developed to ensure that the bitumen/polymer mixture is stable and does not segregate during storage and transport.

ORLEN Asphalt supplies products manufactured at two Polish polymer-bitumen manufacturing plants – in Płock and Trzebinia. Fig. 1.4. shows the diagram for the Płock site's bitumen modification system.

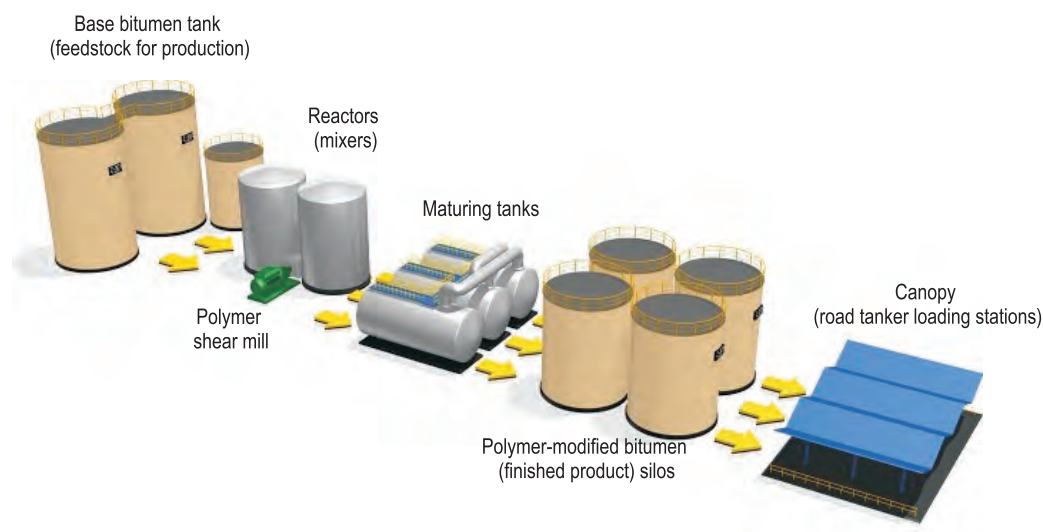


Fig. 1.4. Płock site bitumen modification system diagram

Both systems (Płock and Trzebinia sites) are automatically controlled using the DCS computer system, which enables full production process control and historical process data readout.

The application of modifier in the production process helps to achieve substantial benefits in terms of bitumen properties in both high and low temperatures. Thanks to their characteristics, polymer-modified bitumen products can be used for the construction of road pavements operating under heavy and very heavy traffic.

More details regarding the properties of each type of polymer bitumen can be found in the chapters on: ORBITON modified bitumens (Chapter 3), and in the chapter on ORBITON HiMA highly-modified bitumen (Chapter 4).

## Chapter 2

### PAVING-GRADE BITUMEN ACC. TO EN 12591

#### 2.1. OVERVIEW OF STANDARD EN 12591

##### 2.1.1. Introduction

Since 2010 ORLEN Asphalt has been involved in the production of paving-grade bitumen according to the requirements of European standard EN 12591, which represents a part of a set of standards related to bituminous binders.

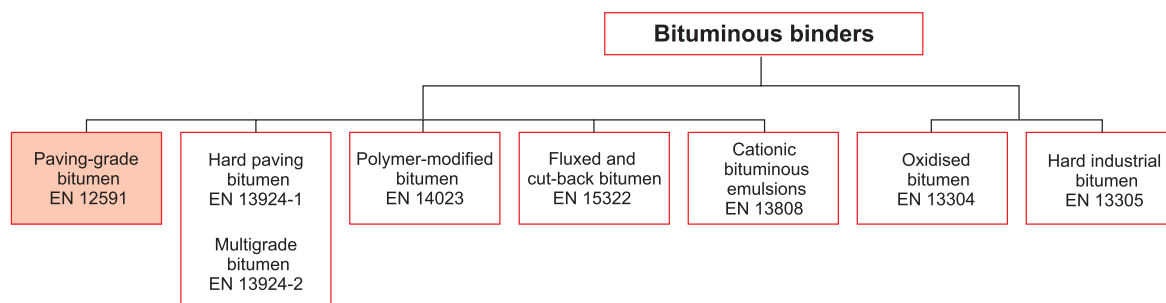


Fig. 2.1. Attribution of European Standards to various types of binders.  
The analysed standard has been shown in colour.

Standard EN 12591:2009 has been developed on the basis of a mandate, i.e. a request issued by the European Commission to the European Committee for Standardization. Initially, it supported the essential requirements of the *EU Construction Products Directive CPD 89/106/EEC*. As of 1 July 2013, construction products (including bituminous binders) are governed by Regulation No 305/2011 (CPR) of the European Parliament and of the Council, which defines the harmonised conditions of their marketing.

**Standard EN 12591:2009 „Bitumen and bituminous binders – Specifications for paving grade bitumens“** is a partially classifying standard, i.e. most of the requirements for paving-grade bitumens are obligatory, but CEN member countries have the freedom to select some of them. This standard sets out the principles of defining properties and adequate testing techniques for bituminous binders used for the construction and upkeep of roads, airports and other road traffic-bearing pavements. It also includes all requirements regarding the assessment of conformity.

An amendment of standard EN 12591:2009 is expected to take place in the nearest future. The current version of standard EN 12591 (dated 2009) still refers e.g. to old regulations, according to the CPD directive, while the new version is expected to replace them with requirements compliant with the CPR directive.

##### 2.1.2. Paving-grade Bitumen Classification

Paving-grade bitumen types, manufactured according to the requirements of standard EN 12591, are labelled according to classification provided in table 2.1.

Table 2.1. Classification of paving grade bitumen types manufactured to European standard EN 12591

Bituminous binder	Paving Grade Bitumen
Reference document	EN 12591:2009
Standard designation of bituminous binder	XX/YY
Type of bituminous binder manufactured by ORLEN Asphalt	20/30, 35/50, 50/70, 70/100, 100/150, 160/220
Notes to designations: XX – lower penetration limit at 25°C for a given bitumen type [0.1 mm] YY – upper penetration limit at 25°C for a given bitumen type [0.1 mm]	

### 2.1.3. National application documents

In the European standards which set out the requirements for bituminous binders it is assumed that each Member State of CEN selects the properties and the related requirement levels requirement levels in the so-called Application Documents to the discussed standards.

Member States develop such documents in the form of “National Annexes” to the standards, or in the form of information about the selected requirements published in separate documents on asphalt pavement construction materials and technologies. This enables each Member State to specify its own requirements to be met by bituminous binders used within its territory. The differentiation results from diverse climate conditions in various parts of Europe and many other process factors.

Table 2.2. presents general requirements concerning paving-grade bitumen, according to information provided in standard EN 12591.

Table 2.2. Requirements concerning all types of paving-grade bitumen of the penetration from 20 to 220x0.1 mm, according to standard EN 12591:2009 (Table 1A and 1B)

	Property	Test method	Unit	Paving grade bitumen type							
				20/30	30/45	35/50	40/60	50/70	70/100	100/150	160/220
Properties applied to all paving-grade bitumen types specified in this table	Penetration at 25°C	EN 1426	0,1 mm	20-30	30-45	35-50	40-60	50-70	70-100	100-150	160-220
	Softening point	EN 1427	°C	55-63	52-60	50-58	48-56	46-54	43-51	39-47	35-43
	Resistance to hardening at 163°C	EN 12607-1									
	Retained penetration		%	≥ 55	≥ 53	≥ 53	≥ 50	≥ 50	≥ 46	≥ 43	≥ 37
	Softening point increase		°C	≤ 8 or ≤ 10	≤ 8 or ≤ 11	≤ 8 or ≤ 11	≤ 9 or ≤ 11	≤ 9 or ≤ 11	≤ 9 or ≤ 11	≤ 10 or ≤ 12	≤ 11 or ≤ 12
	Change in mass* (absolute)		%	≤ 0.5	≤ 0.5	≤ 0.5	≤ 0.5	≤ 0.5	≤ 0.8	≤ 0.8	≤ 1.0
	Flash point	EN ISO 2592	°C	≥ 240	≥ 240	≥ 240	≥ 230	≥ 230	≥ 230	≥ 230	≥ 220
	Solubility	EN 12592	%	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0
Properties adapted to country-specific conditions	Penetration index	EN 12591 Annex A	–	-1.5 ÷ +0.7 or NR	-1.5 ÷ +0.7 or NR	-1.5 ÷ +0.7 or NR	-1.5 ÷ +0.7 or NR	-1.5 ÷ +0.7 or NR	-1.5 ÷ +0.7 or NR	-1.5 ÷ +0.7 or NR	-1.5 ÷ +0.7 or NR
	Dynamic viscosity at 60°C	EN 12596	Pa · s	≥ 440 or NR	≥ 260 or NR	≥ 225 or NR	≥ 175 or NR	≥ 145 or NR	≥ 90 or NR	≥ 55 or NR	≥ 30 or NR
	Fraass breaking point	EN 12593	°C	NR	≤ -5 or NR	≤ -5 or NR	≤ -7 or NR	≤ -8 or NR	≤ -10 or NR	≤ -12 or NR	≤ -15 or NR
	Kinematic viscosity at 135°C	EN 12595	mm²/s	≥ 530 or NR	≥ 400 or NR	≥ 370 or NR	≥ 325 or NR	≥ 295 or NR	≥ 230 or NR	≥ 175 or NR	≥ 135 or NR
* Change in mass may be a positive or negative value NR – (No Requirement) – indicates that there are no requirements for a specific property											

Table 2.3. shows the requirements applicable to paving-grade bitumen types used for road construction **in Poland**, with the consideration of country-specific conditions (standard PN-EN 12591:2010 includes the NA National Annex, which consists of Table NA.1A and Table NA.1B).

**Table 2.3. Requirements related to different types of paving-grade bitumen used in Poland, acc. to the NA National Annex to standard PN-EN 12591:2010.**

Property	Test method	Unit	Paving grade bitumen type					
			20/30	35/50	50/70	70/100	100/150	160/220
Penetration at 25°C	EN 1426	0.1 mm	20-30	35-50	50-70	70-100	100-150	160-220
Softening point	EN 1427	°C	55-63	50-58	46-54	43-51	39-47	35-43
Resistance to hardening at 163°C	EN 12607-1 (RTFOT method)							
Retained penetration		%	≥ 55	≥ 53	≥ 50	≥ 46	≥ 43	≥ 37
Softening point increase		°C	≤ 8	≤ 8	≤ 9	≤ 9	≤ 10	≤ 11
Change in mass* (absolute)		%	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,8	≤ 0,8	≤ 1.0
Flash point	EN ISO 2592	°C	≥ 240	≥ 240	≥ 230	≥ 230	≥ 230	≥ 220
Solubility	EN 12592	% (m/m)	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0	≥ 99.0
Penetration index	EN 12591 Annex A	–	NR	NR	NR	NR	NR	NR
Dynamic viscosity at 60°C	EN 12596	Pa · s	NR	NR	NR	NR	NR	NR
Fraass breaking point	EN 12593	°C	NR	≤ -5	≤ -8	≤ -10	≤ -12	≤ -15
Kinematic viscosity at 135°C	EN 12595	mm <sup>2</sup> /s	NR	NR	NR	NR	NR	NR
* Change in mass may be a positive or negative value NR – (No Requirement) – indicates that there are no requirements for a specific property								

## 2.1.4. Evaluation of Conformity

According to standard EN 12591:2009 and EN 12591:2010, the manufacturer is obliged to establish, document and maintain Factory Production Control (FPC).

The conformity of the properties of paving-grade bitumen with standard requirements and with values provided therein shall be demonstrated by:

- The performance of an initial type test for each type of bitumen,
- implementation and application of the Factory Production Control (FPC).

Paving-grade bitumen types used for road construction and surface dressing are covered by a “2+” conformity assessment system, which requires every manufacturer to implement the Factory Production Control system, and this implementation needs to be certified by a FPC Certificate. This Certificate shall be issued by a notified body.

The FPC system consists of procedures, regular inspections and tests and/or assessments, while the results serve to evaluate the quality of the finished product. Additionally, the manufacturer must have in place a sample testing plan and he should perform type tests for each product. The numbers of FPC Certificates applicable to manufacturing plants ORLEN Asphalt in Trzebinia and PKN ORLEN in Plock can be found in Chapter 1.

Annex ZA to standard EN 12591 also includes the procedure for paving-grade bitumen conformity assessment, the division of responsibilities between the manufacturer and the notified body, a chapter on the certification and declaration of conformity, CE marking and labelling.





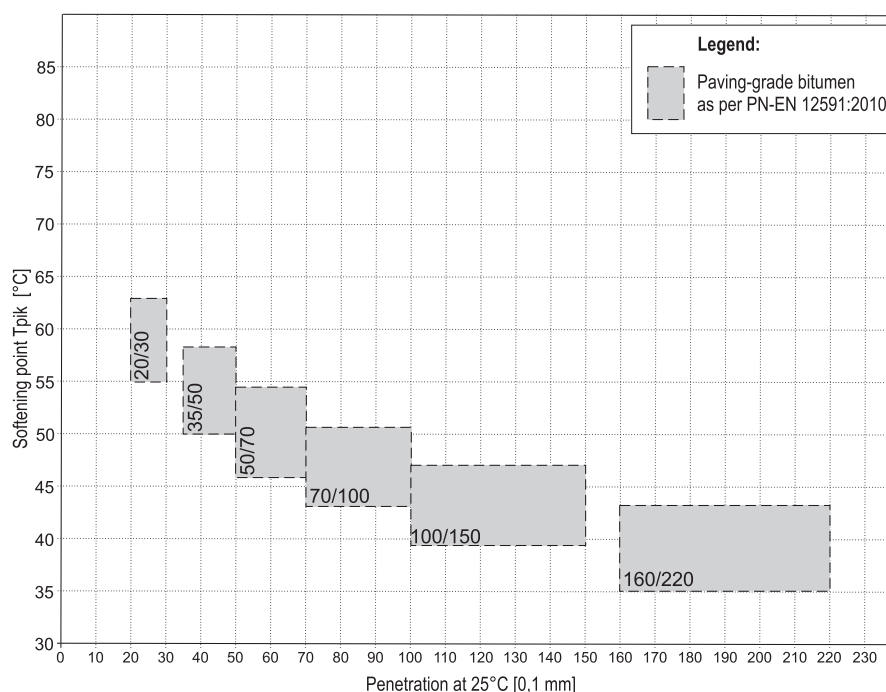


Fig. 2.3. Schematic comparison of Paving Grade Bitumen types acc. to EN 12591:2009 as a function of penetration at 25°C and softening point R&B

### 2.2.2. Intended use

The typical applications of individual types of paving-grade bitumen have been shown below.

**Paving-grade bitumen 20/30** is the hardest paving-grade bitumen according to EN 12591. 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 and rather mild winters. Courses with bitumen 20/30 should not be left over the winter period without the being covered with the next course.

**Paving-grade bitumen 35/50 and 50/70** are the most popular binder types used for the production of asphalt mixtures.

- **Paving-grade bitumen 35/50** can be used for the production of asphalt concrete (AC) for base and binder courses, or in the wearing course as mastic asphalt (MA) used for roads designed to carry light traffic. 35/50 bitumen should not be used in wearing courses for the production of asphalt concrete or SMA.
- **Paving-grade bitumen 50/70** can be used primarily for asphalt concrete and SMA in wearing courses, provided that the mixture complies with the requirements concerning resistance to rutting. The use of 50/70 bitumen for the production of base and binder courses also requires the verification of the mixture's resistance to rutting. The use of 50/70 bitumen for the production of any layer of pavement designed to carry slow traffic (slow lanes, approaches to crossroads, etc.) is not recommended.



**Paving-grade bitumen 70/100** can be used to a limited extent for asphalt concrete and SMA in wearing courses on roads designed for light traffic, on the assumption that the mix resistance to rutting is confirmed. It can also be used for the production of bitumen emulsions.

**Paving-grade bitumens 70/100, 100/150 and 160/220** comprise a group of binders designed for the production of bitumen emulsions of various applications.

Due to the risk of rutting, the use of paving-grade bitumens should always be preceded by testing the mixture's resistance to rutting, according to EN 12697-22 (in Poland, the following conditions of testing have been implemented: method B, small-size apparatus, in the air, temperature +60°C, 10000 cycles and requirements for the  $WTS_{air}$  parameter dependant on the density of traffic). The above refers especially to road sections located within crossroad zones, slow traffic zones, parking zones, etc.

### 2.2.3. Properties

The following parts of this chapter introduce a set of paving-grade bitumen properties specified according to EN 12591, and additional information obtained via tests carried out according to the American *Superpave* method. The chapter also contains the classification of paving-grade bitumen types according to traffic loads, developed on the basis of results of MSCR tests (detailed analysis of the MSCR test can be found in Chapter 7).

This chapter also provides approximate process temperatures for bitumen application in asphalt mixtures, viscosity data and viscosity dependence on temperature.

#### 2.2.3.1. Paving-grade bitumen 20/30

##### Properties as per EN 12591:2009

The requirements for paving-grade bitumen 20/30 and the results of laboratory testing carried out in 2016 have been shown in table 2.4.

**Table 2.4.** Properties of 20/30 paving-grade bitumen manufactured in 2016 (*results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484*)

Property	Test method	Unit	Requirement acc. to EN 12591	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	20 – 30	27
Softening point	EN 1427	°C	55 – 63	61.5
Fraass breaking point	EN 12593	°C	NR	-9
Flash point	EN ISO 2592	°C	≥ 240	325
Solubility	EN 12592	% (m/m)	≥ 99.0	99.92
Change in mass after RTFOT	EN 12607-1	% (m/m)	≤ 0.5	-0.07
Retained penetration after RTFOT	EN 12607-1 EN 1426	%	≥ 55	73
Softening point increase after RTFOT	EN 12607-1 EN 1427	°C	≤ 8	7.3
Kinematic viscosity at 135°C	EN 12595	mm <sup>2</sup> /s	NR	1 588
Dynamic viscosity at 60°C	EN 12596	Pa*s	NR	2 674

## Properties acc. to Superpave

In the following section we present the properties of 20/30 bitumen specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

- **Performance Grade**, classification acc. to 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)  $T_{crit} = 84.7^\circ\text{C}$
- $G^* \cdot \sin\delta = 5000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 26.0^\circ\text{C}$

- **Low critical temperatures** (AASHTO PP 42; EN 14771):

- temperature at  $S(60) = 300$  MPa  $T(S)_{60} = -14.7^\circ\text{C}$
- temperature at  $m(60) = 0.3$   $T(m)_{60} = -8.1^\circ\text{C}$
- stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 370.5$  MPa

- **results and classification of bitumens based on the MSCR method**

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.047	0.130	0.305	0.038	0.215	1.040
$J_{nr}$ 3.2 kPa	0.048	0.137	0.342	0.038	0.231	1.260
$J_{nr}$ diff	3.2	5.4	12.3	1.9	7.3	20.8
R 0,1 kPa	51.4	39.9	31.1	49.5	31.1	17.2
R 3.2 kPa	49.5	36.6	23.9	48.3	26.4	6.5
R diff	3.7	8.4	23.4	2.4	15.2	62.2
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Extreme	not subject to classification		

## Viscosity dependence on temperature

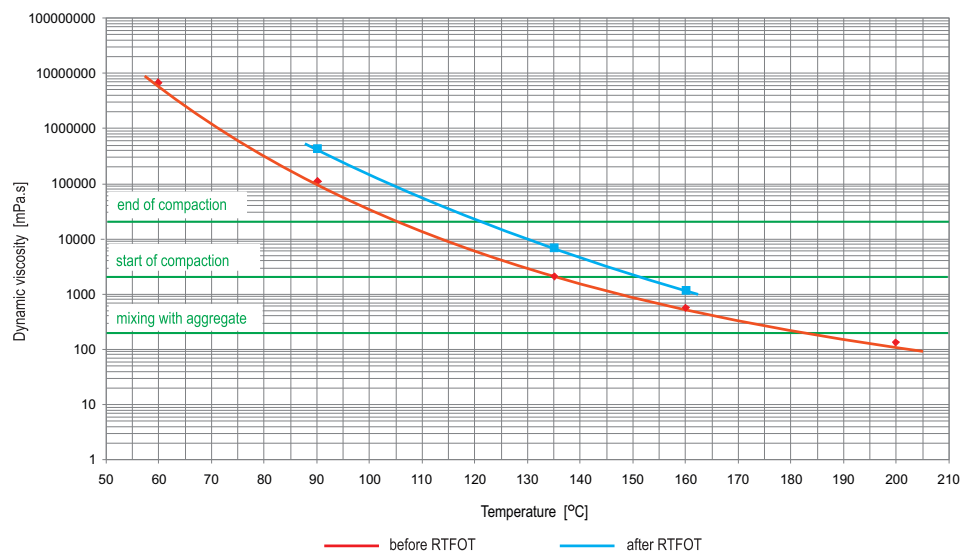


Fig. 2.4. Relation between viscosity and temperature for paving-grade bitumen 20/30

Table 2.5. Example results of viscosity tests on bitumen 20/30 manufactured in 2015. Tests carried out 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	2 579
	Brookfield viscometer	ASTM D4402 EN 13302	spindle No 21, 29	Pa*s	90°C	94.08
					135°C	1.87
					160°C	0.48
					200°C	0.12
			spindle No 27	Pa*s	90°C after RTFOT	414.33
					135°C after RTFOT	5.64
					160°C after RTFOT	1.32
Kinematic	BS/IP/RF viscometer	EN 12595	—	mm <sup>2</sup> /s	135°C	1 280

## Process temperatures

<b>At laboratory:</b>	
Sample compaction temperature (Marshall compactor or samples compacted in gyratory compactor)	155 ÷ 160°C
<b>At mixing plant:</b>	
Bitumen pumping temperature	> 145°C
Temperature of bitumen for asphalt mixture production	175 ÷ 185°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 6h)	< 220°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 2h)	< 230°C
<b>Attention:</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 asphalt mixture (spreader's hopper)	165°C

## Storage

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

- recommended bitumen storage temperature: ≤ 185°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 in high temperatures (up to 185°C) for over 10 days, it is recommended to inspect the binder ageing rate before the use of bitumen for the production of asphalt mixtures. Penetration at 25°C as per EN 1426 or softening point as per EN 1427 should be tested.

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 necessary to store paving-grade bitumen 20/30 for a much longer period than 10 days, it is recommended to reduce the temperature of bitumen and to reheat it before reuse. If a very long storage period is expected without any production of asphalt mixture, 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.

After reheating penetration at 25°C as per EN 1426 or softening point as per EN 1427 should be tested.

### 2.2.3.2. Paving-grade bitumen 35/50

#### Properties as per EN 12591:2009

The requirements for paving-grade bitumen 35/50 and the results of laboratory testing carried out in 2016 have been shown in table 2.6.

Table 2.6. Properties of 35/50 paving-grade bitumen manufactured in 2016 (*results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484*)

Property	Test method	Unit	Requirement acc. to EN 12591	Mean value for 2016
Penetration at 25°C	EN 1426	0,1 mm	35 – 50	42
Softening point	EN 1427	°C	50 – 58	54.8
Fraass breaking point	EN 12593	°C	≤ -5	-13
Flash point	EN ISO 2592	°C	≥ 240	324
Solubility	EN 12592	% (m/m)	≥ 99.0	99.94
Change in mass after RTFOT	EN 12607-1	% (m/m)	≤ 0.5	-0.09
Retained penetration after RTFOT	EN 12607-1 EN 1426	%	≥ 53	63
Softening point increase after RTFOT	EN 12607-1 EN 1427	°C	≤ 8	7.1
Kinematic viscosity at 135°C	EN 12595	mm²/s	NR	745
Dynamic viscosity at 60°C	EN 12596	Pa*s	NR	695

#### Properties acc. to Superpave

In the following section, we present the properties of 35/50 paving-grade bitumen specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

- **Performance Grade**, 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)  $T_{crit} = 74.2^\circ\text{C}$
  - $G^* \cdot \sin\delta = 5000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 23.1^\circ\text{C}$
- **Low critical temperatures** (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) = 300$  MPa  $T(S)_{60} = -23.1^\circ\text{C}$
  - temperature at  $m(60) = 0,3$   $T(m)_{60} = -11.5^\circ\text{C}$
  - stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 338.5$  MPa

# • Results and classification of bitumens based on the MSCR method

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.146	0.380	0.807	0.174	0.858	3.520
$J_{nr}$ 3.2 kPa	0.153	0.419	0.926	0.179	0.960	4.010
$J_{nr}$ diff	4.6	10.2	14.8	2.9	11.9	13.8
R 0.1 kPa	32.5	23.7	16.5	25.4	13.3	3.5
R 3.2 kPa	29.4	17.2	8.7	23	6.5	0.2
R diff	9.7	27.2	47.6	9.9	51.3	92.8
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Very Heavy	not subject to classification		

## Process temperatures

<b>At laboratory:</b>	
Sample compaction temperature (Marshall compactor or samples compacted in gyratory compactor)	140 ÷ 145°C
<b>At mixing plant:</b>	
Bitumen pumping temperature	> 140°C
Temperature of bitumen for asphalt mixture production	165 ÷ 175°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 6h)	< 220°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 2h)	< 230°C
<b>Attention:</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 asphalt mixture (spreader's hopper)	150°C

## Viscosity dependence on temperature

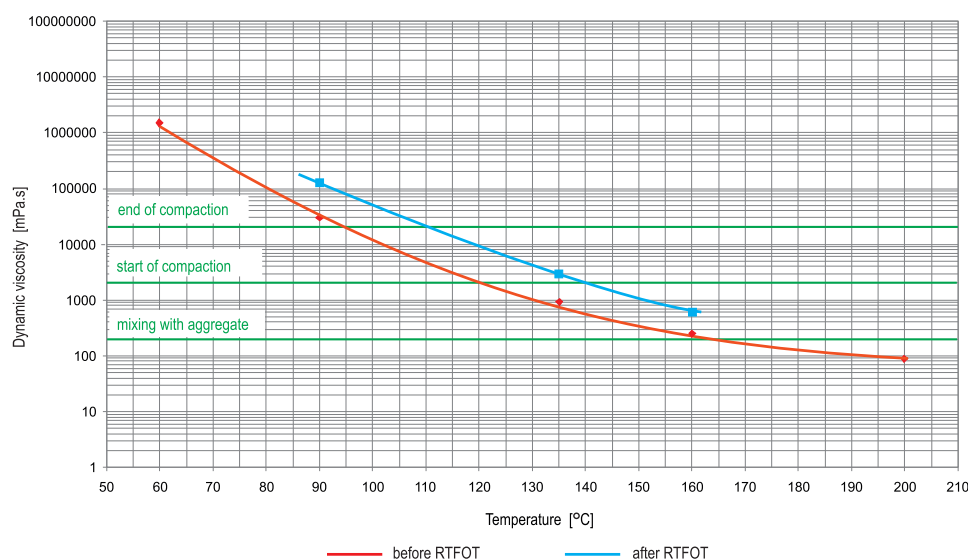


Fig. 2.5. Relation between viscosity and temperature for paving-grade bitumen 35/50

Table 2.7. Example results of viscosity tests on bitumen 35/50 manufactured in 2015. Tests carried out 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	747
	Brookfield viscometer	ASTM D4402 EN 13302	spindle No 27	Pa*s	90°C	29.39
					135°C	0.95
					160°C	0.28
					200°C	0.08
			spindle No 27	Pa*s	90°C after RTFOT	112.00
					135°C after RTFOT	2.23
					160°C after RTFOT	0.55
Kinematic	BS/IP/RF viscometer	EN 12595	—	mm <sup>2</sup> /s	135°C	748

## 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 in high temperatures (up to 185°C) for over 10 days, it is recommended to inspect the binder ageing rate before the use of bitumen for the production of asphalt mixtures. Penetration at 25°C as per EN 1426 or softening point as per EN 1427 should be tested.

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 necessary to store paving-grade bitumen 35/50 for a much longer period than 10 days, it is recommended to reduce the temperature of bitumen and to reheat it before reuse. If a very long storage period is expected without any production of asphalt mixture, 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.

After reheating penetration at 25°C as per EN 1426 or softening point as per EN 1427 should be tested.

### 2.2.3.3. Paving-grade bitumen 50/70

#### Properties as per EN 12591:2009

The requirements for paving-grade bitumen 50/70 and the results of laboratory testing carried out in 2016 have been shown in table 2.8.

Table 2.8. Properties of 50/70 paving-grade bitumen manufactured in 2016 (results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484)

Property	Test method	Unit	Requirement acc. to EN 12591	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	50 – 70	60
Softening point	EN 1427	°C	46 – 54	49.4
Fraass breaking point	EN 12593	°C	≤ -8	-15
Flash point	EN ISO 2592	°C	≥ 230	316
Solubility	EN 12592	% (m/m)	≥ 99.0	99.95
Change in mass after RTFOT	EN 12607-1	% (m/m)	≤ 0.5	-0.01
Retained penetration after RTFOT (absolute)	EN 12607-1 EN 1426	%	≥ 50	70
Softening point increase after RTFOT	EN 12607-1 EN 1427	°C	≤ 9	4.9
Kinematic viscosity at 135°C	EN 12595	mm <sup>2</sup> /s	NR	499
Dynamic viscosity at 60°C	EN 12596	Pa*s	NR	321

### Properties acc. to Superpave

In the following section, we present the properties of 50/70 paving-grade bitumen specified acc. to the American Superpave method and carried out between 2012 and 2015.

- **Performance Grade**, classification acc. to AASHTO MP 1: **PG 64-22**

- **High critical temperatures** (AASHTO T 315):

- $G^*/\sin\delta = 1$  kPa (unaged 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  $S(60) = 300$  MPa  $T(S)_{60} = -16.6^\circ\text{C}$
- temperature at  $m(60) = 0.3$   $T(m)_{60} = -15.0^\circ\text{C}$
- stiffness at  $-16^\circ\text{C}$   $S(T)_{-16} = 294$  MPa

- **Results and classification of bitumens based on the MSCR method**

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
J <sub>nr</sub> 0.1 kPa	0.841	2.010	4.390	0.627	2.510	7.200
J <sub>nr</sub> 3.2 kPa	0.921	2.240	4.980	0.669	2.790	10.900
J <sub>nr</sub> diff	9.6	11.5	13.4	6.6	11.0	51.0
R 0.1 kPa	11.3	5.2	3.2	11.2	4.0	2.8
R 3.2 kPa	5.8	1.7	-0.5	7.3	0.9	-2.6
R diff	48.8	68.1	115.8	34.8	76.8	195.3
Final classification of suitability for road traffic, according to J <sub>nr</sub> 3.2 kPa (at test temperature)	Very Heavy	Standard	*	not subject to classification		
* result for J <sub>nr</sub> 3.2 kPa outside classification						

### Process temperatures

At laboratory	
Sample compaction temperature (Marshall compactor or samples compacted in gyratory compactor)	135 ÷ 140°C
At mixing plant	
Bitumen pumping temperature	> 130°C
Temperature of bitumen for asphalt mixture production	155 ÷ 165°C
At site	
Minimum temperature of the supplied asphalt mixture (spreader's hopper)	145°C

### Viscosity dependence on temperature

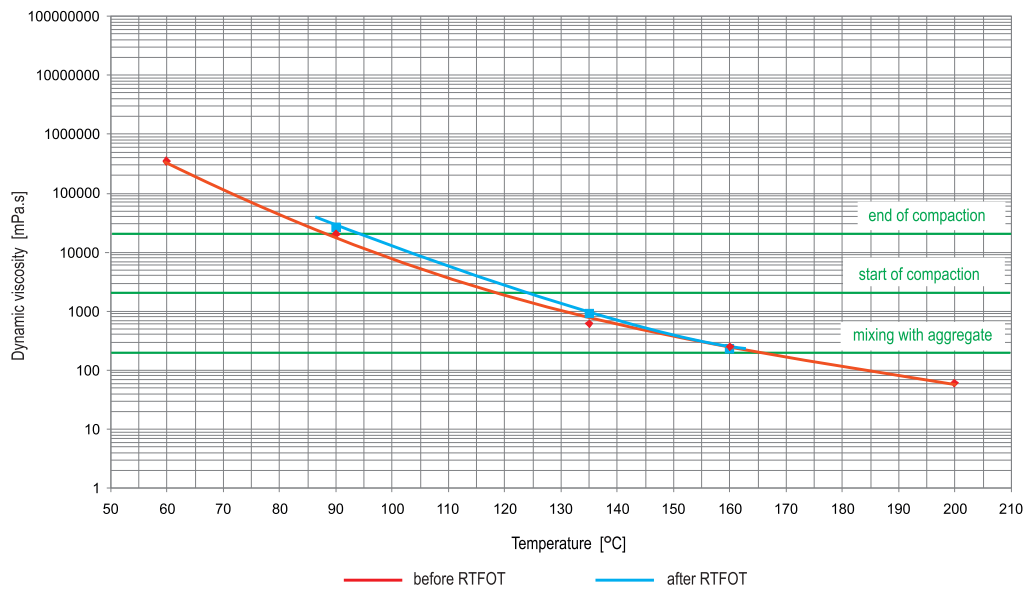


Fig. 2.6. Relation between viscosity and temperature for paving-grade bitumen 50/70

Table 2.9. Example results of viscosity tests on bitumen 50/70 manufactured in 2015. Tests carried out 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	291
	Brookfield viscometer	ASTM D4402 EN 13302	spindle No 21	Pa*s	90°C	17.10
					135°C	0.63
					160°C	0.22
					200°C	0.06
			spindle No 27	Pa*s	90°C after RTFOT	19.49
					135°C after RTFOT	0.65
					160°C po RTFOT	0.21
Kinematic	BS/IP/RF viscometer	EN 12595	—	mm²/s	135°C	486



## 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 in high temperatures (up to  $185^{\circ}\text{C}$ ) for over 10 days, it is recommended to inspect the binder ageing rate before the use of bitumen for the production of asphalt mixtures. Penetration at  $25^{\circ}\text{C}$  as per EN 1426 or softening point as per EN 1427 should be tested.

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 necessary to store paving-grade bitumen 50/70 for a much longer period than 10 days, it is recommended to reduce the temperature of bitumen and to reheat it before reuse. If a very long storage period is expected without any production of asphalt mixture, 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.

After reheating penetration at  $25^{\circ}\text{C}$  as per EN 1426 or softening point as per EN 1427 should be tested.

## 2.2.3.4. Paving-grade bitumen 70/100

### Properties as per EN 12591:2009

The requirements for paving-grade bitumen 70/100 and the results of laboratory testing carried out in 2016 have been shown in table 2.10.

Table 2.10. Properties of 70/100 paving-grade bitumen manufactured in 2016 (*results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484*)

Property	Test method	Unit	Requirement acc. to EN 12591	Mean value for 2016
Penetration at $25^{\circ}\text{C}$	EN 1426	0.1 mm	70 – 100	87
Softening point	EN 1427	$^{\circ}\text{C}$	43 – 51	45.5
Fraass breaking point	EN 12593	$^{\circ}\text{C}$	$\leq -10$	-16
Flash point	EN ISO 2592	$^{\circ}\text{C}$	$\geq 230$	323
Solubility	EN 12592	% (m/m)	$\geq 99.0$	99.96
Change in mass after RTFOT	EN 12607-1	% (m/m)	$\leq 0.8$	-0.01
Retained penetration after RTFOT	EN 12607-1 EN 1426	%	$\geq 46$	69
Softening point increase after RTFOT	EN 12607-1 EN 1427	$^{\circ}\text{C}$	$\leq 9$	4.0
Kinematic viscosity at $135^{\circ}\text{C}$	EN 12595	$\text{mm}^2/\text{s}$	NR	358
Dynamic viscosity at $60^{\circ}\text{C}$	EN 12596	$\text{Pa}\cdot\text{s}$	NR	154

## Properties acc. to Superpave

In the following section, we present the properties of 70/100 paving-grade bitumen specified acc. to the American Superpave method and carried out between 2012 and 2015.

- **Performance Grade**, classification acc. to AASHTO MP 1: **PG 58-22**

- **High critical temperatures** (AASHTO T 315):

– $G^*/\sin\delta = 1$ kPa (unaged bitumen)	$T_{crit} = 63.4^\circ\text{C}$
– $G^*/\sin\delta = 2.2$ kPa (bitumen after RTFOT)	$T_{crit} = 63.6^\circ\text{C}$
– $G^*\cdot\sin\delta = 5000$ kPa (bitumen after RTFOT and PAV)	$T_{crit} = 19.1^\circ\text{C}$

- **Low critical temperatures** (AASHTO PP 42; EN 14771):

– temperature at $S(60) = 300$ MPa	$T(S)_{60} = -16.9^\circ\text{C}$
– temperature at $m(60) = 0.3$	$T(m)_{60} = -16.2^\circ\text{C}$
– stiffness at $-16^\circ\text{C}$	$S(T)_{-16} = 285$ MPa

- **Results and classification of bitumens based on the MSCR method**

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
J <sub>nr</sub> 0.1 kPa	2.010	4.570	10.300	1.280	5.850	21.400
J <sub>nr</sub> 3.2 kPa	2.180	5.070	11.400	1.370	6.500	23.300
J <sub>nr</sub> diff	8.3	11.0	10.5	6.6	11.1	9.0
R 0.1 kPa	5.4	2.6	-2.5	4.6	1.6	-5.6
R 3.2 kPa	1.2	-0.7	-2.9	2.5	-1.4	-6.3
R diff	77.1	128.0	-17.0	45	189.1	-13.0
Final classification of suitability for road traffic, according to J <sub>nr</sub> 3.2 kPa (at test temperature)	Standard	*	*	not subject to classification		
* result for J <sub>nr</sub> 3.2 kPa outside classification						

## Process temperatures

<b>At laboratory:</b>	
Sample compaction temperature (Marshall samples (rammed samples) or samples compacted in gyratory compactor)	130 ÷ 135°C
<b>At mixing plant:</b>	
Bitumen pumping temperature	> 130°C
Temperature of bitumen for asphalt mixture production	150 ÷ 160°C
<b>At site:</b>	
Minimum temperature of the supplied asphalt mixture (spreader's hopper)	140°C

## Viscosity dependence on temperature

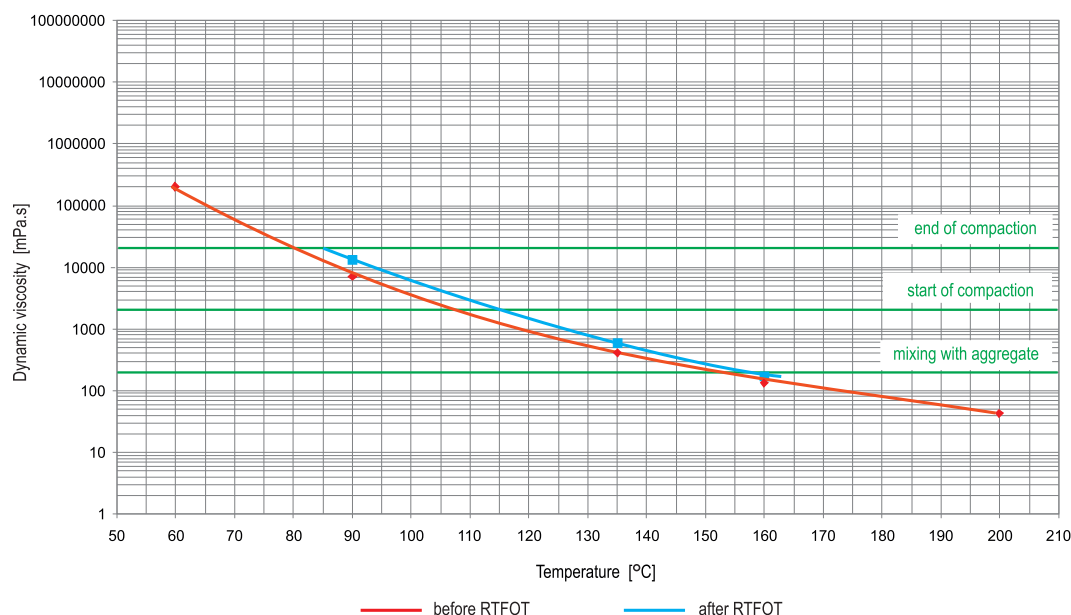


Fig.2.7. Relation between viscosity and temperature for paving-grade bitumen 70/100

Table 2.11. Example results of viscosity tests on bitumen 70/100 manufactured in 2015. Tests carried out 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	145
	Brookfield viscometer	ASTM D4402 EN 13302	spindle No 21	Pa*s	90°C	7.31
					135°C	0.39
					160°C	0.14
					200°C	0.04
			spindle No 27	Pa*s	90°C after RTFOT	12.65
					135°C after RTFOT	0.56
					160°C after RTFOT	0.18
kinematic	BS/IP/RF viscometer	EN 12595	—	mm²/s	135°C	352

## 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 in high temperatures (up to  $180^{\circ}\text{C}$ ) for over 10 days, it is recommended to inspect the binder ageing rate before the use of bitumen for the production of asphalt mixtures. Penetration at  $25^{\circ}\text{C}$  as per EN 1426 or softening point as per EN 1427 should be tested.

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 necessary to store paving-grade bitumen 70/100 for a much longer period than 10 days, it is recommended to reduce the temperature of bitumen and to reheat it before reuse. If a very long storage period is expected without any production of asphalt mixture, 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 overheating during long-term heating.

After reheating penetration at 25°C as per EN 1426 or softening point as per EN 1427 should be tested.

### 2.2.3.5. Paving-grade bitumen 100/150

#### Properties as per EN 12591:2009

The requirements for paving-grade bitumen 100/150 and the results of laboratory testing carried out in 2016 have been shown in table 2.12.

Table 2.12. Properties of 100/150 paving-grade bitumen manufactured in 2016 (results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484)

Property	Test method	Unit	Requirement acc. to EN 12591	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	100 – 150	132
Softening point	EN 1427	°C	39 – 47	42.2
Fraass breaking point	EN 12593	°C	≤ -12	-16
Flash point	EN ISO 2592	°C	≥ 230	323
Solubility	EN 12592	% (m/m)	≥ 99.0	99.92
Change in mass after RTFOT	EN 12607-1	% (m/m)	≤ 0.8	-0.14
Retained penetration after RTFOT	EN 12607-1 EN 1426	%	≥ 43	58
Softening point increase after RTFOT	EN 12607-1 EN 1427	°C	≤ 10	5.0
Kinematic viscosity at 135°C	EN 12595	mm <sup>2</sup> /s	NR	283
Dynamic viscosity at 60°C	EN 12596	Pa*s	NR	82

### Viscosity dependence on temperature

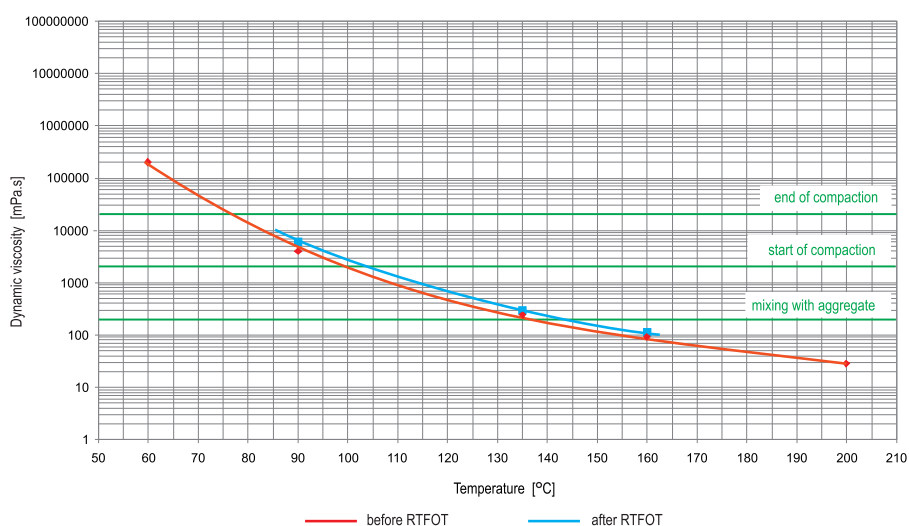


Fig. 2.8. Relation between viscosity and temperature for paving-grade bitumen 100/150

Table 2.13. Example results of viscosity tests on bitumen 100/150 manufactured in 2015. Tests carried out 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	81
	Brookfield viscometer	ASTM D4402 EN 13302	spindle No 18, 21	Pa*s	90°C	4.08
					135°C	0.26
					160°C	0.10
					200°C	0.03
			spindle No 21	Pa*s	90°C after RTFOT	6.41
					135°C after RTFOT	0.35
					160°C after RTFOT	0.13
kinematic	BS/IP/RF viscometer	EN 12595	—	mm <sup>2</sup> /s	135°C	267

## 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 in high temperatures (up to 180°C) for over 10 days, it is recommended to inspect the binder ageing rate before the use of bitumen for the production of asphalt mixtures or emulsions. Penetration at 25°C as per EN 1426 or softening point as per EN 1427 should be tested.

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 necessary to store paving-grade bitumen 100/150 for a much longer period than 10 days, it is recommended to reduce the temperature of bitumen and to reheat it before reuse. If a very long storage period is expected without any production of asphalt mixture, 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.

After reheating penetration at 25°C as per EN 1426 or softening point as per EN 1427 should be tested.

### 2.2.3.6. Paving-grade bitumen 160/220

#### Properties as per EN 12591:2009

The requirements for paving-grade bitumen 160/220 and the results of laboratory testing carried out in 2016 have been shown in table 2.14.

Table 2.14. Properties of 160/220 paving-grade bitumen manufactured in 2016 (results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484)

Property	Test method	Unit	Requirement acc. to EN 12591	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	160 – 220	188
Softening point	EN 1427	°C	35 – 43	38.6
Fraass breaking point	EN 12593	°C	≤ -15	-19
Flash point	EN ISO 2592	°C	≥ 220	313
Solubility	EN 12592	% (m/m)	≥ 99.0	99.95
Change in mass after RTFOT	EN 12607-1	% (m/m)	≤ 1.0	-0.03
Retained penetration after RTFOT	EN 12607-1 EN 1426	%	≥ 37	60
Softening point increase after RTFOT	EN 12607-1 EN 1427	°C	≤ 11	4.4
Kinematic viscosity at 135°C	EN 12595	mm <sup>2</sup> /s	NR	216
Dynamic viscosity at 60°C	EN 12596	Pa*s	NR	50

#### Viscosity dependence on temperature

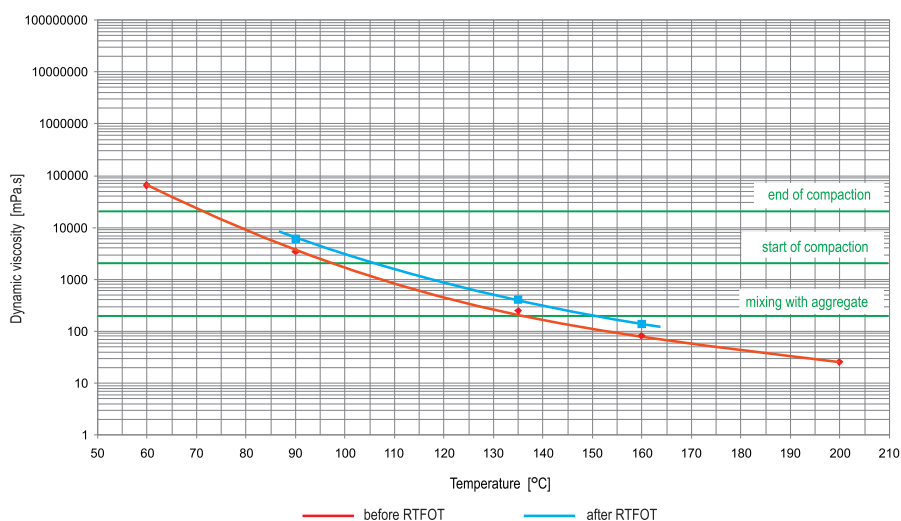


Fig. 2.9. Relation between viscosity and temperature for paving-grade bitumen 160/220

Table 2.15. Example results of viscosity tests on bitumen 160/220 manufactured in 2015. Tests carried out 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	52
	Brookfield viscometer	ASTM D4402 EN 13302	spindle No 21	Pa*s	90°C	3.36
					135°C	0.23
					160°C	0.08
					200°C	0.03
			spindle No 21	Pa*s	90°C after RTFOT	5.80
					135°C after RTFOT	0.34
					160°C after RTFOT	0.14
Kinematic	BS/IP/RF viscometer	EN 12595	—	mm <sup>2</sup> /s	135°C	218

## 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 in high temperatures (up to  $180^{\circ}\text{C}$ ) for over 10 days, it is recommended to inspect the binder ageing rate before the use of bitumen for the production of asphalt mixtures or emulsions. Penetration at  $25^{\circ}\text{C}$  as per EN 1426 or softening point as per EN 1427 should be tested.

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 necessary to store paving-grade bitumen 160/220 for a much longer period than 10 days, it is recommended to reduce the temperature of bitumen and to reheat it before reuse. If a very long storage period is expected without any production of asphalt mixture, 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.

After reheating penetration at  $25^{\circ}\text{C}$  as per EN 1426 or softening point as per EN 1427 should be tested.

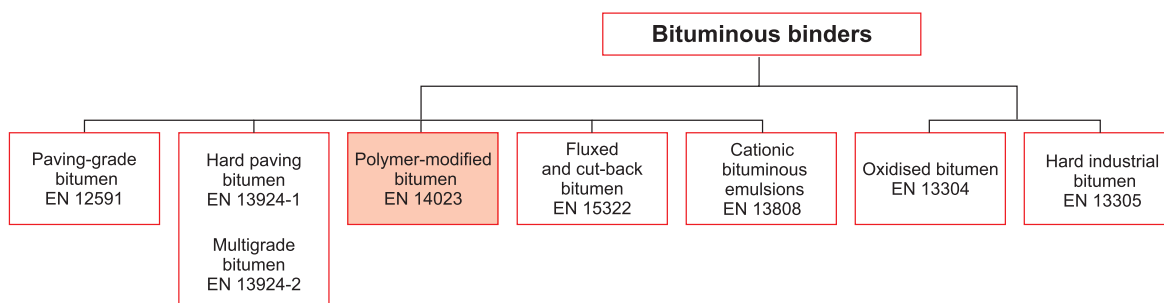
## Chapter 3

### ORBITON POLYMER-MODIFIED BITUMEN ACC. TO EN 14023

#### 3.1. OVERVIEW OF STANDARD EN 14023

##### 3.1.1. Introduction

ORLEN Asphalt has been manufacturing and delivering polymer-modified bitumen in accordance with the requirements of standard EN 14023:2010 since March 2011. Standard EN 14023 is included in the package of European standards on bituminous binders.



*Fig. 3.1. Attribution of European Standards to various types of binders. Standard discussed in this chapter is highlighted in colour*

Standard EN 14023, analogically to standard EN 12591 for paving-grade bitumen, has been developed on the basis of mandate, i.e. standardisation request from the European Commission. Originally, the standard supported essential requirements of the Construction Products Directive 89/106/EEC, the so-called CPD.

As of 01/07/2013, construction products, including bituminous binders, are governed by Regulation No 305/2011 (CPR) of the European Parliament and of the Council, which defines the harmonised conditions of their marketing.

**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 do), but is a classification standard. It means that it provides a set of properties and assigns to them various requirement levels (classes). This enables each Member State to specify its own requirements to be met by bituminous binders used in its territory.



### 3.1.2. Bituminous binder classification

Classification of polymer-modified bitumens manufactured or supplied by ORLEN Asphalt in accordance with European Standard EN 14023 has been shown in table 3.1.

Table 3.1. Classification of polymer-modified bitumen types manufactured to European standard EN 14023

Bituminous binder	Modified bitumen
Reference document	EN 14023:2010
Standard designation of bituminous binder	PMB X/Y-Z
Type of bituminous binder manufactured by ORLEN Asphalt	ORBITON 10/40-65 ORBITON 25/55-55 EXP/DE <sup>1)</sup> ORBITON 25/55-60 ORBITON 25/55-60 EXP/CZ <sup>2)</sup> ORBITON 25/55-65 EXP <sup>3)</sup> ORBITON 45/80-55 ORBITON 45/80-65 ORBITON 45/80-75 SK <sup>4)</sup> ORBITON 65/105-60
1) ORBITON modified bitumen subtype for the German market 2) ORBITON modified bitumen subtype for the Czech market 3) ORBITON modified bitumen subtype for the Czech, Hungarian, Romanian and Slovakian market 4) ORBITON modified bitumen subtype for the Slovakian market  <b>Notes to designations:</b> X – lower penetration limit at 25°C [0.1 mm] acc. to EN 1426, Y – upper penetration limit at 25°C [0.1 mm] acc. to EN 1426, Z – lower softening point (R&B) limit [°C] as per EN 1427. PMB – stands for Polymer Modified Bitumen (usually replaced by the manufacturer's trade name)	

### 3.1.3. National application documents

European Standard EN 14023 assumes that each Member State of CEN shall select the properties and their assigned requirement levels in the so-called Application Documents to the discussed standard.

Standard EN 14023:2010 contains a set of primary properties and a set of additional properties, which are presented in three separate tables:

- Table 1 – properties required for all polymer-modified bitumens;
- Table 2 – properties related to legal regulations or other country-specific conditions;
- Table 3 – additional properties.

The first table contains a set of primary requirements, whereas the second and third tables contain a set of additional requirements. In the described tables of standard EN 14023, each property of polymer modified bitumen (PMB) has been divided into a certain number of classes, from which a desired requirement level can be chosen.

Such structure of class and parameter groups allow each European state to freely select a set of requirements – that means it is possible to create any specification for a specific PMB. Each country may therefore perform an analysis of requirements and create its own application document (National Annex), which will adapt the standard for use on its territory.

In tables 3.2.-3.4. the primary and additional requirements for ORBITON PMB 45/80-55 have been marked off using red frames as an example. Specifications for other polymer modified bitumens have been designed in a similar fashion.

Table 3.2. Principles of classification of polymer-modified bitumen – Properties applicable to all polymer-modified bitumens (EN 14023:2010). Example selection of properties for ORBITON 45/80-55 modified bitumen

Property		Test method	Unit	Requirement 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	10-40	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 tested using ductilometer <sup>a</sup> (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 15°C	≥ 2 at 15°C	≥ 0.5 at 20°C	≥ 0.5 at 25°C	
	Direct stretching <sup>a</sup> (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 <sup>a</sup> (hammering method)	EN 13588	J/cm <sup>2</sup>	≥ 0.7										
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		°C	≤ 8	≤ 10	≤ 12								
	Change in mass after RTFOT <sup>c</sup>		%	≤ 0.3	≤ 0.5	≤ 0.8	≤ 1.0							
Flash point		EN ISO 2592	°C	≥ 250	≥ 235	≥ 220								

a) Only one cohesion testing method 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) RTFOT at 163°C is the primary testing method. In the case of certain polymer-modified bitumens with excessively high viscosity, it is impossible to establish the RTFOT value in the reference temperature of 163°C due to the need to ensure movement of the bitumen layer. In such cases, the determination of the required value shall be made in the temperature of 180°C, in accordance with EN 12607-1.

c) Change in mass may be a positive or negative value.

Table 3.3. Rules for polymer-modified bitumen classification – Additional properties (EN 14023:2010)

Property		Test method	Unit	Classes for national 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 – may be used in situations when no country-specific requirements for a given property at the location of its intended use are in place.

b) TBR. To be declared – may be used in situations when no country-specific requirements for a given property at the location of its intended use are in place; however, the given property has been considered useful for the description of polymer-modified bitumen.

c) If necessary, polymer-modified bitumens should comply with the requirement for elastic recovery at 25°C or 10°C.

Table 3.4. Rules for polymer-modified bitumen classification – Additional properties (EN 14023:2010)

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

a) NR. No requirements – may be used in situations when no country-specific requirements for a given property at the location of its intended use are in place.  
b) Conditions of storage of polymer-modified bitumen shall be provided by the supplier. Homogeneity of polymer-modified bitumens is a necessary requirement.  
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.

Polish requirements for modified bitumen have been presented in the national Annex NA to standard PN-EN 14023:2011 (table 3.5.).

Table 3.5. Division based on types and requirements for polymer-modified bitumen in Poland acc. to national annex NA to standard PN-EN 14023:2011/Ap1:2014-04

Property		Test method	Unit	Type of polymer-modified bitumen											
				PMB 10/40-65		PMB 25/55-60		PMB 45/80-55		PMB 45/80-65		PMB 65/105-60		PMB 90/150-45	
				Scope	Class	Scope	Class	Scope	Class	Scope	Class	Scope	Class	Scope	Class
Primary properties	Penetration at 25°C	EN 1426	0.1 mm	10-40	2	25-55	3	45-80	4	45-80	4	65-105	6	90-150	8
	Softening point	EN 1427	°C	≥ 65	5	≥ 60	6	≥ 55	7	≥ 65	5	≥ 60	6	≥ 45	9
	Force ductility tested using durometer (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	≥ 2 at 10°C	6	≥ 2 at 10°C	6	≥ 3 at 5°C	2	≥ 2 at 10°C	6	≥ 3 at 5°C	2	NR	0
	Change in mass after RTFOT*	EN 12607-1	% m/m	≤ 0.5	3	≤ 0.5	3	≤ 0.5	3	≤ 0.5	3	≤ 0.5	3	≤ 0.5	3
	Retained penetration at 25°C after RTFOT	EN 12607-1 EN 1426	%	≥ 60	7	≥ 60	7	≥ 60	7	≥ 60	7	≥ 60	7	≥ 50	5
	Softening point increase after RTFOT	EN 12607-1 EN 1427	°C	≤ 8	2	≤ 8	2	≤ 8	2	≤ 8	2	≤ 10	3	≤ 10	3
	Flash point	EN ISO 2592	°C	≥ 235	3	≥ 235	3	≥ 235	3	≥ 235	3	≥ 235	3	≥ 235	3
Additional properties	Fraass breaking point	EN 12593	°C	≤ -5	3	≤ -10	5	≤ -15	7	≤ -15	7	≤ -15	7	≤ -18	8
	Elastic recovery at 25°C	EN 13398	%	≥ 60	4	≥ 60	4	≥ 70	3	≥ 80	3	≥ 70	3	≥ 50	5
	Elastic recovery at 10°C	EN 13398	%	NR	0	NR	0	NR	0	NR	0	NR	0	NR	0
	Service temperature range	EN 14023	°C	NR	0	NR	0	NR	0	NR	0	NR	0	NR	0
	Softening point drop after RTFOT	EN 12607-1 EN 1427	°C	TBR	1	TBR	1	TBR	1	TBR	1	TBR	1	TBR	1
	Elastic recovery at 25°C after RTFOT	EN 12607-1 EN 13398	%	≥ 50	4	≥ 50	4	≥ 50	4	≥ 60	3	≥ 60	3	≥ 50	4
	Elastic recovery at 10°C after RTFOT	EN 12607-1 EN 13398	%	NR	0	NR	0	NR	0	NR	0	NR	0	NR	0
	Storage stability – difference in softening point	EN 13399 EN 1427	°C	≤ 5	2	≤ 5	2	≤ 5	2	≤ 5	2	≤ 5	2	≤ 5	2
	Storage stability – difference in penetration	EN 13399 EN 1426	0.1 mm	NR	0	NR	0	NR	0	NR	0	NR	0	NR	0

\* Change in mass may be a positive or negative value  
NR – No Requirement  
TBR – To Be Reported`

### 3.1.4. Evaluation of Conformity

The conformity of the properties of modified bitumen with the requirements of standard EN 14023 and with values provided therein (including classes) shall be demonstrated by:

- initial type tests,
- Factory Production Control (FPC).


The standard obliges the manufacturer to implement, document and support the Factory Production Control. The FPC system should comprise of procedures, regular inspections and tests, while the results shall be used for the finished product quality assessment. This chapter of the standard contains requirements related to the verification and preservation of manufacturing equipment and devices. It also presents different methods of monitoring of properties, namely:

- all properties, according to the provision related to type tests, shall be subject to testing at least once in a year;
- regular monitoring of product quality should consist of the verification of type and frequency of monitoring, should be documented and should ensure that the properties remain essentially similar to those defined during the initial type tests.

Modified bitumen types used for the construction of roads, airports and other road traffic-carrying surfaces are covered by a “2+” conformity assessment system, which requires every manufacturer to implement the Factory Production Control system, and this implementation needs to be certified by a FPC Certificate issued by a notified body. Numbers of FPC Certificates for production units of ORLEN in Poland have been specified in Chapter 1.

Annex ZA also includes the procedure for modified bitumen conformity assessment, the division of responsibilities between the manufacturer and the notified body, a chapter on the certification and declaration of performance, CE marking and labelling.

Figure 3.2 shows example information accompanying the CE marking of modified bitumen ORBITON 45/80-55 manufactured by ORLEN Asfalt in 2015.

 1434	
ORLEN Asphalt Sp. z o.o. 09-400 Płock, ul. Łukasiewicza 39 Poland  14  5/CPR/2015	
PN-EN 14023:2011 (EN 14023:2010)	
Polymer-modified bitumen:	ORBITON 45/80-55
Penetration at 25°C	45-80 x 0.1mm
Softening point	≥ 55°C
Elastic recovery at 25°C	≥ 70%
Fraass breaking point	≤ -15°C
Flash point	≥ 235°C
Force ductility (50 mm/min)	≥ 3 J/cm² at 5°C
Service temperature range	NR
<b>Resistance to hardening by RTFOT</b>	
Change of mass after RTFOT	≤ 0.5%
Softening point increase after RTFOT	≤ 8°C
Softening point drop after RTFOT	≤ 2°C
Retained penetration at 25°C after RTFOT	≥ 60%
Elastic recovery at 25°C after RTFOT	≥ 50%
<b>Storage stability</b>	
Softening point difference	≤ 5°C
Difference in penetration at 25°C	NR

CE conformity marking, comprising  
the CE mark as indicated  
in Directive 93/68/EEC

Identification number of the notified body

Name or identification mark  
and the manufacturer's registered address

Two last digits of the year  
in which the marking was placed

Declaration of performance number

European Standard number

Product description and information  
about properties subject  
to laboratory control

Fig. 3.2. CE marking for modified bitumen 45/80-55 manufactured by ORLEN Asphalt in 2015

## 3.2. GENERAL DESCRIPTION OF POLYMER-MODIFIED BITUMENS

### 3.2.1. Description

Polymer-Modified Bitumens represent a group of road paving binders designed specifically to improve the durability of bitumen pavements and to counteract the most frequent road problems, such as viscoplastic deformations of roads carrying heavy traffic, low-temperature cracking-up of wearing courses during winter, and fatigue-related cracks. The application of modifier – the SBS (Styrene-Butadiene-Styrene) elastomer – in the production process helps to achieve substantial benefits in terms of bitumen properties in both high and low temperatures. Asphalt pavements produced using modified bitumen are more durable than pavements made using paving-grade bitumens.

Key differences between paving-grade bitumens and modified bitumens with reference to the two primary binder parameters, namely penetration and softening point, have been schematically shown in Figure 3.3.

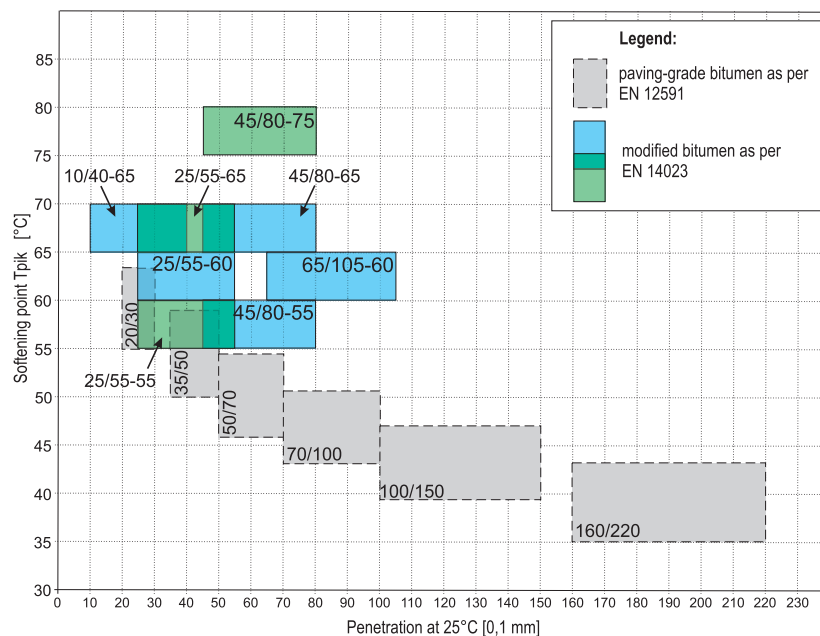


Fig. 3.3. Schematic comparison of paving grade and modified bitumen types as a function of penetration at 25°C and softening point  $T_{R\&B}$

This Handbook contains a detailed description of ORBITON modified bitumens manufactured according to standard PN-EN 14023:2011, with the intention of their application in the road building industry in Poland. ORLEN Asphalt also produces ORBITON PMB adapted to local requirements of countries to which ORBITON products are exported (e.g. Romania, Lithuania, Latvia, Czech Republic, Slovakia, Germany, Hungary). The types of produced bitumens have been shown in Table 3.6.

Table 3.6. Types of modified bitumens manufactured by ORLEN Asphalt

Types of ORBITON modified bitumens as per annex NA for Poland	Types of ORBITON modified bitumens as per annexes NA for other EU countries
ORBITON 10/40-65 ORBITON 25/55-60 ORBITON 45/80-55 ORBITON 45/80-65 ORBITON 65/105-60	ORBITON 25/55-55 EXP <sup>1)</sup> ORBITON 25/55-60 EXP <sup>2)</sup> ORBITON 25/55-65 EXP <sup>3)</sup> ORBITON 45/80-75 SK <sup>4)</sup>
1) ORBITON modified bitumen subtype for the German market 2) ORBITON modified bitumen subtype for the Czech market 3) ORBITON modified bitumen subtype for the Czech, Hungarian, Romanian and Slovakian market 4) ORBITON modified bitumen subtype for the Slovakian market	

### 3.2.2. Intended use

ORBITON modified bitumens represent a group of binders intended for use in pavements designed to carry heavy traffic, or in special pavements (on bridges, thin wearing courses, etc.). Well-designed asphalt mixtures made using these bitumens demonstrate better properties than their counterparts characterised by similar hardness (paving-grade bitumens).

The range of applications for modified bitumens is very broad, both in terms of asphalt mixture types and road traffic categories. The typical applications of individual types of modified bitumen have been shown below.

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

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

**ORBITON 25/55-65 modified bitumen** is a binder that has similar properties to ORBITON 25/55-60, the only difference being that its softening point is 5°C higher. It can be used to good effect in courses which require high resistance to permanent deformation, i.e. AC asphalt concrete base courses and binder courses, and high-rigidity AC EME asphalt concrete. It can also be used in wearing courses at sections designed to carry heavy traffic and in mastic asphalt (MA) mixtures.

**ORBITON 45/80-55 modified bitumen** is one of the most popular modified bitumen types. It is designed for use in all types of asphalt mixtures for wearing courses (AC, SMA, BBTM).

**ORBITON 45/80-65 modified bitumen** is designed for use in wearing courses and for special applications. It is characterised by very high elasticity, high softening point and favourable characteristics in low temperatures. High polymer content makes it difficult to incorporate in thin layers in adverse weather conditions (quick stiffening of layer, compaction problems). High softening point and high modification level render it suitable for application at locations where high tensile strength and fatigue resistance are necessary in combination with excellent low-temperature properties. ORBITON 45/80-65 modified bitumen has been primarily designed for application in wearing courses, as well as for porous asphalt PA mixtures.

**ORBITON 65/105-60 modified bitumen** is designed for use in thin-layered hot mix wearing courses, within mixtures having a good mineral skeleton. It is produced using soft base bitumen with a high polymer content, which allows to obtain a product with excellent low-temperature properties and elasticity.

ORBITON 65/105-60 is characterised by higher penetration at 25°C in comparison to modified bitumen 45/80-65, and at the same time demonstrates high levels of elasticity and flexibility. The combination of those features renders the product a very good binder for thin-layered gap grading mixes. Such applications include porous asphalt mixes, 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. This binder can also be used in bridge deck mixes, whenever excellent elasticity of the binder is required.

<sup>1)</sup> AC EME is the designation for Asphalt Concrete with a high stiffness modulus. Other designations of this mixture are WMS (in Poland), EME Enrobé à Module Elevé in France) or HMB (High Modulus Base in USA and UK).

### 3.2.3. Properties

The following parts of this chapter introduce a set of polymer-modified bitumen properties specified according to EN 14023, and additional information obtained via tests carried out according to the American *Superpave* and *Superpave plus* methods. The chapter also contains the classification of modified bitumen types according to traffic loads, developed on the basis of results of MSCR tests (a detailed description of the MSCR test can be found in Chapter 7).

This chapter also specifies approximate process temperatures for bitumen application in asphalt mixtures and data on the relation between viscosity and temperature.

#### 3.2.3.1. ORBITON 10/40-65 modified bitumen

##### Properties as per EN 14023:2010

The requirements for ORBITON 10/40-65 modified bitumen and the results of laboratory testing carried out in 2016 have been shown in table 3.7.

Table 3.7. Requirements and properties of ORBITON 10/40-65 modified bitumen manufactured in 2016 (*results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484*)

Property	Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	10 ÷ 40	30
R&B softening point	EN 1427	°C	≥ 65	72.6
Elastic recovery at 25°C	EN 13398	%	≥ 60	79
Fraass breaking point	EN 12593	°C	≤ -5	-16
Flash point	EN ISO 2592	°C	≥ 235	> 245
Force ductility (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	≥ 2 at 10°C	5.0
Change in mass after RTFOT	EN 12607-1	%	≤ 0.5	0.07
Softening point increase after RTFOT	EN 1427	°C	≤ 8	3.4
Retained penetration after RTFOT	EN 1426	%	≥ 60	79
Elastic recovery at 25°C after RTFOT	EN 12607-1 EN 13398	%	≥ 50	75
Storage stability: Softening point difference	EN 13399 EN 1427	°C	≤ 5	1.1
Softening point decrease after RTFOT	EN 12607-1 EN 1427	°C	TBR <sup>a</sup>	0.0
a) TBR – To Be Reported				



## Properties acc. to Superpave

In the following section, we present the properties of ORBITON 10/40-65 modified bitumen specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

- **Performance Grade**, 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)	$T_{crit} = 83.8^{\circ}\text{C}$
– $G^*\sin\delta = 5000$ kPa (bitumen after RTFOT and PAV)	$T_{crit} = 19.5^{\circ}\text{C}$

- **Low critical temperatures** (AASHTO PP 42; EN 14771):

– temperature at $S(60) = 300$ MPa	$T(S)_{60} = -17.2^{\circ}\text{C}$
– temperature at $m(60) = 0.3$	$T(m)_{60} = -8.6^{\circ}\text{C}$
– stiffness at $-16^{\circ}\text{C}$	$S(T)_{-16} = 271.5$ MPa

## Process temperatures

At laboratory:	
Sample compaction temperature (Marshall compactor or gyratory press)	150–155°C
At mixing plant:	
Bitumen pumping temperature	>150°C
Temperature of bitumen for asphalt mixture production	180–190°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 4h)	<220°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 2h)	<230°C
<b>Attention:</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 asphalt mixture (spreader's hopper)	160°C

## Viscosity dependence on temperature

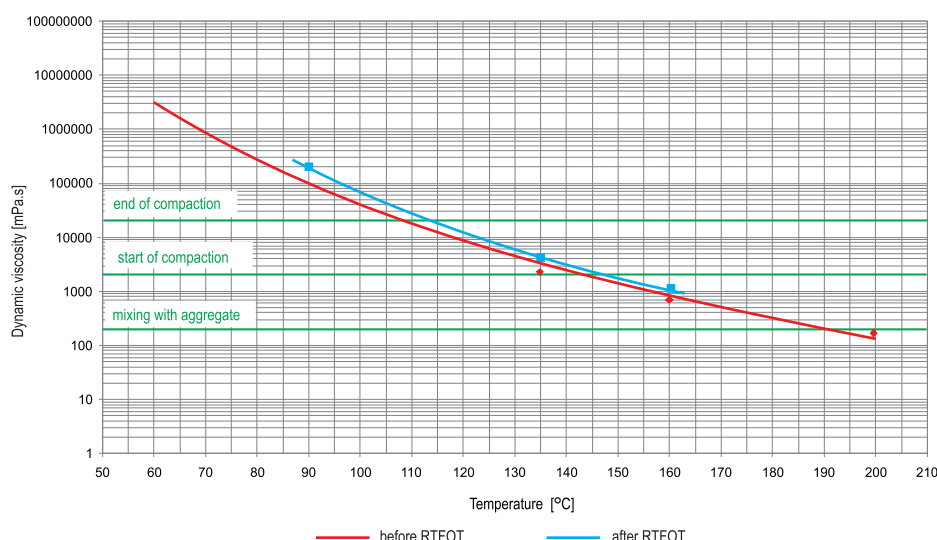


Fig. 3.4. Relation between viscosity and temperature for ORBITON 10/40-65 modified bitumen

Table 3.8. Example results of viscosity tests on ORBITON 10/40-65 modified bitumen manufactured in 2013. Tests carried out 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 viscometer	ASTM D4402 PN-EN 13302	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

### Polymer structural properties

- polymer dispersion code as per 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 mixture production: 7 days

It is recommended to conduct basic inspection tests of modified bitumen properties after 5 days, in order to make sure that the product has not lost its properties due to the loss of stability 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 as per EN 1426
- softening point as per EN 1427
- elastic recovery at 25°C as per 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)

Storage of modified bitumen for the duration of more than 7 days is not recommended. 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.

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

#### Properties as per EN 14023:2010

The requirements for ORBITON 25/55-60 modified bitumen and the results of laboratory testing carried out in 2016 have been shown in table 3.9.

Table 3.9. Requirements and properties of ORBITON 25/55-60 modified bitumen manufactured in 2016 (results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484)

Property	Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	25 ÷ 55	40
R&B softening point	EN 1427	°C	≥ 60	63.7
Elastic recovery at 25°C	EN 13398	%	≥ 60	77
Fraass breaking point	EN 12593	°C	≤ -10	-16
Flash point	EN ISO 2592	°C	≥ 235	327
Force ductility (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	≥ 2 at 10°C	4.5
Change of mass after RTFOT	EN 12607-1	%	≤ 0.5	-0.01
Softening point increase after RTFOT	EN 1427	°C	≤ 8	5.6
Retained penetration after RTFOT	EN 1426	%	≥ 60	73
Elastic recovery at 25°C after RTFOT	EN 12607-1 EN 13398	%	≥ 50	71
Storage stability: Softening point difference	EN 13399 EN 1427	°C	≤ 5	1.4
Storage stability: Difference in penetration at 25°C	EN 13399 EN 1427	0.1 mm	NR <sup>b</sup>	0.4
Softening point decrease after RTFOT	EN 12607-1 EN 1427	°C	TBR <sup>a</sup>	0.0
a) TBR – To Be Reported b) NR – No Requirement				

#### Properties acc. to Superpave

In the following section, we present the properties of ORBITON 25/55-60 modified bitumen specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

- **Performance Grade**, classification acc. to AASHTO MP 1: **PG 76-22**

- **High critical temperatures** (AASHTO T 315):

- $G^*/\sin\delta = 1$  kPa (unaged bitumen)
- $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)
- $G^* \cdot \sin\delta = 5000$  kPa (bitumen after RTFOT and PAV)

$$T_{crit} = 83.1^{\circ}\text{C}$$

$$T_{crit} = 80.5^{\circ}\text{C}$$

$$T_{crit} = 22.0^{\circ}\text{C}$$

- **Low critical temperatures** (AASHTO PP 42; EN 14771):

- temperature at  $S(60) = 300$  MPa
- temperature at  $m(60) = 0.3$
- 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 of bitumens based on the MSCR method

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.059	0.150	0.340	0.050	0.207	0.722
$J_{nr}$ 3.2 kPa	0.061	0.163	0.408	0.052	0.219	0.946
$J_{nr}$ diff	3.6	8.6	20.1	3.2	6.1	31.1
R 0.1 kPa	71.5	65.6	58.8	70.7	64.8	56.9
R 3.2 kPa	70.6	63.3	52.4	69.9	63.8	47.4
R diff	1.3	3.5	10.9	1.1	1.7	16.7
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Extreme	not subject to classification		

### Process temperatures

At laboratory:	
Sample compaction temperature (Marshall compactor or gyratory press)	145–150°C
At mixing plant:	
Bitumen pumping temperature	>150°C
Temperature of bitumen for asphalt mixture production	175–185°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 4h)	<220°C
Mastic asphalt temperature in the mixer (asphalt mixture storage time of up to 2h)	<230°C
<b>Attention:</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 asphalt mixture (spreader's hopper)	155°C

### Viscosity dependence on temperature

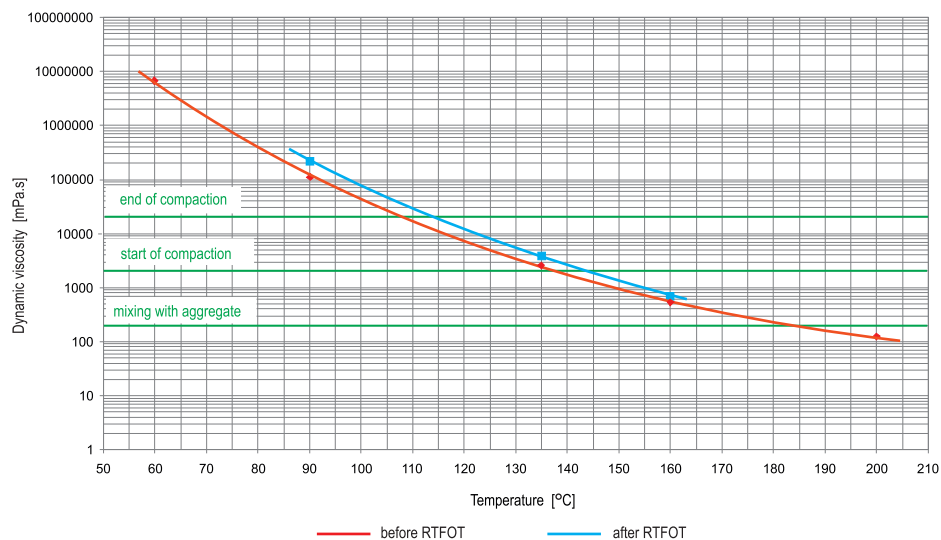


Fig. 3.5. Relation between viscosity and temperature for ORBITON 25/55-60 modified bitumen

Table 3.10. Example results of viscosity tests on ORBITON 25/55-60 modified bitumen manufactured in 2015. Tests carried out 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	6 250
	Brookfield viscometer	ASTM D4402 PN-EN 13302	spindle No 21	Pa•s	90°C	112.00
					135°C	2.14
					160°C	0.63
					200°C	0.15
			spindle No 27	Pa•s	90°C after RTFOT	210.00
					135°C after RTFOT	3.37
					160°C after RTFOT	0.87

## Polymer structural properties

- polymer dispersion code as per 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 mixture production: 7 days

It is recommended to conduct basic inspection tests of modified bitumen properties after 5 days, in order to make sure that the product has not lost its properties due to the loss of stability 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 as per EN 1426
- softening point as per EN 1427
- elastic recovery at 25°C as per 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)

Storage of modified bitumen for the duration of more than 7 days is not recommended. 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.

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

#### Properties as per EN 14023:2010

The requirements for ORBITON 45/80-55 modified bitumen and the results of laboratory testing carried out in 2016 have been shown in table 3.11.

Table 3.11. Requirements and properties of ORBITON 45/80-55 modified bitumen manufactured in 2016 (*results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484*)

Property	Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	45-80	62
Softening point	EN 1427	°C	≥ 55	61.6
Elastic recovery at 25°C	EN 13398	%	≥ 70	83
Fraass breaking point	EN 12593	°C	≤ -15	-18
Flash point	EN ISO 2592	°C	≥ 235	325
Force ductility at 5°C (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	≥ 3 at 5°C	6.7
Change in mass after RTFOT	EN 12607-1	%	≤ 0.5	-0.06
Softening point increase after RTFOT	EN 1427	°C	≤ 8	4.0
Retained penetration after RTFOT	EN 1426	%	≥ 60	71
Elastic recovery at 25°C after RTFOT	EN 12607-1 EN 13398	%	≥ 50	81
Storage stability: Softening point difference	EN 13399 EN 1427	°C	≤ 5	0.1
Storage stability: Difference in penetration at 25°C	EN 13399 EN 1427	0.1 mm	NR <sup>b</sup>	0.8
Softening point decrease after RTFOT	EN 12607-1 EN 1427	°C	TBR <sup>a</sup>	0.0
a) TBR – To Be Reported b) NR – No Requirement				

#### Properties acc. to Superpave

In the following section, we present the properties of ORBITON 45/80-55 modified bitumen specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

- **Performance Grade**, 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)
- $G^* \cdot \sin\delta = 5000$  kPa (bitumen after RTFOT and PAV)

$$T_{crit} = 74.5^{\circ}\text{C}$$

$$T_{crit} = 72.9^{\circ}\text{C}$$

$$T_{crit} = 17.7^{\circ}\text{C}$$

- **Low critical temperatures** (AASHTO PP 42; EN 14771):

- temperature at  $S(60) = 300$  MPa
- temperature at  $m(60) = 0.3$
- stiffness at  $-16^{\circ}\text{C}$

$$T(S)_{60} = -18.1^{\circ}\text{C}$$

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

$$S(T)_{-16} = 242 \text{ MPa}$$

• Results and classification of bitumens based on the MSCR method

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.156	0.351	0.445	0.114	0.347	1.030
$J_{nr}$ 3.2 kPa	<b>0.169</b>	<b>0.384</b>	<b>0.515</b>	0.121	0.344	1.540
$J_{nr}$ diff	8.7	9.4	15.8	5.7	1.0	49.8
R 0.1 kPa	75.5	71.2	67.9	76.6	76.3	71.8
R 3.2 kPa	74.0	69.5	66.3	75.6	77.0	58.7
R diff	2.1	2.4	5.0	1.2	-0.8	18.2
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Very Heavy	not subject to classification		

## Process temperatures

<b>At laboratory:</b>	
Sample compaction temperature (Marshall compactor or gyratory press)	145–150°C
<b>At mixing plant:</b>	
Bitumen pumping temperature	>150°C
Temperature of bitumen for asphalt mixture production	175–185°C
<b>At site:</b>	
Minimum temperature of the supplied asphalt mixture (spreader's hopper)	155°C

## Viscosity dependence on temperature

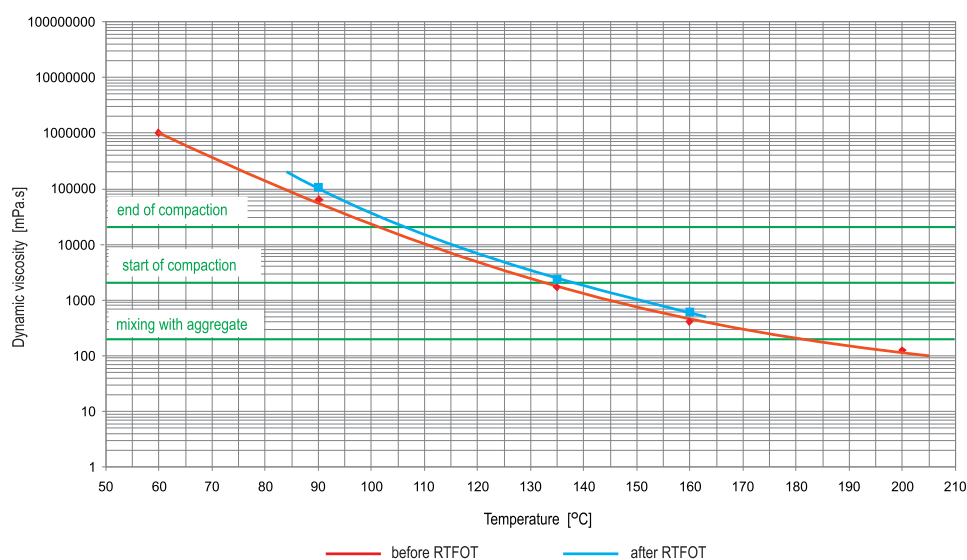


Fig. 3.6. Relation between viscosity and temperature for ORBITON 45/80-55 modified bitumen

Table 3.12. Example results of viscosity tests on ORBITON 45/80-55 modified bitumen manufactured in 2015. Tests carried out 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	569
	Brookfield viscometer	ASTM D4402 PN-EN 13302	spindle No 27	Pa•s	90°C	69.33
					135°C	1.47
					160°C	0.44
					200°C	0.12
			spindle No 27	Pa•s	90°C after RTFOT	108.00
					135°C after RTFOT	1.97
					160°C after RTFOT	0.56

### Polymer structural properties

- polymer dispersion code as per 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 mixture production: 7 days

It is recommended to conduct basic inspection tests of modified bitumen properties after 5 days, in order to make sure that the product has not lost its properties due to the loss of stability 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 as per EN 1426
- softening point as per EN 1427
- elastic recovery at 25°C as per 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)

Storage of modified bitumen for the duration of more than 7 days is not recommended. 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)

The storage of this binder cooled down to ambient temperature (e.g. over winter) is not recommended, however if this is necessary, it a certain time period needed for the fluidisation of the binder should be provided.

After reheating and homogenization, penetration at 25°C as per EN 1426, softening point as per EN 1427 and elastic recovery as per EN 13398 should be tested.



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

#### Properties as per EN 14023:2010

The requirements for ORBITON 45/80-65 modified bitumen and the results of laboratory testing carried out in 2016 have been shown in table 3.13.

Table 3.13. Requirements and properties of ORBITON 45/80-65 modified bitumen manufactured in 2016 (*results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484*)

Property	Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	45-80	55
Softening point	EN 1427	°C	≥ 65	75.4
Elastic recovery at 25°C	EN 13398	%	≥ 80	87
Fraass breaking point	EN 12593	°C	≤ -15	-20
Flash point	EN ISO 2592	°C	≥ 235	327
Force ductility at 10°C (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	≥ 2 at 10°C	5.0
Change of mass after RTFOT	EN 12607-1	%	≤ 0.5	0.01
Softening point increase after RTFOT	EN 1427	°C	≤ 8	3.7
Retained penetration after RTFOT	EN 1426	%	≥ 60	78
Elastic recovery at 25°C after RTFOT	EN 12607-1 EN 13398	%	≥ 60	86
Storage stability: Softening point difference	EN 13399 EN 1427	°C	≤ 5	1.6
Softening point decrease after RTFOT	EN 12607-1 EN 1427	°C	TBR <sup>a</sup>	0.7
Storage stability: Difference in penetration at 25°C	EN 13399 EN 1427	0.1 mm	NR <sup>b</sup>	—
a) TBR – To Be Reported b) NR – No Requirement				

#### Properties acc. to Superpave

In the following section, we present the properties of ORBITON 45/80-65 modified bitumen specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

- **Performance Grade**, classification acc. to AASHTO MP 1: **PG 76-22**

- **High critical temperatures** (AASHTO T 315):

- $G^*/\sin\delta = 1$  kPa (unaged bitumen)
- $G^*/\sin\delta = 2.2$  kPa (bitumen after RTFOT)
- $G^* \cdot \sin\delta = 5000$  kPa (bitumen after RTFOT and PAV)

$$\begin{aligned} T_{crit} &= 83.2^{\circ}\text{C} \\ T_{crit} &= 77.7^{\circ}\text{C} \\ T_{crit} &= 17.6^{\circ}\text{C} \end{aligned}$$

- **Low critical temperatures** (AASHTO PP 42; EN 14771):

- temperature at  $S(60) = 300$  MPa
- temperature at  $m(60) = 0.3$
- stiffness at  $-16^{\circ}\text{C}$

$$\begin{aligned} T(S)_{60} &= -18.3^{\circ}\text{C} \\ T(m)_{60} &= -14.3^{\circ}\text{C} \\ S(T)_{-16} &= 235 \text{ MPa} \end{aligned}$$

- Results and classification of bitumens based on the MSCR method

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.099	0.188	0.369	0.042	0.081	0.223
$J_{nr}$ 3.2 kPa	0.110	0.207	0.474	0.051	0.099	0.313
$J_{nr}$ diff	10.8	10.2	28.7	20.4	22.2	40.7
R 0.1 kPa	83.2	82.1	80.0	89.6	92.1	91.1
R 3.2 kPa	82.3	81.2	76.0	88.1	90.9	87.3
R diff	1.1	1.0	5.0	1.7	1.3	4.1
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Extreme	not subject to classification		

## Process temperatures

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

## Viscosity dependence on temperature

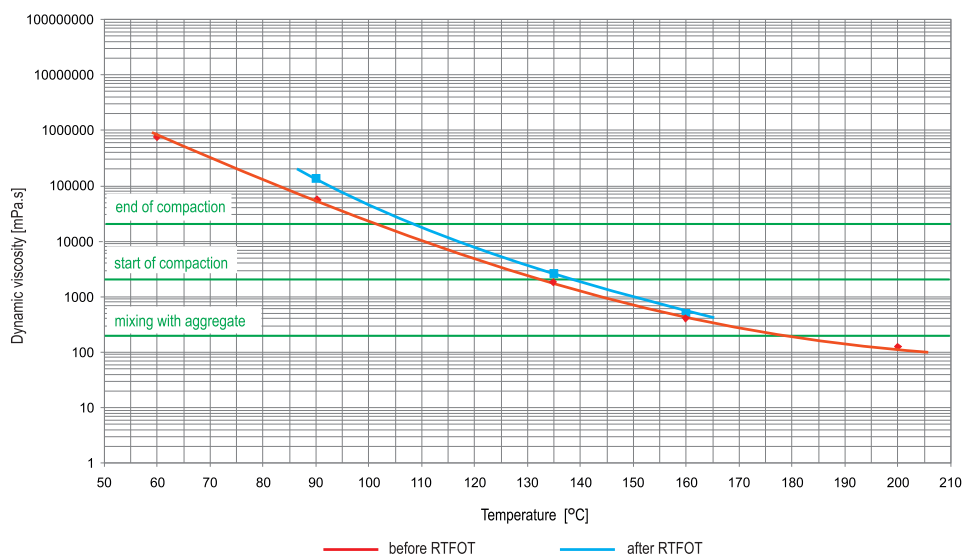


Fig. 3.7. Relation between viscosity and temperature for ORBITON 45/80-65 modified bitumen

Table 3.14. Example results of viscosity tests on ORBITON 45/80-65 modified bitumen manufactured in 2015. Tests carried out 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	647
	Brookfield viscometer	ASTM D4402 EN 13302	spindle No 21	Pa•s	90°C	45.70
					135°C	1.59
					160°C	0.46
					200°C	0.16
			spindle No 27	Pa•s	90°C after RTFOT	136.00
					135°C after RTFOT	2.05
					160°C after RTFOT	0.55

## Polymer structural properties

- polymer dispersion code as per 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 mixture production: 7 days

It is recommended to conduct basic inspection tests of modified bitumen properties after 5 days, in order to make sure that the product has not lost its properties due to the loss of stability 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 as per EN 1426
- softening point as per EN 1427
- elastic recovery at 25°C as per 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)

Storage of modified bitumen for the duration of more than 7 days is not recommended. 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)

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.

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

#### Properties as per EN 14023:2010

The requirements for ORBITON 65/105-60 modified bitumen and the results of laboratory testing carried out in 2016 have been shown in table 3.15.

Table 3.15. Requirements and properties of ORBITON 65/105-60 modified bitumen manufactured in 2016 (*results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484*)

Property	Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C	EN 1426	0.1 mm	65-105	74
Softening point	EN 1427	°C	≥ 60	64.0
Elastic recovery at 25°C	EN 13398	%	≥ 70	80
Fraass breaking point	EN 12593	°C	≤ -15	-18
Flash point	EN ISO 2592	°C	≥ 235	>245
Force ductility (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	≥ 3 at 5°C	5.2
Change of mass after RTFOT	EN 12607-1	%	≤ 0.5	-0.03
Softening point increase after RTFOT	EN 1427	°C	≤ 10	4.2
Retained penetration after RTFOT	EN 1426	%	≥ 60	73
Elastic recovery at 25°C after RTFOT	EN 12607-1 EN 13398	%	≥ 60	74
Storage stability: Softening point difference	EN 13399 EN 1427	°C	≤ 5	2.6
Softening point decrease after RTFOT	EN 12607-1 EN 1427	°C	TBR <sup>a</sup>	0.0
a) TBR – To Be Reported				

#### Properties acc. to Superpave

In the following section, we present the properties of ORBITON 65/105-60 modified bitumen specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

- **Performance Grade**, classification acc. to AASHTO MP 1: **PPG 64-28**
- **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)  $T_{crit} = 69.2^{\circ}\text{C}$
  - $G^*\cdot\sin\delta = 5000$  kPa (bitumen after RTFOT and PAV)  $T_{crit} = 13.6^{\circ}\text{C}$
- **Low critical temperatures** (AASHTO PP 42; EN 14771):
  - temperature at  $S(60) = 300$  MPa  $T(S)_{60} = -20.5^{\circ}\text{C}$
  - temperature at  $m(60) = 0.3$   $T(m)_{60} = -20.6^{\circ}\text{C}$
  - stiffness at  $-16^{\circ}\text{C}$   $S(T)_{-16} = 172$  MPa

## Process temperatures

<b>At laboratory:</b>	
Sample compaction temperature (Marshall compactor or gyratory press)	145–150°C
<b>At mixing plant:</b>	
Bitumen pumping temperature	> 150°C
Temperature of bitumen for asphalt mixture production	175–185°C
<b>At site:</b>	
Minimum temperature of the supplied asphalt mixture (spreader's hopper)	160°C

## Viscosity dependence on temperature

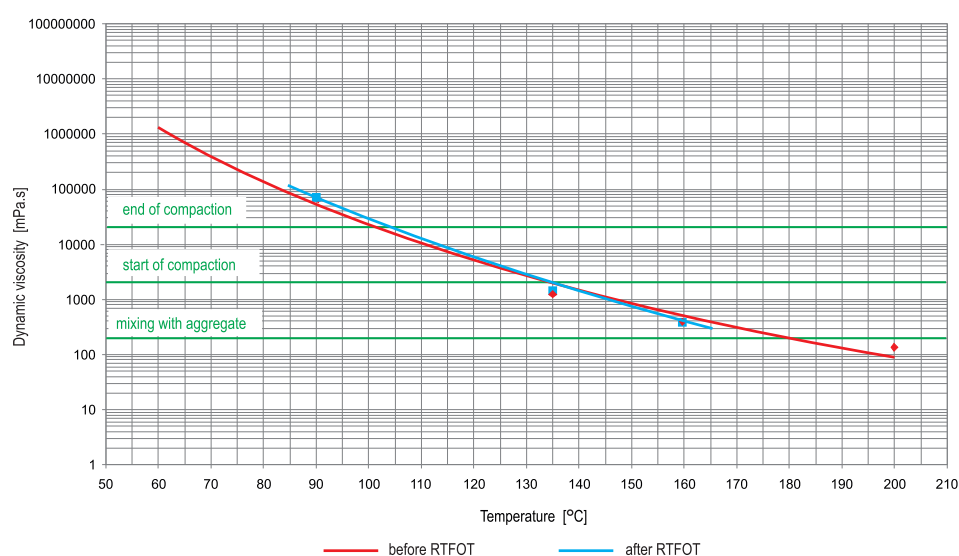


Fig. 3.8. Relation between viscosity and temperature for ORBITON 65/105-60 modified bitumen

Table 3.16. Example results of viscosity tests on ORBITON 65/105-60 modified bitumen manufactured in 2013. Tests carried out 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 viscometer	ASTM D4402 PN-EN 13302	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

## Polymer structural properties

- polymer dispersion code as per 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 mixture production: 7 days

It is recommended to conduct basic inspection tests of modified bitumen properties after 5 days, in order to make sure that the product has not lost its properties due to the loss of stability 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 as per EN 1426
- softening point as per EN 1427
- elastic recovery at 25°C as per 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)

Storage of modified bitumen for the duration of more than 7 days is not recommended. 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)

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.

## Chapter 4

### HIGHLY MODIFIED BITUMEN ORBITON HIMA ACC. TO PN-EN 14023

Since 2011, the Technology, Research and Development Department of ORLEN Asphalt has been conducting research and development works intended to develop a new range of products. Laboratory tests and production trials have resulted in the development of innovative bituminous binders – bitumens highly modified with polymers.

The first experimental section of road pavement made using highly modified bitumen was completed in Poland in October 2013. The section was located on a road managed by the Road Administration in Katowice (Silesian Voivodeship DoT). It was the 5th road section in Europe made using highly modified binder that contains the special KRATON polymer.

In April 2014, the Polish Standardisation Committee published, in the form of a National Annex to standard PN-EN 14023 the revised requirements for polymer-modified bitumens, including an additional table containing requirements for highly modified bitumens. As a result, highly modified bitumens under the trade name of ORBITON HIMA, were officially included in ORLEN Asphalt's product portfolio in May 2014. These binders are produced according to the requirements of a harmonised standard, they are marked with the CE sign.

Chapter 4 of this Handbook presents a description, principles of operation, laboratory test results and experience obtained from experimental sections related to highly modified bitumens – the new binders manufactured by ORLEN Asphalt.

#### 4.1. INTRODUCTION

##### 4.1.1. General Description of Highly Modified Binders

Research conducted by numerous academic centres all over the world has corroborated the claim that higher polymer content in bitumen produces additional quality benefits, substantially contributing to the improvement of durability of asphalt pavements. The effect of improvement of durability can be observed especially in terms of the resistance of the mixture to cracking up, rutting and fatigue. Of particular significance is the achievement of the effect of reversal of phase, i.e. of the moment when the polymer phase becomes a constant phase in PMB (usually with SBS polymer mass content of more than 6.5-7.0%). However, such a significant quantity of typical type of SBS for binder modification carries with it specific technical consequences for the production and application of modified binder, connected among others with following aspects:

- stability problems in storage and transport of modified bitumen (high risk of polymer separation),
- very high viscosity of PMB, which means that such binders would have to be heated in the mixing plant to a much higher temperature than conventional modified binders with lower polymer quantity,
- major problems with the compaction of the asphalt mixture at the road construction site – due to excessive viscosity, rapid stiffening of the mixture occurs within the course, therefore low compaction ratios are achieved.

The above mentioned limitations of the concept of application of highly modified bitumen in road engineering for many years represented a challenge not only for road binder manufacturers, but also for polymer suppliers. However, research work carried out by the polymer industry has provided a positive outcome, and as a result solutions are available, for several years now, which allow the production of highly-modified bitumen free from the above mentioned shortcomings.

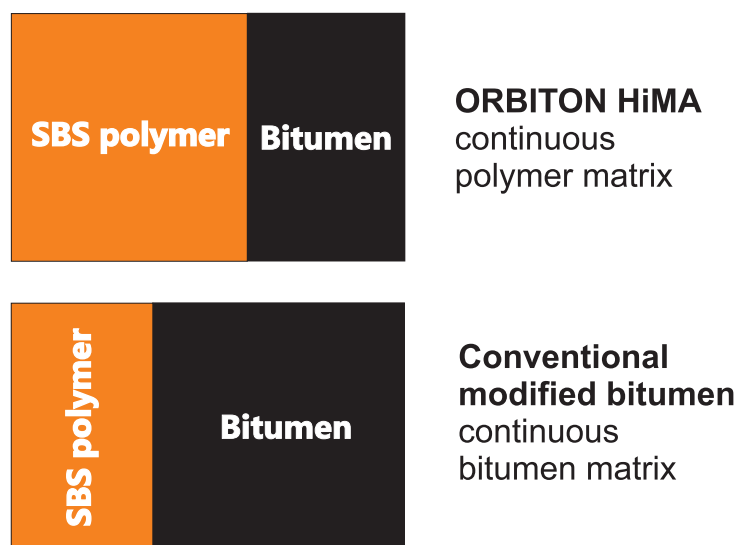
Binders of this type are called **HiMA** – **H**ighly **M**odified **A**sphalt.

Work related to the research and implementation of new highly modified binders have demonstrated that they are products with above-standard functional properties. They are characterised by, among others, very good resistance to rutting, to the effects of water and cold temperatures, and excellent fatigue strength and resistance to cracking.

#### 4.1.2. Operating principles of HiMA

The primary purpose behind highly-modified bitumen is to counteract pavement cracking, ruts, and to increase fatigue resistance of bitumen layers.

To this end we use polymer content in excess of 7% m/m, which leads to phase reversal in the mixing of bitumen with the polymer (Figure 4.1).



*Fig. 4.1. Volumetric proportions between bitumen and polymer in conventional polymer-modified bitumen and highly-modified bitumen*

A continuous polymer network (polymer phase) present in the binder and bituminous mix acts as an elastic reinforcement, which can be clearly demonstrated on the example of limited crack propagation in asphalt mixture courses attributed to highly-modified binders. Figure 4.2 shows the diagrams of three hypothetical cases:

- Figure A: propagation of cracks through the asphalt mixture with a conventional paving-grade binder – in this case, the crack can easily pass through the binder,
- Figure B: propagation of cracks through the asphalt course with conventional modified bitumen with non-continuous polymer network (marked with scattered yellow dots) – in this case, the crack is able to pass (with some delay) through the binder course, finding gaps between the polymer network fragments,
- Figure C: propagation of cracks through the asphalt course with HiMA with continuous polymer network (marked with yellow lines) – in this case, the passage of a crack through the binder course is more difficult, because of a barrier formed by the polymer network.



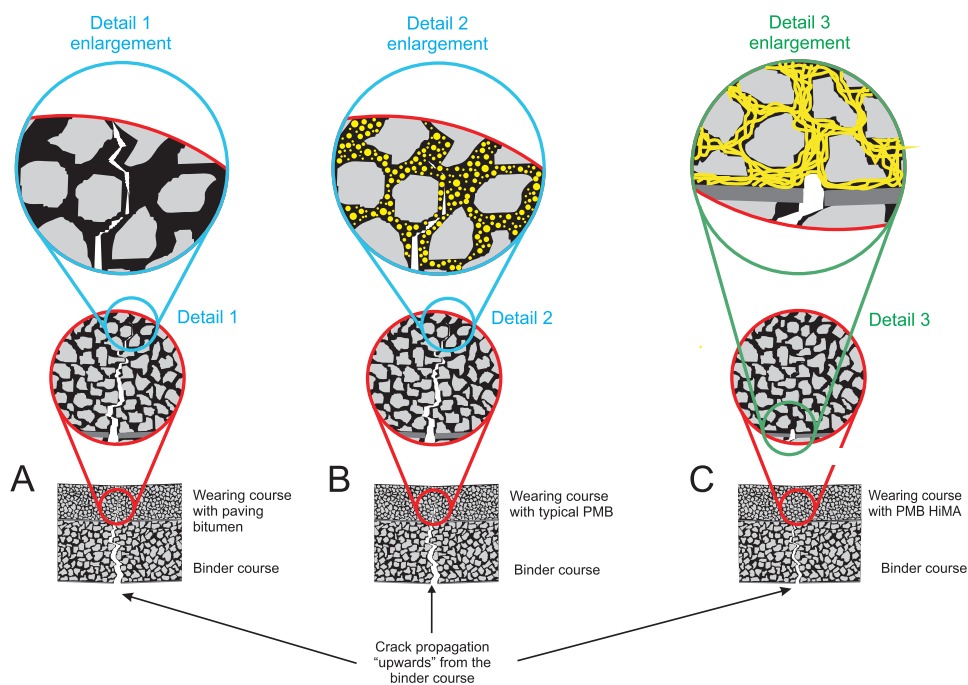


Fig. 4.2. Propagation of cracks through asphalt courses, a) with paving-grade bitumen, b) with modified bitumen, c) with highly-modified bitumen

#### 4.1.3. Principles of Classification of Bitumens Highly Modified with Polymers

All ORBITON HiMA highly modified bitumens are classified according to European Standard PN-EN 14023:2011 „Bitumen and bituminous binders – Specification framework for polymer modified bitumens“. The description of this standard, its requirements, conformity assessment and principles of CE marking of highly modified bitumens are identical to those for conventional modified bitumens, and have been already presented in Chapter 3.

The classification of highly modified bitumens manufactured in accordance with European Standard EN 14023 has been shown in table 4.1.

Table 4.1. Classification of highly modified bitumen types manufactured to European standard PN-EN 14023

Bituminous binder	Highly modified bitumen
Reference document	PN-EN 14023:2011/Ap1:2014-04
Standard designation of bituminous binder	PMB X/Y-Z
Type of bituminous binder supplied by ORLEN Asphalt	ORBITON 25/55-80 HiMA ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA
<b>Notes to designations:</b> X – lower penetration limit at 25°C [0.1 mm] acc. to EN 1426, Y – upper penetration limit at 25°C [0.1 mm] acc. to EN 1426, Z – lower softening point (R&B) limit [°C] as per EN 1427 PMB – stands for Polymer Modified Bitumen (usually replaced by the manufacturer's trade name) ORBITON HiMA – (Highly Modified Asphalt) – the trade name of the bitumen type	

Figure 4.3. displays a Pen25-SP R&B (Penetration at 25°C vs Softening Point Ring&Ball) chart showing how the new products are positioned relative to the paving-grade binders and (conventional) modified binders which have been used to date. A significant increase in the softening point range for all ORBITON HiMA products can be clearly seen, which is a direct result of high polymer content in these bitumen types.

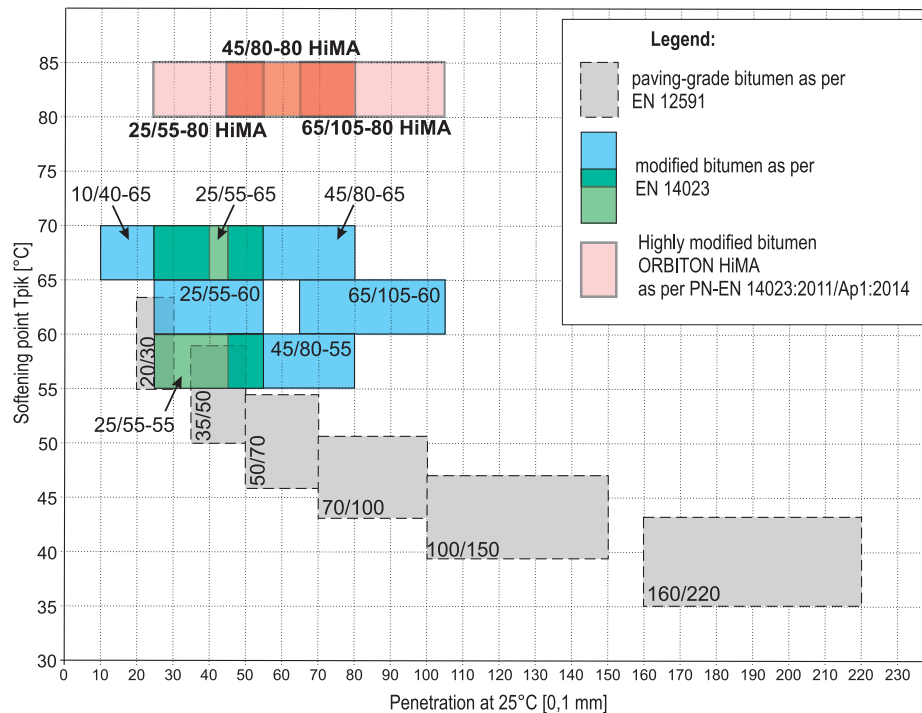


Fig. 4.3. Positioning of ORBITON HiMA highly modified bitumens relative to paving-grade binders and conventional polymer modified binders in the Pen25-SP R&B chart

#### 4.1.4. National Application Document – Requirements for Highly Modified Bitumens

In April 2014, the Polish Standardisation Committee issued a National Annex to standard PN-EN 14023 in Poland, which defines the requirements for bitumens highly modified with polymers.

The division of bitumens highly modified with polymers into types, classes and requirements according to the National Annex, table NA.2, to standard PN-EN 14023:2011, has been shown in table 4.2.

Table 4.2. Division based on types and requirements for highly modified bitumen in Poland acc. to national annex NA, table NA.2, to standard PN-EN 14023:2011/Ap1:2014-04

Property		Test method	Unit	Highly modified bitumen type					
				ORBITON 25/55-80 HiMA		ORBITON 45/80-80 HiMA		ORBITON 65/105-80 HiMA	
				Requirement NA.2 2014	Class	Requirement NA.2 2014	Class	Requirement NA.2 2014	Class
Penetration at 25°C		EN 1426	0.1 mm	25 to 55	3	45 to 80	4	65 to 105	3
Softening point		EN 1427	°C	≥ 80	2	≥ 80	2	≥ 80	2
Cohesion	Force ductility tested using ductilometer (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	TBR (at 15°C)	—	TBR (at 10°C)	—	TBR (at 10°C)	—
Resistance to hardening	Change in mass	EN 12607-1	%	≤0.5	3	≤0.5	3	≤0.5	3
	Retained penetration		%	≥60	7	≥60	7	≥60	7
	Softening point increase		°C	≤8	2	≤8	2	≤8	2
Flash point		EN ISO 2592	°C	≥235	3	≥235	3	≥235	3
Fraass breaking point		EN 12593	°C	≤-15	7	≤-18	8	≤-18	8
Elastic recovery	at 25°C	EN 13398	%	≥80	2	≥80	2	≥80	2
	at 10°C	EN 13398	%	TBR	1	TBR	1	TBR	1
Softening point drop after testing as per EN 12607-1		EN 1427	°C	TBR	1	TBR	1	TBR	1
Elastic recovery at 25°C after testing acc. to EN 12607-1		EN 13398	%	≥60	4	≥60	3	≥60	2
Elastic recovery at 10°C after testing acc. to EN 12607-1		EN 13398	%	NR	0	TBR	1	TBR	1
Storage stability (3 days) Difference in softening point		EN 13399 EN 1427	°C	≤5	2	≤5	2	≤5	2
* Change in mass may be a positive or negative value NR – No Requirement TBR – To Be Reported									

#### 4.1.5. Application of ORBITON HiMA Highly Modified Bitumens

The application of substantially higher quantities of special SBS elastomer in the bitumen production process helps to achieve above-standard properties of bitumen in both high and low temperatures. Therefore, ORBITON HiMA highly modified binders are particularly suitable for applications requiring very high durability, such as:

- Asphalt surfaces subjected to very high stresses and strains,
- Layers with high resistance to low temperatures,
- Thin and ultra-thin wearing courses,
- Asphalt base courses with very high fatigue limit.

Highly modified bitumens are also dedicated to use in perpetual pavement type long-life surfaces, where the lowest asphalt course is characterised by very high elasticity and fatigue strength. **Application of ORBITON HiMA in the special anti-fatigue layer or in asphalt base course (e.g. Rich Bottom Layer) allows the user to achieve a very long-lasting pavement life cycle.**

Well-designed asphalt mixtures made using highly modified bitumens guarantee the achievement of much better properties than their counterparts of similar hardness (paving-grade and conventionally modified bitumens).

Despite their relatively short period of presence on the bitumen product market, the range of applications of ORBITON HiMA products is very wide, both in terms of the possible types of asphalt mixtures, as well as in terms of traffic category.

The typical applications of individual types of highly modified bitumen have been shown below.

**ORBITON 25/55-80 HiMA** is designed for use only in special cases – very stiff base courses and binder courses for container terminal surfaces, static loading etc. Generally, the use of such hard bitumen is recommended only when necessary.

**ORBITON 45/80-80 HiMA** is designed as an universal binder, for use in all courses of asphalt pavements exposed to very heavy loads and working at low temperatures, as well as for other courses in specific locations, e.g. on bridge decks.

**ORBITON 65/105-80 HiMA** is designed for wearing courses and special technologies, e.g. SAMI courses, thin wearing courses made of BBTM, AUTL, DSH, PA, SMA. It can also be used for the production of bituminous emulsions used in slurry seal. Special application of this binder are anti-fatigue courses at the bottom of asphalt structure. For areas with very low temperatures, it is recommended to use PMB 65/105-80 HiMA.

## 4.2. PROPERTIES

The following parts of this chapter contain a review of highly modified bitumen properties specified according to EN 14023, and additional information obtained via tests carried out according to the American *Superpave* and *Superpave plus* methods. The chapter also contains the classification of ORBITON HiMA bitumen types according to traffic loads, developed on the basis of results of MSCR tests (a description of the MSCR test can be found in Chapter 7).

This chapter also specifies approximate process temperatures for highly modified bitumen application in asphalt mixtures and data on the relation between viscosity and temperature.

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

The requirements for ORBITON HiMA highly modified bitumens and the results of laboratory testing carried out in 2016 have been shown in tables 4.3-4.5.

Table 4.3. Requirements and properties of ORBITON 25/55-80 HiMA highly modified bitumen manufactured in 2016 (results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484)

Property		Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C		EN 1426	0.1 mm	25 to 55	45
Softening point		EN 1427	°C	≥ 80	94.0
Cohesion	Force ductility tested using ductilometer (50 mm/min)	EN 13589 EN 13703	J/cm <sup>2</sup>	≥ 0.5 (at 15°C)	4.6
Resistance to hardening	Change in mass <sup>a</sup>	EN 12607-1	%	≤ 0.5	0.03
	Retained penetration		%	≥ 60	82
	Softening point increase		°C	≤ 8	1.6
Flash point		EN ISO 2592	°C	≥ 235	325
Fraass breaking point		EN 12593	°C	≤ -15	-19
Elastic recovery	at 25°C	EN 13398	%	≥ 80	91
	at 10°C	EN 13398	%	TBR <sup>b</sup>	78
Softening point drop after testing as per EN 12607-1		EN 1427	°C	TBR <sup>b</sup>	0.0
Elastic recovery at 25°C after testing acc. to EN 12607-1		EN 13398	%	≥ 60	88
Elastic recovery at 10°C after testing acc. to EN 12607-1		EN 13398	%	NR <sup>c</sup>	75
Storage stability (3 days) Difference in softening point		EN 13399 EN 1427	°C	≤ 5	0.5
a) Change in mass may be a positive or negative value b) TBR – To Be Reported c) NR – No Requirement					

Table 4.4. Requirements and properties of ORBITON 45/80-80 HiMA highly modified bitumen manufactured in 2016  
(results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484)

Property		Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C		EN 1426	0.1 mm	45 to 80	69
Softening point		EN 1427	°C	≥ 80	92.4
Cohesion	Force ductility tested using ductilometer (50 mm/min)	EN 13589 EN 13703	J/cm²	≥ 2.0 (at 10°C)	4.2
Resistance to hardening	Change in mass <sup>a</sup>	EN 12607-1	%	≤ 0.5	-0.02
	Retained penetration		%	≥ 60	80
	Softening point increase		°C	≤ 8	2.5
Flash point		EN ISO 2592	°C	≥ 235	>245
Fraass breaking point		EN 12593	°C	≤ -18	-21
Elastic recovery	at 25°C	EN 13398	%	≥ 80	95
	at 10°C	EN 13398	%	TBR <sup>b</sup>	79
Softening point drop after testing as per EN 12607-1		EN 1427	°C	TBR <sup>b</sup>	1.1
Elastic recovery at 25°C after testing acc. to EN 12607-1		EN 13398	%	≥ 60	91
Elastic recovery at 10°C after testing acc. to EN 12607-1		EN 13398	%	NR <sup>c</sup>	81
Storage stability (3 days) Difference in softening point		EN 13399 EN 1427	°C	≤ 5	2.9
a) Change in mass may be a positive or negative value b) TBR – To Be Reported c) NR – No Requirement					

Table 4.5. Requirements and properties of ORBITON 65/105-80 HiMA highly modified bitumen manufactured in 2016  
(results of tests by ORLEN Laboratorium Sp. z o.o., accreditation of PCA No. AB 484)

Property		Test method	Unit	Requirement acc. to PN-EN 14023	Mean value for 2016
Penetration at 25°C		EN 1426	0.1 mm	65 to 105	85
Softening point		EN 1427	°C	≥ 80	90.6
Cohesion	Force ductility tested using ductilometer (50 mm/min)	EN 13589 EN 13703	J/cm²	TBR <sup>b</sup> (at 10°C)	5.1
Resistance to hardening	Change in mass <sup>a</sup>	EN 12607-1	%	≤ 0.5	0.06
	Retained penetration		%	≥ 60	81
	Softening point increase		°C	≤ 8	2.1
Flash point		EN ISO 2592	°C	≥ 235	> 245
Fraass breaking point		EN 12593	°C	≤ -18	-20
Elastic recovery	at 25°C	EN 13398	%	≥ 80	94
	at 10°C	EN 13398	%	TBR <sup>b</sup>	89
Softening point drop after testing as per EN 12607-1		EN 1427	°C	TBR <sup>b</sup>	0.0
Elastic recovery at 25°C after testing acc. to EN 12607-1		EN 13398	%	≥ 70	93
Elastic recovery at 10°C after testing acc. to EN 12607-1		EN 13398	%	NR <sup>c</sup>	88
Storage stability (3 days) Difference in softening point		EN 13399 EN 1427	°C	≤ 5	1.7
a) Change in mass may be a positive or negative value b) TBR – To Be Reported c) NR – No Requirement					

#### 4.2.2. Properties acc. to Superpave

In the following section, we present the properties of ORBITON HiMA highly modified bitumens specified acc. to the American *Superpave* method and carried out between 2012 and 2015.

##### 4.2.2.1. Testing of low-temperature properties

In the Performance Grade system, the Bending Beam Rheometer (BBR) is used to test binder behaviour in low temperatures.

Table 4.6. presents low-temperature property testing results for ORBITON HiMA, with the test carried out by the Bending Beam Rheometer, 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 4.6. Low-temperature property testing results for ORBITON HiMA after ageing (RTFOT+PAV), using the Bending Beam Rheometer at S(60) = 300 MPa, m(60) = 0.3 and stiffness S at -16°C

Bitumen type	Critical temperature at S(60) = 300 MPa T(S) <sub>60</sub> [°C]	Critical temperature at m(60) = 0.3 T(m) <sub>60</sub> [°C]	Bitumen stiffness at -16°C S(T) <sub>-16</sub> [MPa]
	EN 14771, AASHTO PP 42		
	less = better		
ORBITON 25/55-80 HiMA	-18.5	-16.2	229.5
ORBITON 45/80-80 HiMA	-19.7	-19.8	181.3
ORBITON 65/105-80 HiMA	-20.6	-20.8	171.3

Figure 4.4. shows a comparison of low-temperature properties for ORBITON HiMA with ORBITON conventional modified binders and paving-grade binders with a similar penetration range.

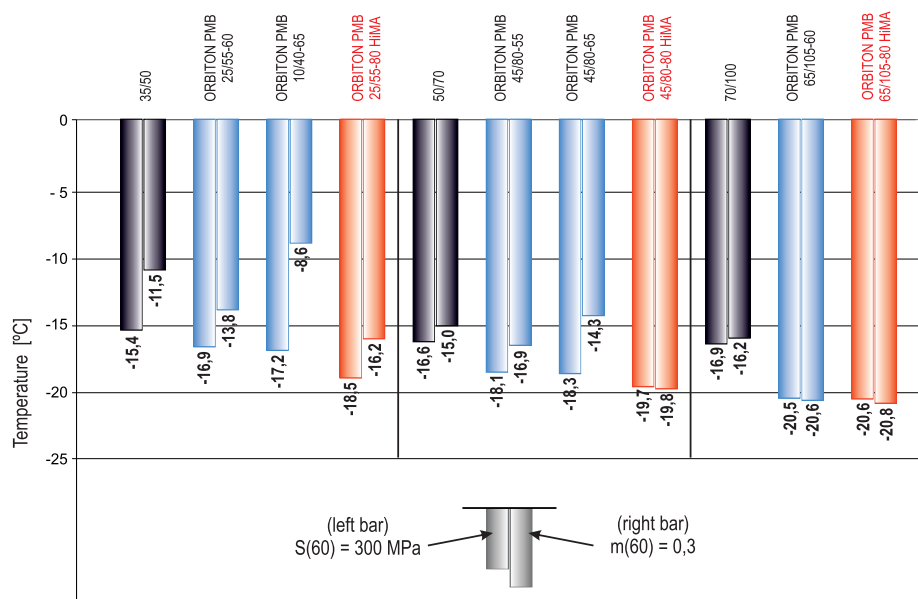


Fig. 4.4. Comparison of low-temperature properties for ORBITON HiMA with ORBITON conventional modified binders and paving-grade binders with a similar penetration range (critical temperature at S(60) = 300 MPa and at m(60) = 0.3)

#### 4.2.2.2. Testing of properties in intermediate temperatures – fatigue resistance

The DSR (Dynamic Shear Rheometer) is used for the binder fatigue test.

The test of binder resistance to fatigue cracks is performed in intermediate temperature (depending on the PG type). The requirements limit the stiffness  $G^* \cdot \sin \delta$  to a maximum value of 5 000 kPa (the newer version of the PG system raises this requirement to 6 000 kPa).

Table 4.3. shows the results of DSR tests to determine the conventional critical temperature depending on fatigue cracking of highly modified bitumens.

Table 4.7. DSR test results for the properties of ORBITON HiMA binders.

Highly modified bitumen type	Critical temperature at $G^* \cdot \sin \delta = 5000$ kPa bitumen after RTFOT+PAV [°C]	Critical temperature at $G^* \cdot \sin \delta = 6000$ kPa bitumen after RTFOT+PAV [°C]
	AASHTO T 315	AASHTO T 315
	less = better	
ORBITON 25/55-80 HiMA	17.9	16.2
ORBITON 45/80-80 HiMA	13.2	11.4
ORBITON 65/105-80 HiMA	12.3	11.3

Fig. 4.5. shows a comparison of intermediate-temperature properties for ORBITON HiMA highly modified bitumens with ORBITON conventional modified binders and paving-grade bitumen of a similar penetration range.

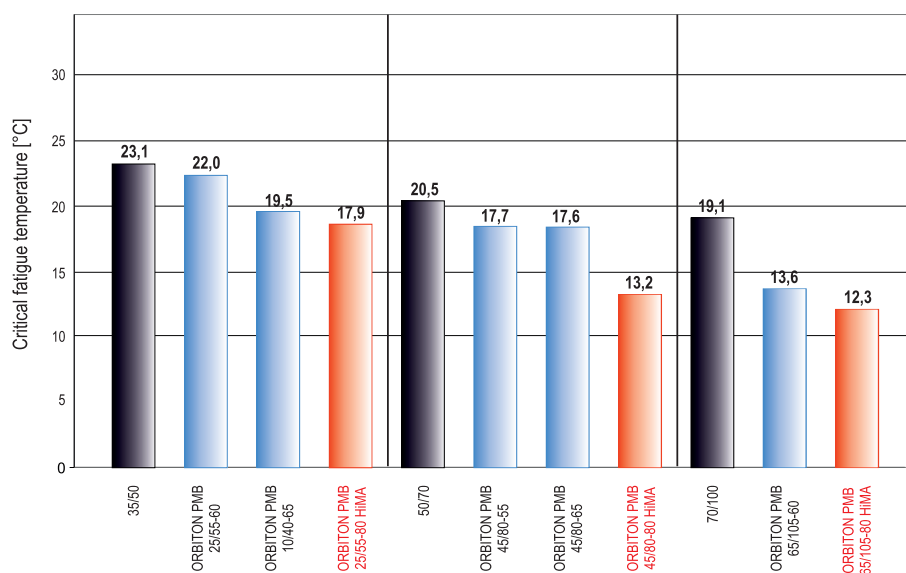


Fig. 4.5. Comparison of fatigue properties of ORBITON HiMA with conventional ORBITON modified bitumens and paving-grade bitumens with similar penetration range, using the DSR method ( $G^* \cdot \sin \delta = 5000$  kPa) acc. to Superpave

### 4.2.2.3. Testing of properties in high temperatures

#### 4.2.2.3.1. Conventional method using DSR ( $G^*$ and $\delta$ )

According to the original *Superpave* method, the resistance of the binder to high temperatures is determined in the DSR by measuring two parameters:

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

It is required that binder demonstrates specific parameters tested in the DSR at its expected maximum pavement service temperature (so-called high PG):

- $G^*/\sin\delta \geq 1.00$  kPa for bitumen before RTFOT,
- $G^*/\sin\delta \geq 2.20$  kPa for bitumen after RTFOT.

Table 4.8 presents the DSR test results for the properties of ORBITON HiMA binders.

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

Table 4.8. DSR test results for the properties of bitumens.

Bitumen type Highly modified	Critical temperature at $G^*/\sin\delta = 1$ kPa bitumen before ageing [°C]	Critical temperature at $G^*/\sin\delta = 2.2$ kPa bitumen after RTFOT [°C]
	AASHTO T 315	AASHTO T 315
	more = better	
ORBITON 25/55-80 HiMA	105.2	95.4
ORBITON 45/80-80 HiMA	98.2	84.3
ORBITON 65/105-80 HiMA	94.3	77.4

Figure 4.6. presents a comparison of upper critical temperature in the DSR test taking into account two parameters ( $G^*/\sin\delta$ ) for ORBITON HiMA bitumens in comparison to conventional ORBITON modified bitumens and paving-grade bitumens of a similar penetration range.

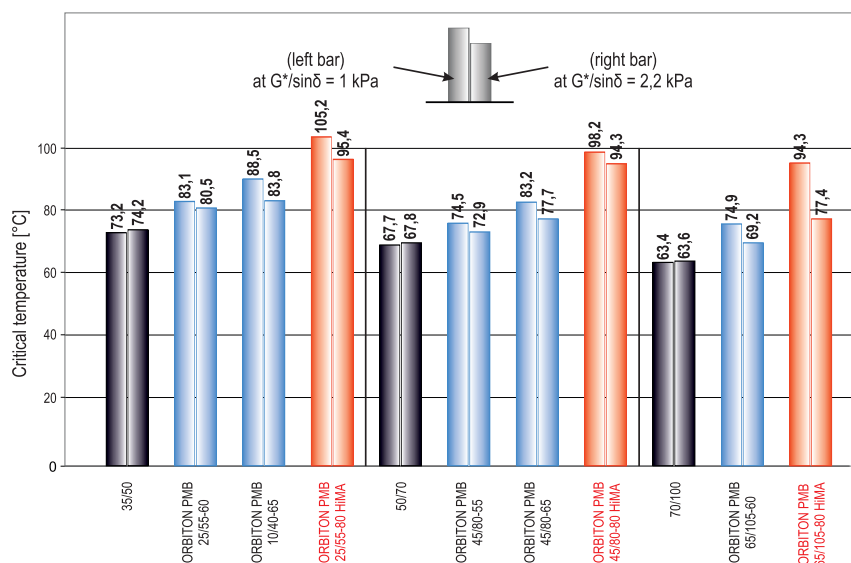


Fig. 4.6. Comparison of upper critical temperature in the DSR test for ORBITON HiMA with ORBITON conventional modified binders and paving-grade binders with a similar penetration range.



Figures 4.7. to 4.9. present Black's curves for paving-grade bitumens and modified bitumens with penetration ranges similar to that of the specific ORBITON HiMA types.

A Black curve is used to evaluate the dependence of the binder's complex stiffness modulus  $G^*$  on phase angle  $\delta$ . As we can see in the below figures, the more elastic is the binder, the more apparent is the elastic part of the binder due to the reduction of the phase angle together with the reduction of the complex stiffness modulus  $G^*$ . Therefore, the lower the phase angle  $\delta$  is – the better.

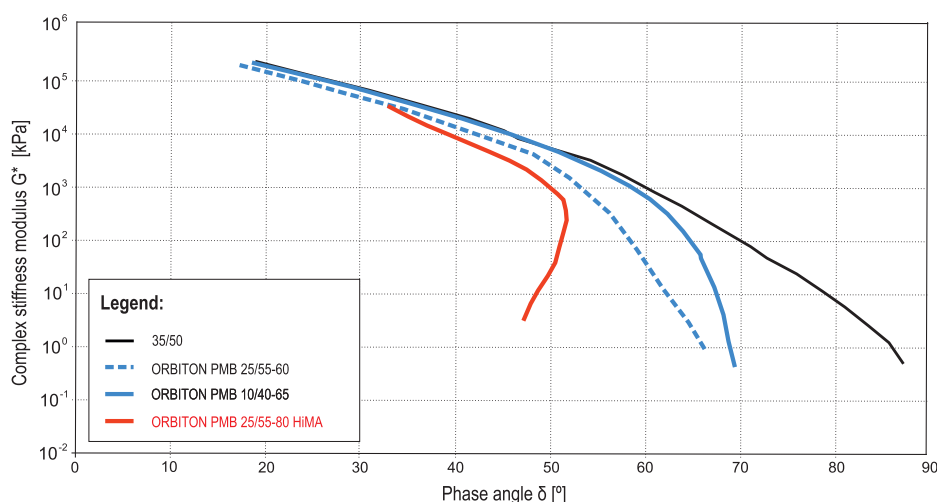


Fig. 4.7. Comparison of Black curves for **ORBITON 25/55-80 HiMA** with **ORBITON 25/55-60**, **ORBITON 10/40-65** and paving-grade bitumen **35/50** (all non-aged bitumens).

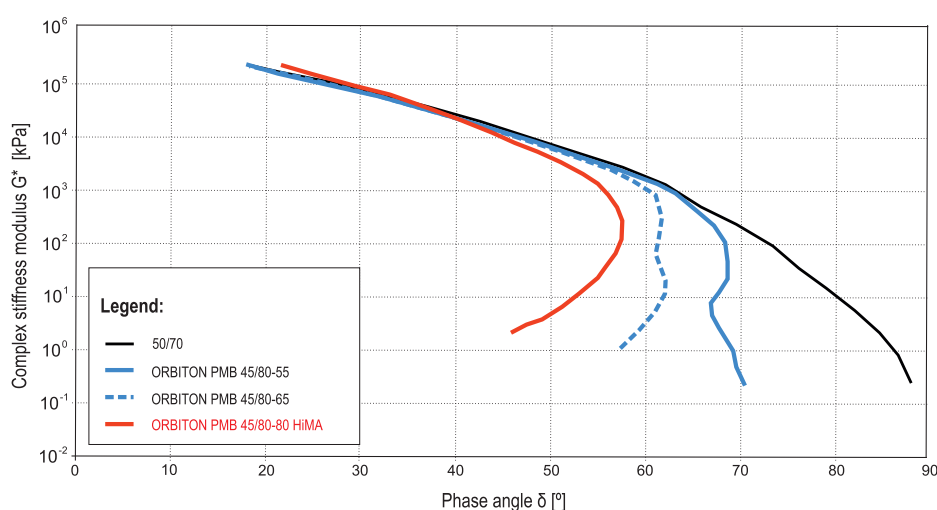


Fig. 4.8. Comparison of Black curves for **ORBITON 45/80-80 HiMA** with **ORBITON 45/80-55**, **ORBITON 45/80-65** bitumens and paving-grade bitumen **50/70** (all non-aged bitumens).

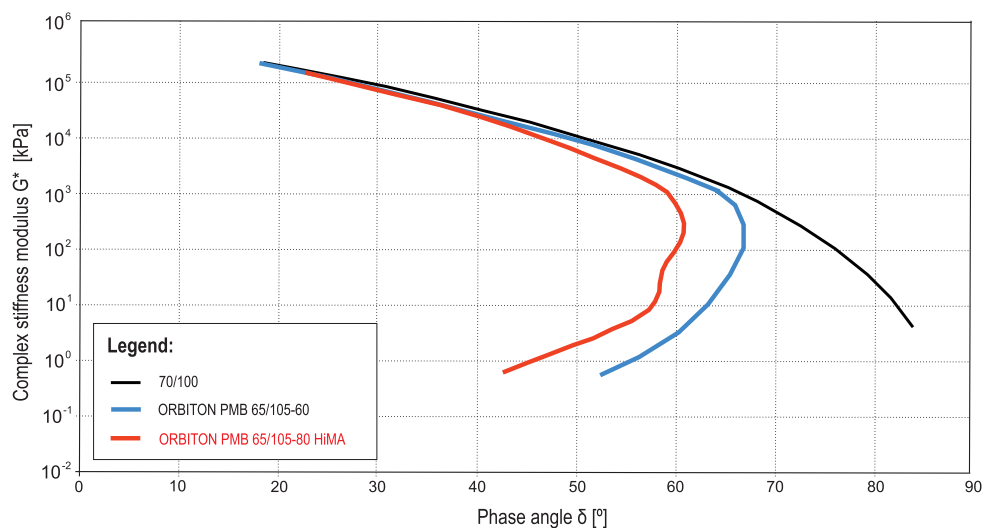


Fig. 4.9. Comparison of Black curves for **ORBITON 65/105-80 HiMA** with **ORBITON 65/105-60 bitumen** and **paving-grade bitumen 70/100** (all non-aged bitumens).

Figures 4.9-4.10 present master curves of the complex stiffness modulus  $G^*$  and phase angle  $\delta$  depending on frequency, performed for all highly modified ORBITON HiMA bitumens.

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.

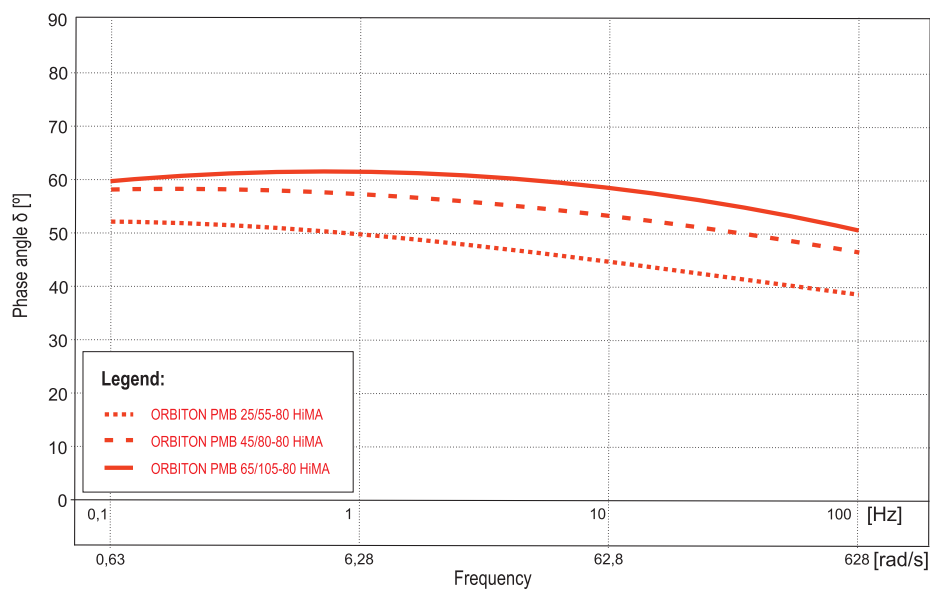


Fig. 4.10. Master curve of the angle phase  $\delta$  depending on the frequency for **ORBITON HiMA binders** before ageing. Sweep in the frequency range from 0.1 to 10 Hz, superposition to 25°C.

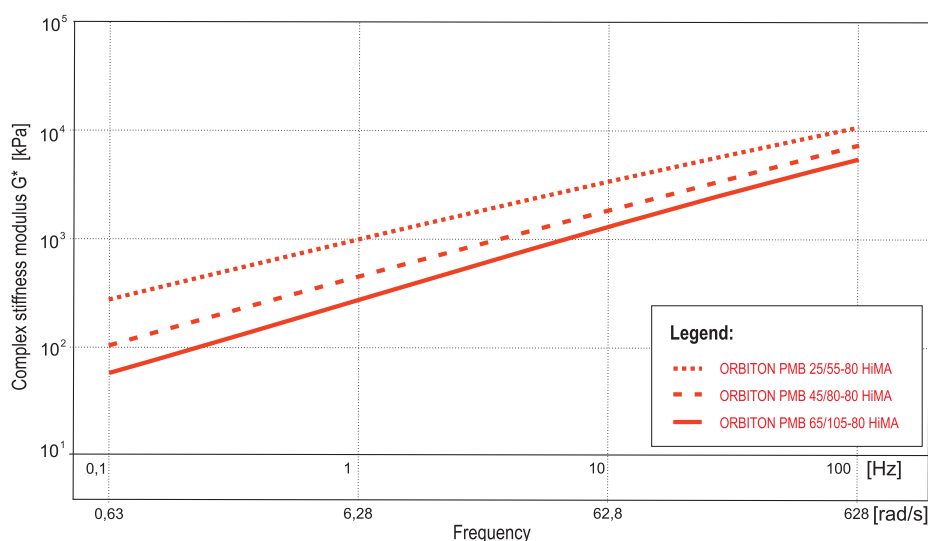


Fig. 4.11. Master curve of the complex stiffness modulus  $G^*$  depending on the frequency for ORBITON HiMA binders before ageing. Sweep in the frequency range from 0.1 to 10 Hz, superposition to 25°C.

#### 4.2.2.3.2. Results and classification of bitumens based on the MSCR method

The MSCR test is an extension of the *Superpave* method. A detailed description of the MSCR test has been presented in Chapter 7 of this Handbook.

Tables 4.9- 4.11. present MSCR test results for ORBITON HiMA highly modified bitumens manufactured by ORLEN Asphalt in 2015, carried out using the *Superpave* Plus method and basing on European Standard EN 16659.

Table 4.9. MSCR test results for **ORBITON 25/55-80 HiMA** bitumen according to the AASHTO TP 70/ ASTM D7405 method and EN 16659

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.013	0.023	0.035	0.020	0.022	0.068
$J_{nr}$ 3.2 kPa	0.015	0.027	0.041	0.013	0.026	0.094
$J_{nr}$ diff	9.1	16.7	15.9	31.0	23.0	38.2
R 0.1 kPa	92.7	92.3	92.9	87.5	94.5	91.5
R 3.2 kPa	92.2	91.6	92.4	91.6	93.5	89.1
R diff	0.5	0.8	0.6	-4.8	1.0	2.6
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Extreme	not subject to classification		

Table 4.10. MSCR test results for **ORBITON 45/80-80 HiMA** bitumen according to the AASHTO TP 70/ ASTM D7405 method and EN 16659

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.023	0.027	0.041	0.020	0.020	0.038
$J_{nr}$ 3.2 kPa	0.023	0.027	0.041	0.020	0.021	0.036
$J_{nr}$ diff	1.9	3.2	3.5	4.9	0.6	6.2
R 0.1 kPa	94.9	96.3	96.1	94.9	97.6	97.3
R 3.2 kPa	95.3	96.4	96.1	94.9	97.6	97.4
R diff	-0.4	-0.2	0.0	0.0	0.0	-0.1
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Extreme	not subject to classification		

Table 4.11. MSCR test results for **ORBITON 65/105-80 HiMA** bitumen according to the AASHTO TP 70/ ASTM D7405 method and EN 16659

Specified parameters	Temperature range according to <i>Superpave</i>			Temperature range according to European standard		
	AASHTO TP 70 ASTM D7405			EN 16659		
	Samples after RTFOT ageing according to EN 12607-1			Samples before ageing		
	58°C	64°C	70°C	50°C	60°C	70°C
$J_{nr}$ 0.1 kPa	0.011	0.010	0.014	0.010	0.008	0.012
$J_{nr}$ 3.2 kPa	0.008	0.009	0.012	0.009	0.007	0.010
$J_{nr}$ diff	26.2	15.1	9.8	11.4	22.5	18.8
R 0.1 kPa	97.5	98.3	98.3	97.5	98.9	98.8
R 3.2 kPa	98.3	98.7	98.4	97.9	99	99
R diff	-0.8	-0.4	-0.2	-0.4	-0.2	-0.2
Final classification of suitability for road traffic, according to $J_{nr}$ 3.2 kPa (at test temperature)	Extreme	Extreme	Extreme	not subject to classification		

#### 4.2.3. Viscosity dependence on temperature

Figures 4.12. to 4.14. show the characteristic viscosity curves of ORBITON HiMA highly modified bitumens before ageing and after ageing, which can be used to determine the viscosity-temperature characteristics. However, given the unusual characteristics of the binder resulting from the reversal of the bitumen-polymer phase and the specific characteristics of the polymer used, the implementation of the viscosity-temperature relation to accurately determine process temperatures does not seem to be appropriate. Temperatures defined in this way are only approximate.

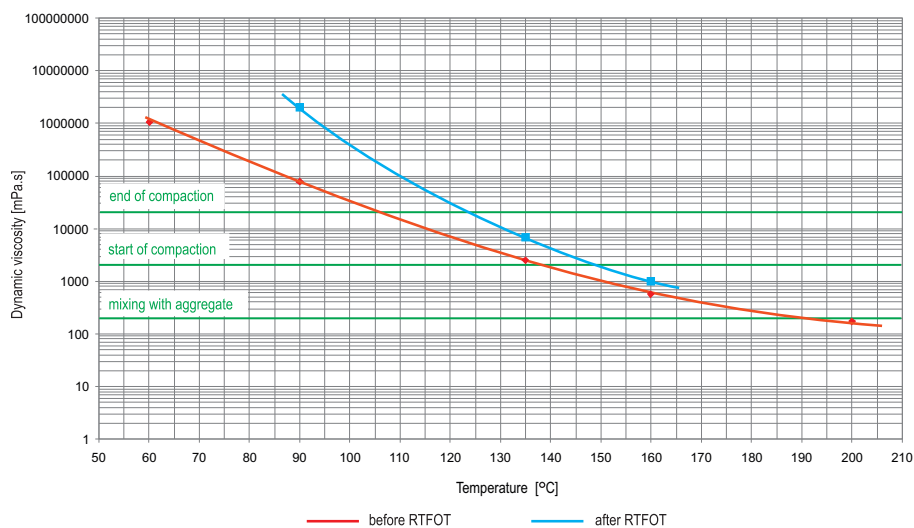


Fig. 4.12. Relation between viscosity and temperature for **ORBITON 25/55-80 HiMA** highly modified bitumen

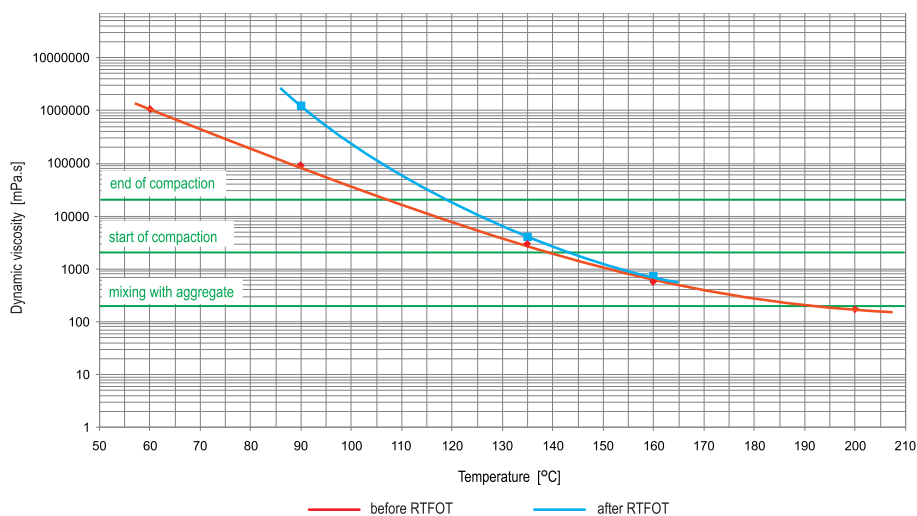


Fig. 4.13. Relation between viscosity and temperature for **ORBITON 45/80-80 HiMA** highly modified bitumen

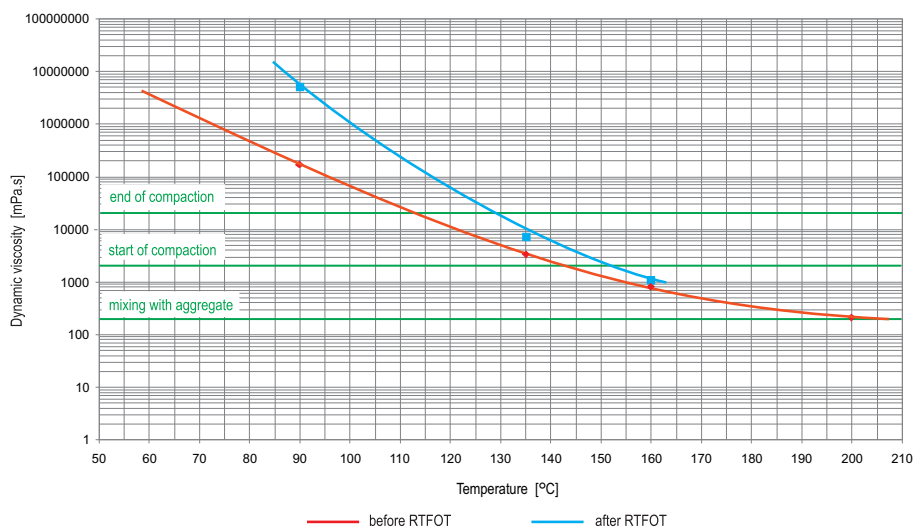


Fig. 4.14. Relation between viscosity and temperature for **ORBITON 65/105-80 HiMA** highly modified bitumen

## 4.3. APPLICATIONS

### 4.3.1. Process temperatures

The authors of this Handbook believe that, in the case of HiMA highly modified bitumens, reliance only on the viscosity of the binder when determining process temperatures leads to their overestimation, and subsequent excessive overheating of the binder. Therefore, process temperatures must be selected very carefully.

Table 4.12. shows the proposed process temperatures for ORBITON HiMA highly modified bitumens in the lab, mixing plant and at the construction site.

Table 4.12. Process temperatures for ORBITON HiMA highly modified bitumens.

	ORBITON 25/55-80 HiMA	ORBITON 45/80-80 HiMA	ORBITON 65/105-80 HiMA
<b>Laboratory:</b>			
Marshall/gyratory press sample compaction temperature	155-160°C	150-155°C	145-150°C
<b>Component temperature at the mixing plant:</b>			
Pumping of bitumen	over 160°C	over 150°C	over 150°C
Short-term binder storage at the mixing plant (up to 3 days)	up to 180°C	up to 180°C	up to 180°C
Long-term binder storage at the mixing plant (more than 3 days)	up to 160°C	up to 160°C	up to 160°C
<b>Ready bituminous mixture temperature in the mixing plant's mixer:</b>			
Asphalt concrete (AC)	max. 190°C	max. 185°C	max. 185°C
SMA	max. 190°C	max. 185°C	max. 185°C
Porous concrete	not applicable	max. 185°C	max. 185°C
Mastic asphalt	max. 220°C	max. 200°C	max. 200°C
<b>Temperature on site:</b>			
Minimum temperature of the supplied bituminous mixture (in the spreader's hopper)	175°C	170°C	165°C
End of effective layer compaction temperature	>140°C	>135°C	>120°C

Note: **Temperature data presented in Table 4.12 has been defined on the basis of preliminary conclusions from experimental sections. They may change with further experience. We recommend the monitoring of process temperatures at the experimental section.**

### 4.3.2. Storage

Because of the special properties of ORBITON HiMA bitumens, we recommend the immediate application of the binder upon its delivery, without any unnecessary storage in the silo.

If longer storage (up to 3 days) is required, the bitumen should be homogenised by closed-cycle mixing in one or multiple silos. At least one of the silos should be fitted with an agitator. Excessive storage time (more than 3 days) in high temperatures may lead to gradual increase of the viscosity of highly modified bitumen, which may reduce the possibility of its easy application.

If storage of bitumen in the silo is planned for longer than 3 days, we recommend the reduction of temperature to maximum 160°C, with periodic mixing (circulation).

It is recommended to conduct basic inspection tests of highly modified bitumen properties after 3 days, in order to make sure that the product has not lost its properties. The following tests should be carried out:

- penetration at 25°C as per EN 1426
- softening point as per EN 1427
- elastic recovery at 25°C as per EN 13398

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

#### Long-term storage in high and low temperature (over 3 days)

Storage of highly modified bitumen for the duration of more than 3 days in high temperatures is not recommended. Storage in lower temperatures is possible, but requires the monitoring of the binder's parameters.

#### Other comments:

- Before changing the type or grade of binder in the silo, the user must make sure that the silo is empty,
- HiMA binders should not be mixed with other bitumens. Such mixing would markedly downgrade the performance of the binder and affect the durability of the pavement,
- Multiple heating and cooling cycles for ORBITON HiMA highly modified bitumens are not recommended.

#### 4.3.3. Bitumen samples in the lab

The way such binder is handled in the laboratory has a major effect on the test results of both bitumen and bituminous mixtures. It should therefore be remembered that a bitumen sample which is heated and/or overheated in the oven multiple times may significantly harden, and thus obtained results will not reflect reality.

Therefore, multiple heating of binder samples should be avoided. The authors of this Handbook 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 method of handling ORBITON HiMA highly modified bitumen samples has been shown in table 4.13.

Table 4.13. Temperature of heating up of samples in the laboratory.

Size of sample in container	ORBITON 25/55-80 HiMA	ORBITON 45/80-80 HiMA	ORBITON 65/105-80 HiMA
container volume up to 1 litre, – time of heating up of sample max. 2 hours	max. 180	max. 180	max. 175
container volume 1 ÷ 2 litres, – time of heating up of sample max. 3 hours	max. 180	max. 180	max. 175
container volume 2 ÷ 3 litres, – time of heating up of sample max. 3.5 hours	max. 185	max. 185	max. 180
container volume 3 ÷ 5 litres, – time of heating up of sample max. 4 hours	max. 185	max. 185	max. 180
container volume more than 5 litres, – time of heating up of sample max. 8 hour	160–200	160–200	160–180

**Additional comments:**

- the sample-holding container must not be tightly closed,
- under no circumstances should the samples be heated up to the temperature exceeding 200°C,
- after samples are heated in the containers, they should be homogenised by mixing, and care must be taken to avoid the introduction of air bubbles into the sample. Maximum mixing (homogenisation) time is 10 minutes,
- and 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.

#### 4.3.4. Asphalt mixture design – binder content

ORBITON HiMA bitumens ensure that the pavement is very resistant to rutting. The bituminous mixture composition therefore should not be designed with a minimum binder content, it is recommended to add about 0.2-0.3 pp (percentage points) more of the binder than in the case of conventional PMBs. This will improve the elasticity of the layer, its resistance to cracking up in winter and its workability during application.

#### 4.3.5. Application of hydrated lime

If hydrated lime is used in the mixture (in order to improve durability and adhesion of bitumen to aggregate), tests of the workability of the mixture must be performed before hand, and the lime and binder content must be adequate to achieve a mixture that can be compacted. The lime content shall not, however, exceed 2.0% of the mineral mixture mass (the amount of lime should be exchanged with added filler).

Most probably, in order to achieve the workability of the mixture, it will be necessary to increase the quantity of the binder in the bituminous mixture. Lime can be added to asphalt mixtures with softer types of HiMA (45/80-80 and 65/105-80), preferably with 65/105-80.

#### 4.3.6. Asphalt mixture production

In the course of mixing bitumen with aggregate, ageing processes accelerate rapidly, therefore “wet” mixing time should be carefully selected. Bearing this in mind, HiMA binders should not be overheated and the indications in Table 4.12. should be followed. The maximum production temperature should never be exceeded, not even to improve the workability and compatibility of the mixture on the construction site.

Temperatures provided in Table 4.12 do not apply to bituminous mixtures containing the “warm-mix” additives for production and placement temperature reduction. ORLEN Asphalt did not perform tests of the compatibility of such substances with ORBITON HiMA, therefore their use is the sole responsibility of the asphalt mixture manufacturer. The use of additives that reduce the production and application temperatures must therefore be preceded by adequate testing in the laboratory.

#### 4.3.7. Transport of bituminous mixtures

During the transport of bituminous mixtures containing highly modified bitumen, the same rules for the transport of mixtures as for other polymer-modified binders apply. Make sure to cover the mixture with tarpaulin.



#### 4.3.8. Placement

During the placement of mixtures containing ORBITON HiMA highly modified bitumen, the same rules as for conventional modified bitumen apply. The number and type of rollers as well as number of their passes may be increased, and the final parameters should be defined at the trial section. A key factor that requires attention is the temperature of mixture production and placement. If the temperature of the mixture is too low, there may be problems with compaction.

During the placement of ORBITON 25/55-80 HiMA and ORBITON 45/80-80 HiMA, it may be necessary to increase the number of rollers, especially when there is a rapid drop in the temperature of the bituminous mixture (Autumn). During compaction, the mixture may behave in an elastic way and move slightly under the weight of the rollers, especially in the first phase of compaction in high temperatures.

After the completion of pavement works, we recommend to clean the equipment of any leftover bituminous mixture immediately, while the mixture is still hot (this comment refers especially to the cleaning of spreaders).

#### 4.3.9. Acceptance tests

The same testing methods as with standard binders are used for the acceptance of bituminous mixture courses with ORBITON HiMA.

Where checks include the determination of polymer content in the recovered binder, it should be noted that in the case of high polymer content the result may be less precise.

## Chapter 5

# EFFECT OF BITUMINOUS BINDER ON FATIGUE RESISTANCE OF ASPHALT MIXTURES

### 5.1. INTRODUCTION

The effect of fatigue of asphalt pavements is one of the key aspects of designing pavement structures. It is significant not only in terms of the durability of the entire pavement, measured in years, but also in terms of the costs of construction and maintenance of roads. It has therefore great effect on the management strategy of a road network, implemented by the roads administrator.

For many road engineers, problems related to the fatigue resistance of asphalt pavements are not important, unknown even. Not many people are interested in the influences on “designed life” of asphalt mixtures and in the extent of such influences, which consequently affect the durability of built roads.

In this chapter, we wish to introduce the primary theory related to the effect of fatigue, and the results of comparative tests of the effect of bituminous binders on the fatigue resistance of asphalt mixtures.

Fatigue tests performed using the 4PB-PR method as per EN 12697-24 were carried out in the laboratory of the Road and Bridge Research Institute in Poland, Warsaw, in 2015.

### 5.2. EFFECT OF FATIGUE IN ASPHALT PAVEMENTS

The conventional layout of the asphalt pavement structure consists of a set of asphalt layers (usually two to three layers, sometimes one) – wearing course, binder course and asphalt base course, which lie on top of an aggregate base and pre-prepared subsoil layers. Upon being loaded with the wheels of a vehicle, the entire structure is subject to bending, and the highest values of stress and tensile strain (see fig. 5.1.) are present in the bottom of the lowest asphalt layer. Tensile strain present in the bottom of the lowest asphalt layer (which is usually the asphalt base course) is regarded as the critical strain  $\epsilon_t$  (also designated as  $\epsilon_c$ ), and defines the resistance to fatigue of the structure.

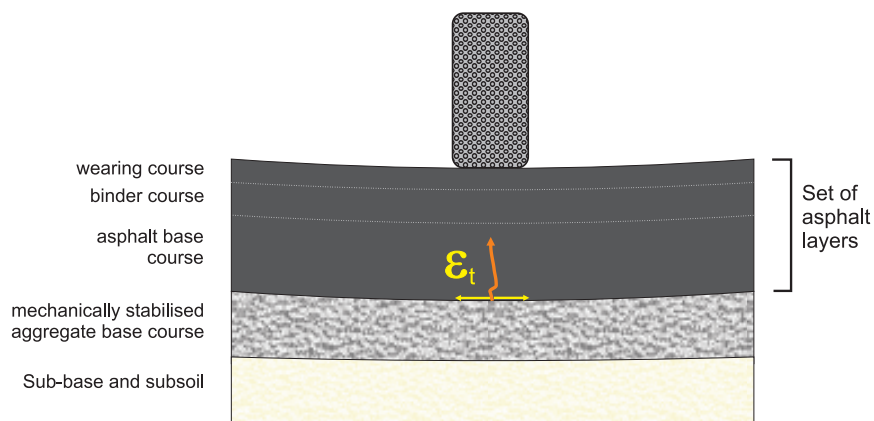


Fig. 5.1. Arrangement of layers of flexible pavement and location of occurrence of critical tensile strain

Despite initiating tensile strain, the single load applied to pavement by the passing of heavy vehicles does not initiate the cracking of the asphalt base course. It is only after the build-up of many loading cycles that the so-called fatigue damage starts to accumulate in the asphalt mixture, and finally causes cracks to appear and the process of degradation of the pavement begins.

It is obvious that larger tensile strain values in the bending pavement require less loading cycles to initiate the cracking of the asphalt base course. When designing the pavement structure, the thickness of layers and material characteristics are selected to achieve adequately low deflection values, which results in lower values of critical tensile strain and therefore a larger number of axes (number of deflections) that the pavement can carry in a defined period of time. In view of the above, it is easy to explain the idea of traffic category, which is usually based on the number of equivalent, standard axes of 100 kN applied to a pavement during a design period, e.g. during 20 or 30 years. Bearing this in mind, one must be very careful when reducing the thickness of asphalt courses.

Theoretical knowledge tells us that the resistance to fatigue of a asphalt mixture depends not only on the properties of the bituminous binder, but also on the asphalt mixture composition and the quality of lay-down on site. Of major significance is the composition of the mixture, as well as volumetric parameters, such as the content of voids in the layer or the degree of filling of voids with bitumen in a asphalt mixture (VFB). Even the best binder cannot ensure the required durability on its own, if any anomalies are experienced during the design or execution (i.e. poor layer compaction) phases of the pavement.

### 5.3. FATIGUE RESISTANCE TESTS

Tests of asphalt mixtures are performed according to standard EN 12697-24 „*Bituminous mixtures – Test method for hot mix asphalt – Part 24: Resistance to fatigue*”. This standard provides several procedures of sample loading, which are used to define the fatigue resistance of asphalt mixtures. In Poland, the implemented procedure is the 4PB-PR, which involves a four-point bending of a rectangular beam (see fig. 5.2.). The other test conditions are as follows: temperature 10°C, sinusoidal frequency of load 10 Hz, controlled strain mode, i.e. each loading cycle causes the same expected level of tensile strain  $\epsilon_t$  in the sample.

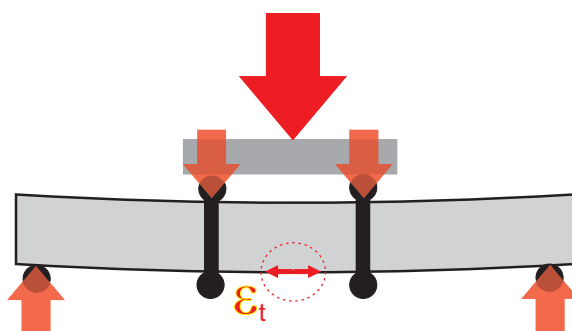


Fig. 5.2. Beam loading diagram for 4PB-PR test

The test is performed for a minimum of three values of tensile strain  $\epsilon_t$ . The test begins with an initial evaluation of the stiffness modulus of the material (the asphalt mix) and includes a series of beam bending cycles, until the measuring device record the drop of the sample's stiffness modulus by 50%. It means that the material has experienced a series of internal damages, which have caused the reduction of its stiffness by one half. The number of cycles needed to achieve this drop is accepted as the conventional fatigue resistance  $N_f$  for the assumed level of strain  $\epsilon_t$ , thus we receive a test result of  $N_f(\epsilon_t)$ .

Following the performance of tests for three  $\epsilon_t$  strain values and obtaining of three corresponding results  $N_f$ , a chart may be drawn up containing the fatigue characteristics of the tested asphalt mix sample, which presents the fatigue resistance of this material as a function of tensile strain. If we are testing two asphalt mixtures and we place them in the same chart, then we can directly compare their fatigue characteristics (fig. 5.3.). The mixture with the higher fatigue curve will sustain more cycles before failure (therefore is better in terms of fatigue resistance).

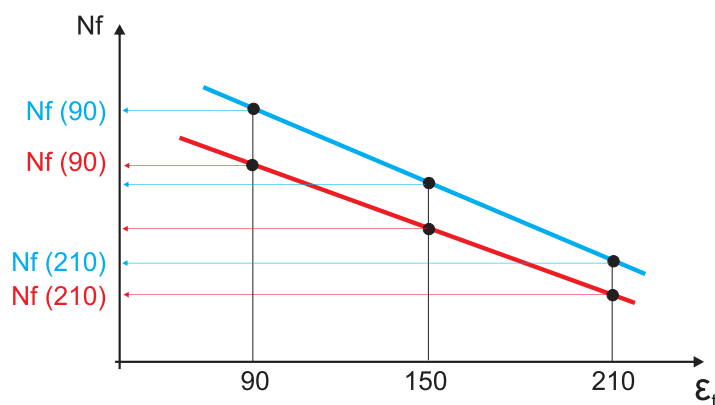


Fig. 5.3. Example of fatigue characteristics for two asphalt mixtures. The blue line mixture has better fatigue parameters – with the same strain, the number of cycles before theoretical failure is higher than for the red line mixture

## 5.4. TEST RESULTS

In 2015, a series of comparative tests of the effect of bituminous binder on the fatigue resistance of asphalt mixtures was carried out.

The tests were performed on a bituminous mixture for binder courses – AC 16. All prepared mixtures had the same mineral aggregate mix, in accordance with the grading curve shown in fig. 5.4. and contained a constant amount of binder – 4.6% m/m. The factor differentiating the mixtures was the used type of bituminous binder. Basalt aggregate was used for the production of the tested mixtures.

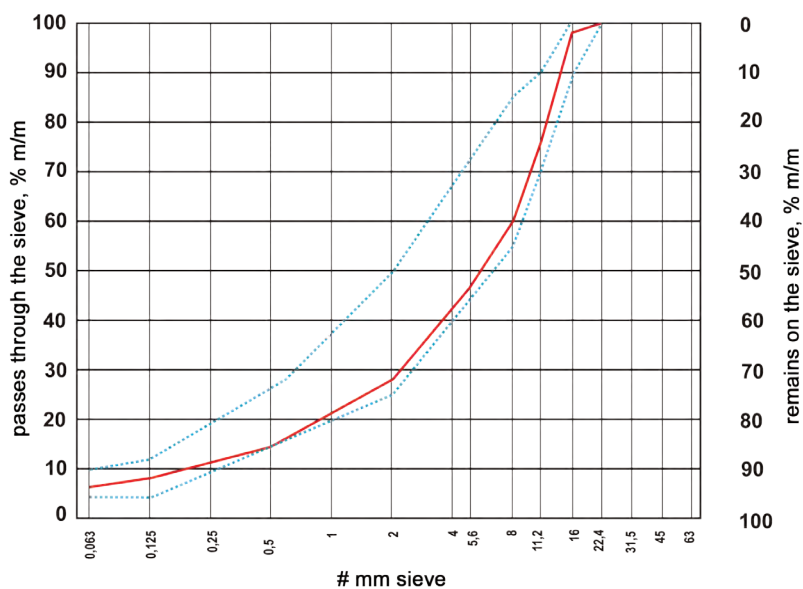


Fig. 5.4. Graining of asphalt mixture AC 16 used for fatigue and SCB tests (Road and Bridge Research Institute recipe)

The tests involved the comparison of the following bitumens:

- Paving grade bitumens: 20/30, 35/50, 50/70
- ORBITON (conventional) modified bitumens: 25/55-60, 45/80-55
- ORBITON HiMA highly modified bitumens: 25/55-80, 45/80-80, 65/105-80

The tests resulted in the definition of fatigue characteristics for eight AC16 asphalt mixtures with 8 binders (fig. 5.5.).

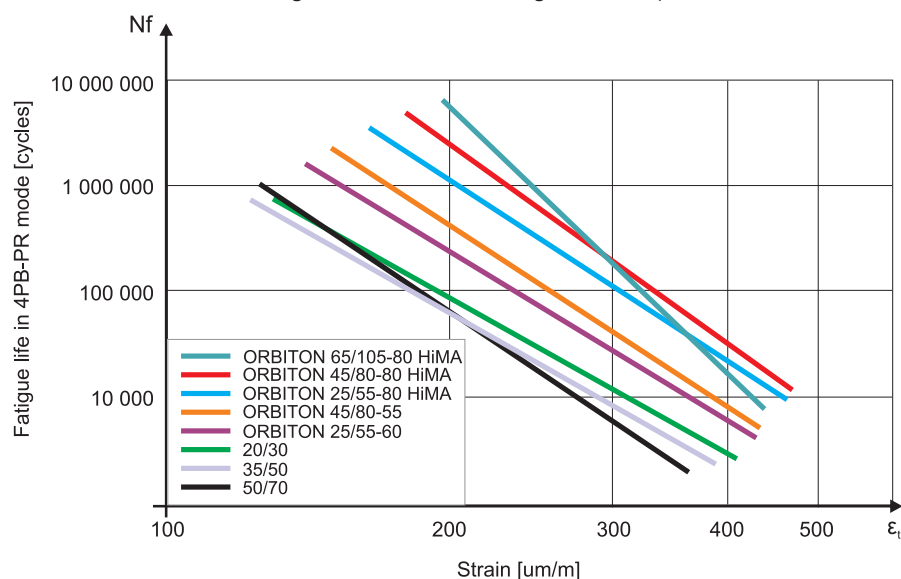


Fig. 5.5. Comparison of fatigue characteristics of 8 AC16 mixtures with different binders

Table 5.1. shows the comparative results for  $\epsilon_g$  (strain equivalent to the resistance for  $N_f=10^6$  fatigue cycles) for the tested AC16 mixture. It represents an estimation-based ranking of binders for this mixture. It must be noted that the binders were compared in an asphalt concrete (AC) mixture used for wearing courses (AC 16), and not in a mixture with a high stiffness modulus (AC EME with a larger binder content). Therefore, the achieved  $\epsilon_g$  values are not as high as those achieved in AC EME and should not be compared to values required for this mixture (usually  $\epsilon_g \geq 130$ ). The results are probably higher than for conventional asphalt concrete (AC) used for asphalt base courses (with higher void content and lower bituminous binder content).

Table 5.1. Comparative results of  $\epsilon_g$  value for the tested AC 16 mixture with different binders

Binder used in AC 16 asphalt mixture	Strain equivalent to resistance of $N_f=10^6$ fatigue cycles $\epsilon_g$
	[ $\mu\text{m/m}$ ]
ORBITON 65/105-80 HiMA (highly modified)	258
ORBITON 45/80-80 HiMA (highly modified)	230
ORBITON 25/55-80 HiMA (highly modified)	203
ORBITON 45/80-55 (conventionally modified)	171
ORBITON 25/55-60 (conventionally modified)	153
50/70 (paving grade unmodified)	125
20/30 (paving grade unmodified)	122
35/50 (paving grade unmodified)	116

Both in fig. 5.5. and in table 5.1. we can observe a clear division of binders into three groups with different fatigue characteristics.

- The best results are achieved with ORBITON HiMA highly modified bitumens, where the soft HiMA (65/105-80) mixture has the best resistance, followed by medium HiMA (45/80-80) mixture and hard HiMA (25/55-80) mixture. In the case of these mixtures, very good results are determined by the reversed binder phase – advantage of polymer phase over the bituminous phase and continuity of the elastomer network, which substantially increases the resistance of the binder and the mixture to tension.
- The second group consists of conventional ORBITON modified bitumens. Similarly, the softer polymer-bitumen (45/80-55) turned out to be the most fatigue-resistant in this group. Modified bitumens are better than paving-grade bitumens, but are inferior to bitumens highly modified with polymers.
- The last group consists of non-modified, paving-grade bitumens. All three binders within this group, used for testing, are characterised by similar fatigue resistance properties.

All three groups demonstrate the tendency of obtaining better results by softer binders, which is directly connected with the implementation of the controlled strain mode testing method. This testing method promotes more elastic and flexible binders and mixtures, with higher elasticity. Thus the advantage of binder of the penetration range of 65/105 and 45-80 over binders of the penetration 25-55 was visible for each binder group.

The results obtained above can be used as guidance during the process of selection of binders for various applications, including for special courses, e.g. *anti-fatigue AF layers in perpetual pavements*.

## Chapter 6

# EFFECT OF BITUMINOUS BINDER ON CRACKING RESISTANCE OF ASPHALT MIXTURES

## 6.1. INTRODUCTION

It is estimated that cracking represents the most common type of asphalt pavement damage, which occurs even more frequently than the well-known effect of rutting. There are many reasons for cracks in pavement and they are a result of various effects and mechanisms. They can appear as low-temperature shrinkage cracks, cracks reflected from lower layers with hydraulic binders, fatigue cracks, etc. In many of those cases, the key role is performed by the composition of the asphalt mix, which to a certain extent may cause or prevent (delay) the occurrence of cracks.

One of the factors that can be taken into account when counteracting the effect of cracking is the correct choice of asphalt mixture, adequate formula of its composition, and, finally, the use of the appropriate type of bituminous binder.

This chapter contains the description of results of tests for cracking resistance of asphalt mixtures containing various bituminous binders. The tests were performed using the SCB – *Semi Circular Bending* method as per standard EN 12697-44 in the laboratory of the Road and Bridge Research Institute in Poland, Warsaw, in 2015.

## 6.2. EFFECT OF CRACKING OF ASPHALT PAVEMENT

From the present analysis of numerous reasons for the appearance of cracks in pavement, we shall exclude problems caused by shrinkage, temperature, etc., dealing instead with the subject of propagation of cracks by the asphalt mixture. It is most frequently associated with reflected cracks, i.e. those cracks, which are transferred from a fractured lower layer through the asphalt mix layers. This effect often occurs when the so-called overlay is applied to an old, fractured pavement, as part of maintenance procedures.

Cracked, discontinuous old layers under the asphalt overlay become relocated, either due to vehicle traffic loads or due to changes in temperature (shrinkage-expansion and warping) – fig. 6.1. This causes tensile and shearing stresses to appear in the asphalt mixture used for the overlay, and these stresses result in the propagation of cracks to the overlay surface.

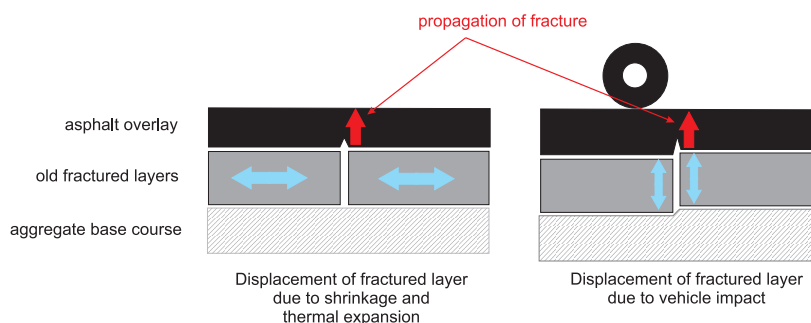


Fig. 6.1. Diagram of the propagation of cracks in overlay made of bituminous mixture due to horizontal (temperature changes) and vertical (traffic loads) movements

The SCB test, with the use of a semi-cylindrical sample with notch (see fig. 6.2.), is intended to define the resistance of the asphalt mixture to the propagation of artificially started crack at the bottom of the layer, during tensioning with bending (or plainly speaking – during the tearing of the sample).

### 6.3. TESTING USING THE SCB METHOD

Tests of bituminous mixtures are performed according to EN 12697-44 „*Bituminous mixtures – Test methods for hot mix asphalt – Part 44: Crack propagation by semi-circular bending test.*”

The testing is performed on a semi-cylindrical sample of the diameter of 150 mm and thickness of 50 mm, with a notch of the length of 10 mm and width of 0.35 mm. The loading force is applied to the sample in such way so that the obtained relocation amounts to 50 mm/min. The test is performed in the temperature of 0°C.

The diagram of the test is shown in Figure 6.2.

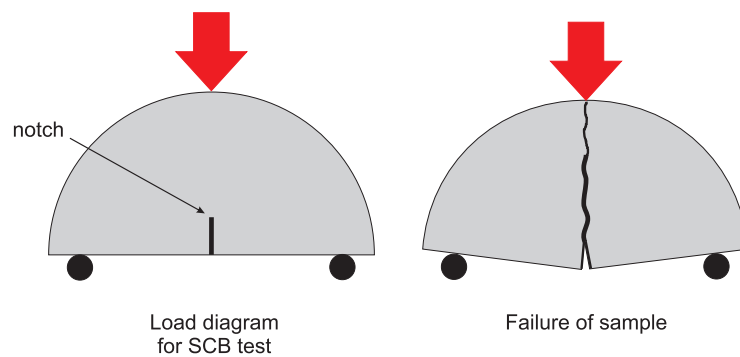


Fig. 6.2. Testing using the SCB method

The typical course of the test is shown on the force-relocation chart (fig. 6.3). The first phase of the test sees a rapid growth of the value of force  $F$  and minor deformation of the sample – this is the phase of the “resistance” of material, when the cohesion of the mixture resists tensile stresses present in the sample. The second phase is the attainment of the value of tensile strength of the sample (maximum value of force  $F$ ), with corresponding relocation and deformation  $\epsilon_{\max}$  of the sample. Phase three, having exceeded the tensile strength of the sample, demonstrates rapid progress (propagation) of the initiated crack through the sample and its failure.

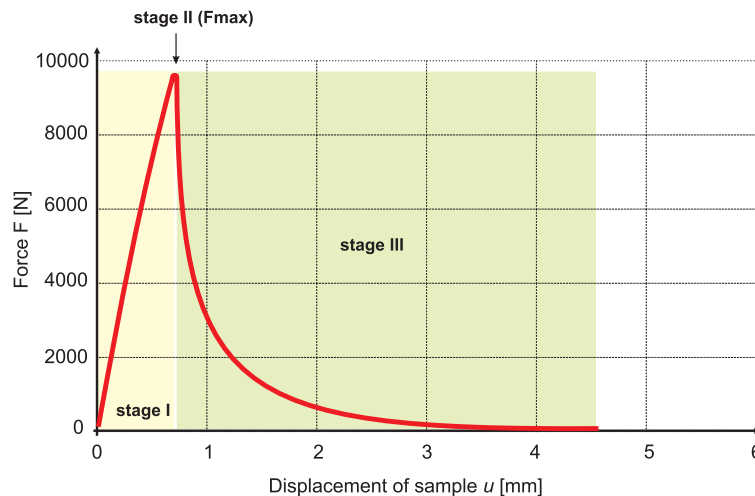


Fig. 6.3. Progress of SCB test – relocation-force chart



Fig. 6.4. shows an example of the fracturing of an AC 16 mixture.

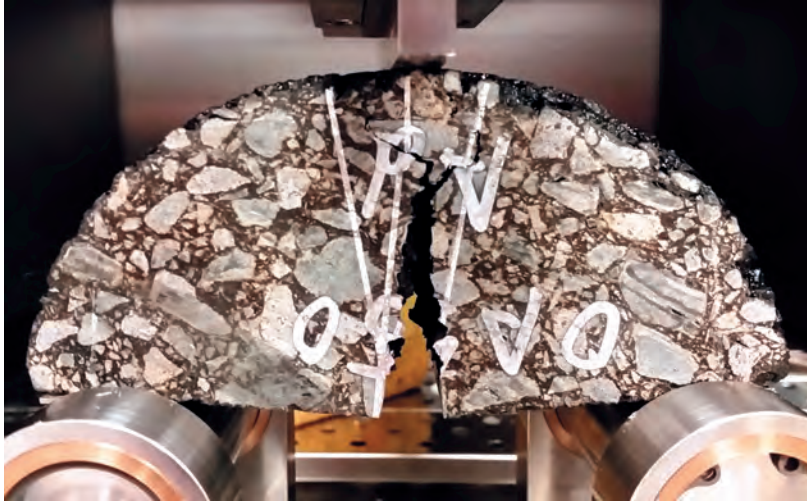


Fig. 6.4. Fracturing of AC 16 mixture during the SCB test (photo by Road and Bridge Research Institute, Warsaw)

## 6.4. TEST RESULTS

The tests were performed on the same bituminous AC 16 mixture (binder content of 4.6% m/m, which was described in Chapter 5.

The tests involved the comparison of the following bitumens:

- Paving grade bitumens: 20/30, 35/50, 50/70
- ORBITON modified bitumens: 25/55-60, 45/80-55
- ORBITON HiMA highly modified bitumens: 25/55-80, 45/80-80, 65/105-80

Standard EN 12697-44 provides the  $K_{Ic}$  [N/mm<sup>1.5</sup>] index (see equation 1.) as the result of the SCB test and a criterion of the resistance of the mixture to cracking. Table 6.1. lists the results for this index, and also additionally provides the deformation of sample  $\epsilon_{max}$  for the maximum value of force  $F$ . The information on deformation is interesting, because it indicates how far a sample with a given type of bitumen can deform while resisting the propagation of cracks. This indicates the elasticity of the binder and the elasticity of the mastic in the mixture.

According to EN 12697-44 the  $K_{Ic}$  index is calculated with equation:

$$K_{Ic} = \sigma_{max} \sqrt{\pi \cdot a} \cdot f\left(\frac{a}{W}\right) \quad (\text{eq. 1})$$

where:

$a$  – notch depth of specimen, mm

$W$  – height of specimen, mm

$\sigma_{max}$  – stress at failure of specimen, N/mm<sup>2</sup>

$$\sigma_{max} = \frac{4,263 \cdot F_{max}}{D \cdot t}$$

$D$  – diameter of specimen, mm

$t$  – thickness of specimen, mm

$F_{max}$  – maximum force of specimen, N

Table 6.1. Results of cracking resistance tests,  $K_{Ic}$  index

Binder used in the AC 16 W bituminous mixture	Cracking resistance $K_{Ic}$ [N/mm <sup>1.5</sup> ]	Deformation $\epsilon_{max}$ for maximum value of force F [%]
ORBITON 25/55-80 HiMA	36.1	1.1
ORBITON 45/80-80 HiMA	31.9	1.9
ORBITON 65/105-80 HiMA	23.6	1.9
ORBITON 25/55-60	24.8	0.8
ORBITON 45/80-55	28.3	0.9
20/30	24.1	0.8
35/50	23.1	0.8
50/70	22.8	0.8

In conclusion to the results of tests of the AC16 mixture with different binders (fig. 6.5.), as in the case of the fatigue tests, the results divided the binders into three groups in terms of the resistance to crack propagation. ORBITON HiMA highly modified bitumens turned out to be the best, followed by conventional ORBITON modified bitumens, and finally by paving-grade bitumens.

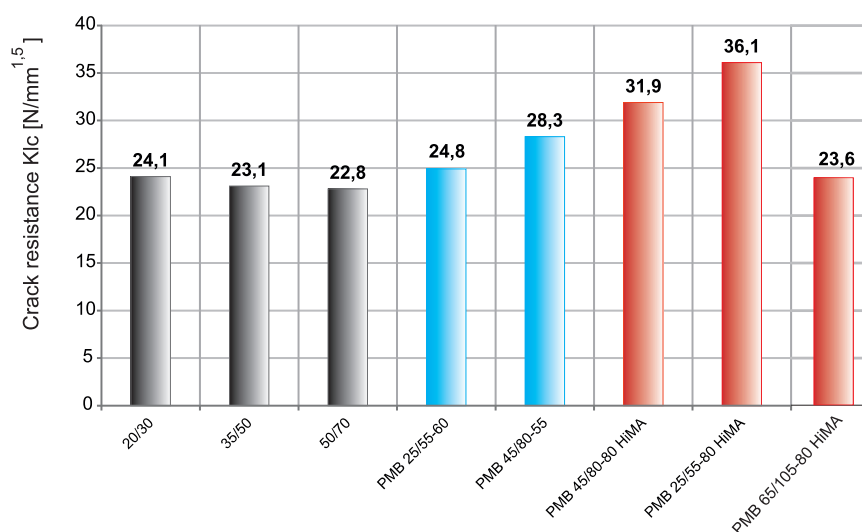


Fig. 6.5. Cumulative results for  $K_{Ic}$  index

Cracking resistance  $K_{Ic}$  characterises only the first phase of the test, i.e. the phase where the extreme values of force is achieved. During the analysis of the results, one must bear in mind that the  $K_{Ic}$  index does not take into account the deformation of sample during cracking, and only considers the value of breaking stress (see equation 6.1.).

Therefore we can conclude that cracking resistance  $K_{Ic}$  is connected with the stiffness of the mixture and does not directly account for the deformability (flexibility) of the mixture, which results directly from the equation for  $K_{Ic}$  given in standard EN 12697-44. Deformability for maximum force value expressed by  $\epsilon_{max}$  indicates additional, beneficial properties of the mixture and binder that allow the mixture to compensate the movement of the base without cracking, and in this context ORBITON 65/105-80 HiMA and 45/80-80 HiMA turn out to be the best.

## Chapter 7

### MSCR – MULTIPLE STRESS CREEP RECOVERY TEST

#### 7.1. INTRODUCTION

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 final system, together with the method for designing asphalt mixtures, has been named as *Superpave* (**SU**perior **PER**forming Asphalt **PAVE**ments).

As a result of the introduction of the *Superpave* system, the "conventional" parameters of classification of pavement binders, such as penetration or viscosity, have not been used in the USA for many years now. The basis for the division classifying binders according to *Superpave* is the theoretical temperature range in which each type of bitumen should "operate" correctly, called PG – **Performance Grade**.

Binder performance grade is designated as PG X-Y, where X is the maximum pavement temperature ("high PG"), while Y is the minimum pavement temperature ("low PG"), at which a given binder type is able to operate as required. It can therefore be argued that the requirements regarding the type and properties of bitumen (PG type) to be used are primarily determined by the weather records in the area where the road is constructed. Additionally, the conventional PG system implements a correction of high PG value, depending on traffic load values. Finally, high and low PG are selected from the list, with intervals of 6°C (Table 7.1.). Performance grades of ORLEN Asphalt binders established as above have been presented in Chapters 2÷4.

Table 7.1. 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

#### 7.2. TESTING OF BITUMEN PROPERTIES IN HIGH TEMPERATURES

Tests of bituminous binders in high temperatures are intended to define the ability of binders to counteract the viscoplastic deformations of the pavement. The tests are performed on the basis of AASHTO M 320 (*Specification for Performance-Graded Asphalt Binder*) and using ASTM D7175 (*Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*), with the use of a Dynamic Shear Rheometer – DSR.

The DSR test specifies high-temperature resistance, by testing the complex stiffness modulus  $G^*$  and angle phase  $\delta$  for bitumen, before and after RTFOT.

The binder has to demonstrate specific parameters tested in the DSR (so-called high PG) at its maximum pavement service temperature, which is determined on the basis of historic meteorological data:

- $G^*/\sin\delta \geq 1.00$  kPa for bitumen before RTFOT,,
- $G^*/\sin\delta \geq 2.20$  kPa for bitumen after RTFOT.

However, the classification based on  $G^*/\sin\delta$  parameters has proved to be ineffective in terms of the resistance of pavement to rutting. In a series of tests conducted in the USA when the original PG classification was in use, it was stated that the correlation between the results of rutting and  $G^*/\sin\delta$  (in both versions) is unsatisfactory [44], especially the problems with specification based on  $G^*/\sin\delta$  appeared in relation to polymer-modified bitumens [45]. Since approximately 2010, the classification system was supplemented with the MSCR test, and the specification was published in AASHTO MP 19 (*Standard Specification for Performance-Graded Asphalt Binder Using Multiple Stress Creep Recovery (MSCR) Test*) and revised in AASHTO M 332-2014 (*Standard Specification for Performance-Graded Asphalt Binder Using Multiple Stress Creep Recovery (MSCR) Test*).

### 7.3. MSCR TESTING IN USA

The MSCR test is conducted in the USA 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)* or ASTM D7405: *Standard Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer*. According to the above standards, tests can be performed on non-aged binder samples, on samples aged in RTFOT, PAV and RTFOT+PAV.

The primary assumption for the classification and evaluation of binders on the basis of MSCR test results is the **testing of bitumen sample after RTFOT**. An additional classification of the *Performance Grade* system was developed, on the basis of results obtained from this sample, where the type of traffic that the particular bituminous binder is most appropriate for is determined using letter symbols (table 7.2.). The adequacy of bitumen for a given traffic category is evaluated on the basis of parameter  $J_{nr,3,2}$  [kPa<sup>-1</sup>]. The introduction of this criterion has eliminated the so-called grade bumping – the elevation of the required “high PG” by one or two grades for heavy or slow traffic, which was implemented in the original PG system acc. to AASHTO M 320.

Table 7.2. Bituminous binder designations and requirements relative to traffic volume and characteristics according to the current *Superpave* system

Traffic designation (letter code)	Required for the binder at the high PG temperature	Required for the binder at the high PG temperature	
		Requirement for $J_{m3,2}$	Additional requirements for $J_{nr,diff}$ (Stress sensitivity parameter*)
<i>S – standard</i>	< 10 million axles and standard traffic	> 4.0	≤ 75%
<i>H – heavy</i>	10-30 million axles or slow traffic	> 2.0	
<i>V– very heavy</i>	>30 million axles or vehicle parking	> 1.0	
<i>E – extreme</i>	>30 million axles and vehicle parking	> 0.5	
*) binder sensitivity to stress change			

The MSCR test is to replace additional testing of modified bitumens defined in the PG “plus” method.

- elastic recovery,
- force ductility,
- toughness and tenacity.

## 7.4. MSCR TESTING IN EUROPE

Changes in the *Superpave* system also affected Europe. Because of the practical value of the MSCR test results, many research centres have started to use this measuring method on their own. The first tests using the MSCR method at ORLEN Asphalt were carried out already in 2011.

Simultaneously at CEN, at the technical committee TC 336 „*Bituminous binders*“, a programme of round-robin tests of the MSCR test was developed. The results of these tests were used for the development of standard EN 16659:2015 „*Bitumen and Bituminous Binders – Multiple Stress Creep and Recovery Test (MSCRT)*“.

European Standard EN 16659 does not define the method of preparation of the sample, therefore as a default the testing of non-aged bitumen is performed, although of course an aged-sample test may also be performed (the standard specifies e.g. RTFOT, PAV, RCAT methods). The recommended test temperature values have been specified: 50; 60; 70 and 80°C. Other temperatures may be applied for comparison. Contrary to the American system, in Europe there is no defined method for the evaluation of binders on the basis of the MSCR test results.

## 7.5. PERFORMANCE OF MSCR TESTS

The MSCR test, or the Multiple Stress Creep Recovery Test, is based on the measurement of binder properties in the highest expected operating pavement temperature (USA) or in any selected comparative temperature (Europe). **The results describe the effect of the properties of binder on the resistance of asphalt mixture to permanent deformations (rutting) and evaluate the degree and effectiveness of the modification of binder with polymers – in the case of PMB.** The test is conducted using an appropriately configured Dynamic-Shear Rheometer (DSR) (Fig. 7.1). This device uses a set of parallel plates of the diameter of 25 mm and with a gap of 1 mm.



Fig. 7.1. DSR rheometer for MSCR tests (photo by ORLEN Asphalt, with the permission of ORLEN Laboratorium Sp. z o.o.)

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 relieving cycle (after the stress is removed).

The test is conducted for two stress values: 0.1 kPa and 3.2 kPa. The sample is subjected to constant creep stress for 1 s, and then it is recovery for a further 9 s. Ten cycles of creep and recovery are performed with the application of 0.1 kPa of creep stress, and then a further ten cycles with the application of creep stress of 3.2 kPa (fig. 7.2.).

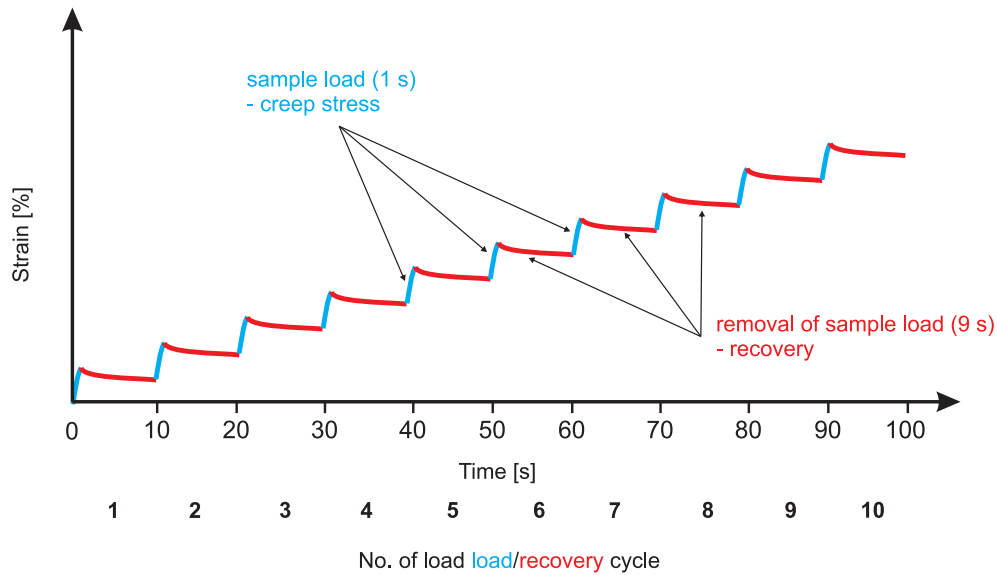


Fig. 7.2. Principle of execution of MSCR test (10 cycles of creep loads and recovery) for one value of creep stress

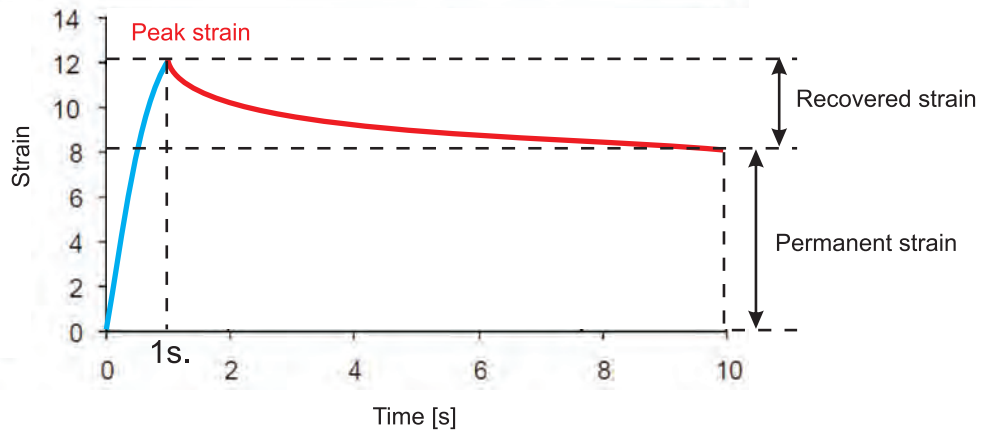


Fig. 7.3. One cycle of creep load (1 s) and recovery (9 s)

In effect, two pairs of results are obtained: non-recoverable creep compliance  $J_{nr}$  [kPa<sup>-1</sup>] and the average percentage deformation  $R$  [%] for two stress values, 0.1 kPa and 3.2 kPa. Thus we obtain the following results:  $J_{nr,0.1}$ ,  $J_{nr,3.2}$ ,  $R_{0.1}$  and  $R_{3.2}$ .

$J_{nr}$  is the **non-recoverable creep compliance** – a measure of the amount of residual strain left in the sample after repeated creep&recovery, relative to the amount of creep stress applied.

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

Two additional indicators are calculated from the results of  $J_{nr, 0.1 \text{ kPa}}$ ,  $J_{nr, 3.2 \text{ kPa}}$ ,  $R_{0.1}$  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 – which is a measure of binder sensitivity to load increase; the increase must not be greater than 75%.  $J_{nr, diff}$  is calculated according to the following equation:

$$J_{nr, diff} = \frac{(J_{nr, 3.2 \text{ kPa}} - J_{nr, 0.1 \text{ kPa}})}{J_{nr, 0.1 \text{ kPa}}} \cdot 100 \%$$

- $R_{diff}$  – percentage indicator of the difference in elastic recovery after the change (increase) in the stress from 0.1 to 3.2 kPa – which is a measure of binder elasticity under load increase conditions. This parameter is calculated according to the following equation:

$$R_{diff} = \frac{(R_{0.1 \text{ kPa}} - R_{3.2 \text{ kPa}})}{R_{0.1 \text{ kPa}}} \cdot 100 \%$$

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 [46]. The line has been shown in Figures 7.4.-7.6.

## 7.6. TEST RESULTS

When planning binder tests using the MSCR method in 2015, the ORLEN Asphalt's Department of Research and Development drew on experiences gained from tests carried out according to *Superpave* requirements between 2010 and 2014. In 2015 tests were also conducted in accordance with the provisions of European Standard EN 16659.

An interesting temperature range of tests was determined at 50-70°C, i.e.:

- tests according to *Superpave*: 58°C, 64°C, 70°C (binders after RTFOT),
- tests according to EN: 50°C, 60°C, 70°C (non-aged binders),

The results should be analysed bearing in mind that they have been obtained from randomly selected samples 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.

### 7.6.1. Tests in temperatures accordant with *Superpave*

Fig. 7.4.-7.6. show test results for various bitumens manufactured by ORLEN Asphalt and tested by MSCR in the temperatures of 58°C, 64°C and 70°C. All bitumen samples were earlier subject to RTFOT ageing. A line has been shown in the figures which separates modified bitumens (e.g. binders which meet the requirements for modified bitumens in terms of recovery  $R_{3.2}$  correlated with  $J_{nr, 3.2 \text{ kPa}}$  range).



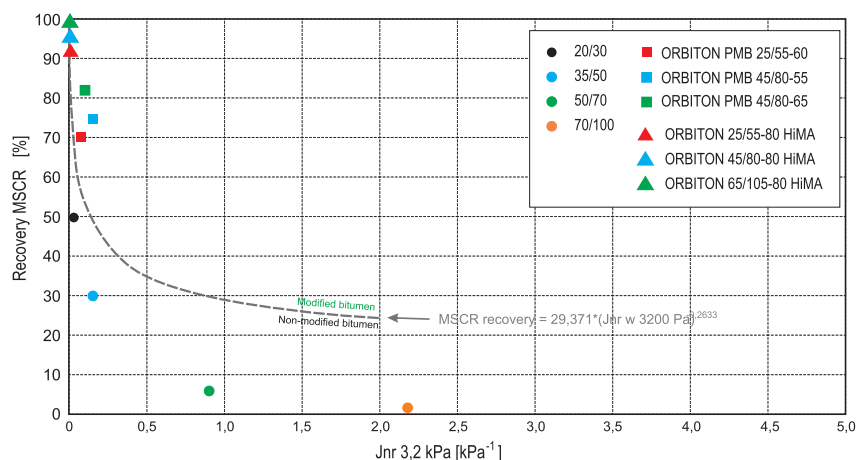


Fig. 7.4. Presentation of bitumen results on the MSCR chart: elastic deformation  $R$  as a function of  $J_{nr}$  with the load of 3.2 kPa, at 58°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder) – samples after RTFOT ageing

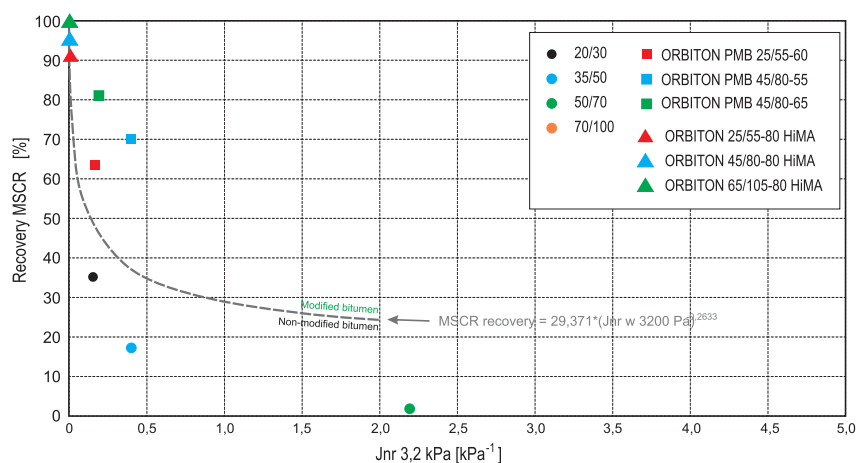


Fig. 7.5. Presentation of bitumen results on the MSCR chart: elastic deformation  $R$  as a function of  $J_{nr}$  with the load of 3.2 kPa, at 64°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder) – samples after RTFOT ageing

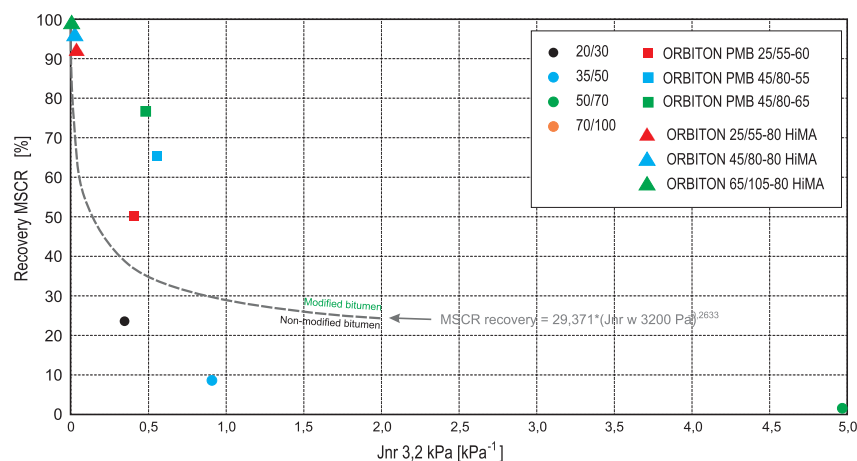


Fig. 7.6. Presentation of bitumen results on the MSCR chart: elastic deformation  $R$  as a function of  $J_{nr}$  with the load of 3.2 kPa, at 70°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder) – samples after RTFOT ageing



Table 7.3. Binder classification after the MSCR test by traffic load (testing of bitumens from 2015) on the basis of ranges from Table 7.2.

Bitumen type	Traffic classification for temperature		
	58°C	64°C	70°C
Paving-grade 20/30	E	E	E
Paving-grade 35/50	E	E	V
Paving-grade 50/70	V	S	*
Paving-grade 70/100	S	*	*
Modified ORBITON 25/55-60	E	E	E
Modified ORBITON 45/80-55	E	E	V
Modified ORBITON 45/80-65	E	E	E
Highly modified ORBITON 25/55-80 HiMA	E	E	E
Highly modified ORBITON 45/80-80 HiMA	E	E	E
Highly modified ORBITON 65/105-80 HiMA	E	E	E

\* unclassified (i.e.  $J_m 3.2 > 4.0$ )  
 S – standard traffic  
 H – heavy traffic  
 V – very heavy traffic  
 E – extreme traffic

## 7.6.2. Tests in temperatures accordant with EN 16659

Fig. 7.7- 7.9 show test results for various bitumens manufactured by ORLEN Asphalt and tested by MSCR in the temperatures of 50°C, 60°C and 70°C. Bitumen samples were not subject to ageing beforehand.

Since *Superpave* classification is not used in Europe, and tests were performed on non-aged binders, and it was not possible to compare the results with the requirements of road traffic classification used in the USA (as in table 7.3.).

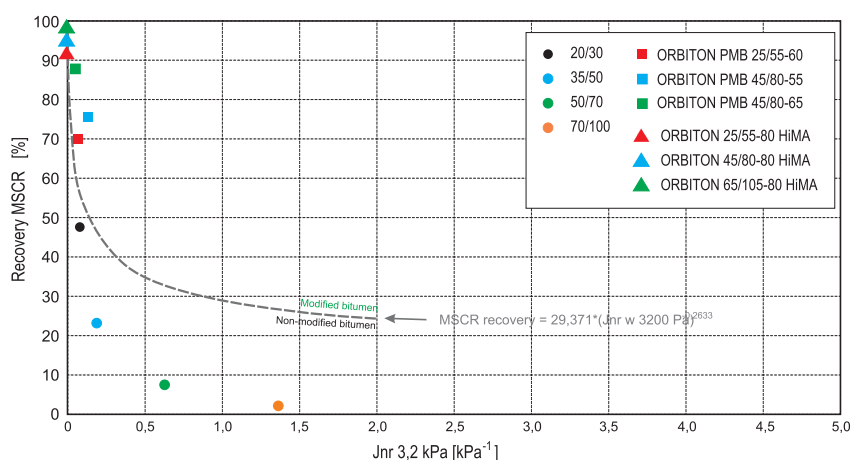


Fig. 7.7. Presentation of bitumen results on the MSCR chart: elastic deformation  $R$  as a function of  $J_{nr}$  with the load of 3.2 kPa, at 50°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder) – samples not aged

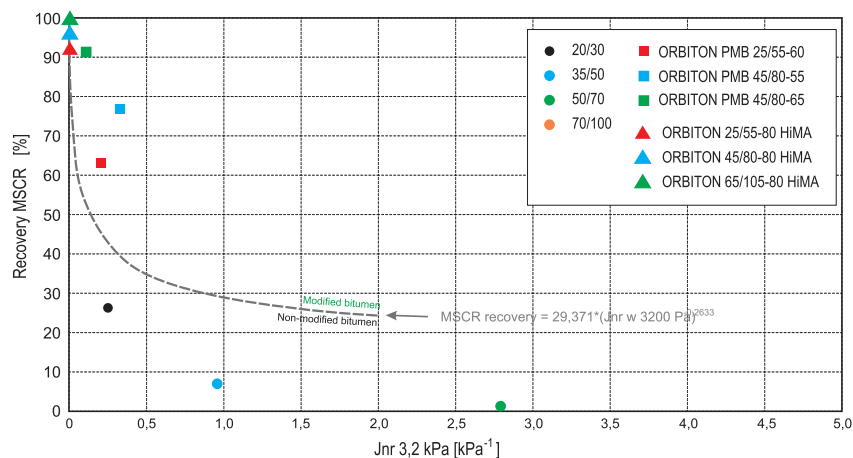


Fig. 7.8. Presentation of bitumen results on the MSCR chart: elastic deformation  $R$  as a function of  $J_{nr}$  with the load of 3.2 kPa, at 60°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder) – samples not aged

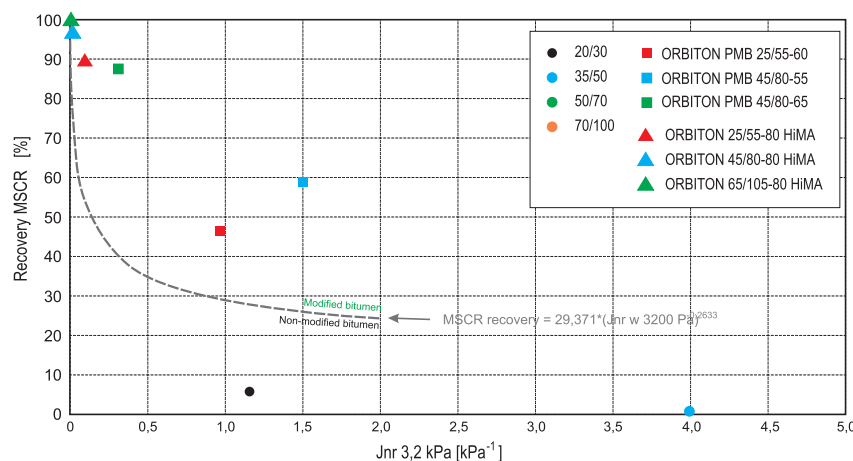


Fig. 7.9. Presentation of bitumen results on the MSCR chart: elastic deformation  $R$  as a function of  $J_{nr}$  with the load of 3.2 kPa, at 70°C (the smaller  $J_{nr}$  the greater rutting resistance, the greater recovery, the more elastic the binder) – samples not aged

## 7.7. SUMMARY

The presented MSCR test results help to classify bitumens tested in high measurement temperatures. These include bitumens aged using the RTFOT method (acc. to *Superpave*) or non-aged bitumens (acc. to EN). The range of bitumen testing temperatures and the correlation of results with the theoretical resistance to rutting according to  $J_{nr,3,2}$  provide the engineers with a very useful tool for the evaluation of the behaviour of selected bitumens in very hot pavement conditions. If we consider that European summers are getting hotter with each year, this may not only scientifically, but also of practically significant. Especially in the context of the provision of many years' warranty for transverse smoothness (resistance to rutting) of the constructed roads.

MSCR test results in constantly rising temperatures indicate rather high sensitivity of binders to very high temperatures. This means that situations of extremely hot air temperatures, which have started to occur in recent years, may significantly affect the creation of ruttings. Not all commonly used bituminous binders, especially those used for wearing courses, are able to withstand the rise of layer temperature from 50 to 70°C. This should be a starting point for discussion on the guidelines for the selection of type and sort of binder for big contracts, where highest traffic loads are prevalent.

## Chapter 8

### PHYSICAL HARDENING OF BITUMINOUS BINDERS

Bitumen is a thermoplastic material, therefore its properties depend on temperature. The change of all bitumen properties translate into its behaviour within a pavement, and affect the occurrence of specific types of damages.

In this chapter we present the results of testing of low-temperature properties of bitumens, which were performed in 2015 by the Research and Development Department of ORLEN Asphalt, on the basis of method LS-308 – „*Method of test for determination of performance grade of physically aged asphalt cement using extended beam rheometer (BBR) method*“ developed in Canada.

The tests covered selected paving-grade, modified bitumen and highly polymer-modified bitumen types.

#### 8.1. INTRODUCTION

Bitumen pavements, especially bitumen mixtures used in wearing courses, are subject to many detrimental factors during the winter period.

Among others, these include:

- the effects of exposure of bitumen to low temperatures: thermal shrinkage, physical hardening of bituminous binders, over-stiffness of layers,
- effects of freezing water,
- effects of de-icing agents.

The cumulation of the above-mentioned factors has a destructive effect on the pavement. However, these factors do not act in the same way throughout the entire low-temperature range. Some of them depend on the temperature drop rate, some affect the pavement right below 0°C, and others require the presence of very low temperatures for a longer period of time.

One of the methods for verification of low-temperature properties of bituminous binders is provided by the Canadian LS-308 method, which determines the degree of stiffening of bituminous binders due to long-term exposure to low temperatures.

#### 8.2. DESCRIPTION OF THE EFFECT OF PHYSICAL HARDENING OF BITUMEN

**Physical hardening of bituminous binders** occurs during their long-term exposure to low temperatures. It entails the increase of the stiffness of material, with the generation of stresses inside the binder that may lead to the occurrence of low-temperature cracks. The scale of this effect depends mainly on the time of exposure of the bitumen sample to low temperatures. This is a reversible process, so after the increase of the temperature, the stiffness of the tested material returns to its previous state. Physical hardening only changes the rheological properties of bituminous binders, without changing their composition and chemical structure [8].

The effect of physical hardening takes place in all types of bitumen, however it is not the same for all of them. The susceptibility of a given bitumen to the effects of low temperatures depends on many factors, such as: chemical composition of bitumen, source of crude oil, technology of production, method of bitumen modification, use of additives [24], [27]. Because of these factors, the effect of physical hardening may run a different course in one theoretically identical type of bitumen, i.e. bitumen 35/50 made by manufacturer A will display a larger tendency to harden physically than bitumen 35/50 made by manufacturer B [25], [26].

This is important, because physical hardening of bitumen, and consequently changes in the properties of bitumen mixtures, have a very significant effect on the behaviour road pavement in low temperatures and may cause more low-temperature cracks to appear.

In practice, the effect of physical hardening of bitumen and changes in asphalt mixtures occur during long-term periods of frost and are related to temperature and time during which the pavement remains frozen.

### 8.3. PHYSICAL HARDENING TESTING METHOD

Testing of the value of physical hardening of asphalt concrete was performed on the basis of the Canadian LS-308 method, using for this purpose the Bending Beam Rheometer (BBR).



Fig. 8.1. BBR Bending Beam Rheometer (photo by ORLEN Asphalt sp. z o.o. with the permission of UniCRE)

The LS-308 test is based on the conditioning (freezing) of the bitumen sample in specified temperature that is defined on the basis of the Performance Grade (PG) of a given binder. The stiffness of bitumen sample is tested after each conditioning period in the Bending Beam Rheometer and the value of physical hardening which occurred in the specific bituminous binder is calculated on the basis of the obtained results.

Below is a detailed description of the LS-308 method:

**Ageing of bituminous binders** – the LS-308 test begins with the ageing of the bituminous binder sample, at first using the RTFOT method which simulates short-term (technological) ageing – the behaviour of binder during its mixing with hot aggregate at the mixing plant, and afterwards using the PAV method which simulates long-term ageing – the behaviour of binder in the road pavement structure during its use.

**Definition of Low Critical Temperature (LCT) of bituminous binders** – following the ageing of bituminous binders, the next step is the definition of their Low Critical Temperature – LCT, in accordance with the American *Superpave* system (with  $S=300$  MPa and  $m=0.3$ ).

The lowest critical temperature of bitumen is defined using the Bending Beam Rheometer. “Low PG” (LCT) defined as above determines further test temperatures.

**Definition of sample freezing temperature** – when the Low Critical Temperature (LCT) of bitumen is established, two values of temperatures in which the samples will be frozen must be defined. The temperatures of conditioning (freezing) of bitumen samples are defined as follows:

- a) first conditioning temperature:  $20^{\circ}\text{C} + \text{YY}$
- b) second conditioning temperature:  $10^{\circ}\text{C} + \text{YY}$

where: YY – low PG (LCT)

The bitumen binder samples are then subject to conditioning in two specified and different temperature values for a defined period of time: 1 h, 24 h, 72 h.

**Testing in the Bending Beam Rheometer (BBR)** – after each conditioning period (1 h, 24 h, 72 h), the stiffness of the bitumen sample must be tested in the Bending Beam Rheometer, and new LCT value and physical hardening value for a specific bituminous binder must be established.

## 8.4. RESULTS

The following types of bitumen were used for tests performed according to the LS-308 method, during the ORLEN Asphalt research programme.

- Paving-grade bitumen 35/50,
- Paving-grade bitumen 50/70,
- ORBITON 25/55-60 modified bitumen,
- ORBITON 45/80-55 modified bitumen,
- ORBITON 45/80-80 HiMA highly modified bitumen.

Table 8.1. shows the results for physical hardening of tested bitumens, depending of time and temperature of the freezing of samples.

Table 8.1. The results for physical hardening of tested bitumen samples, defined according to standard LS-308, on the basis of results read out for stiffness  $S=300$  [MPa].

Sample conditioning temperature [°C]	Sample conditioning time [hours]	35/50	50/70	ORBITON 25/55-60	ORBITON 45/80-55	ORBITON 45/80-80 HiMA
		Physical hardening, calculated as per LS-308				
20°C+YY	1 h	0.7	-2.3	-1.6	0.1	-0.4
	24 h	6.0	3.2	4.0	4.2	2.9
	72 h	6.5	3.9	4.3	4.6	3.4
10°C+YY	1 h	0.0	0.0	0.0	0.0	0.0
	24 h	6.9	7.0	6.4	8.8	4.5
	72 h	7.9	8.2	7.3	9.0	5.3

The charts below are a graphic representation of the comparison of the values of physical hardening for the tested bitumen binders depending on the freezing time and temperature.

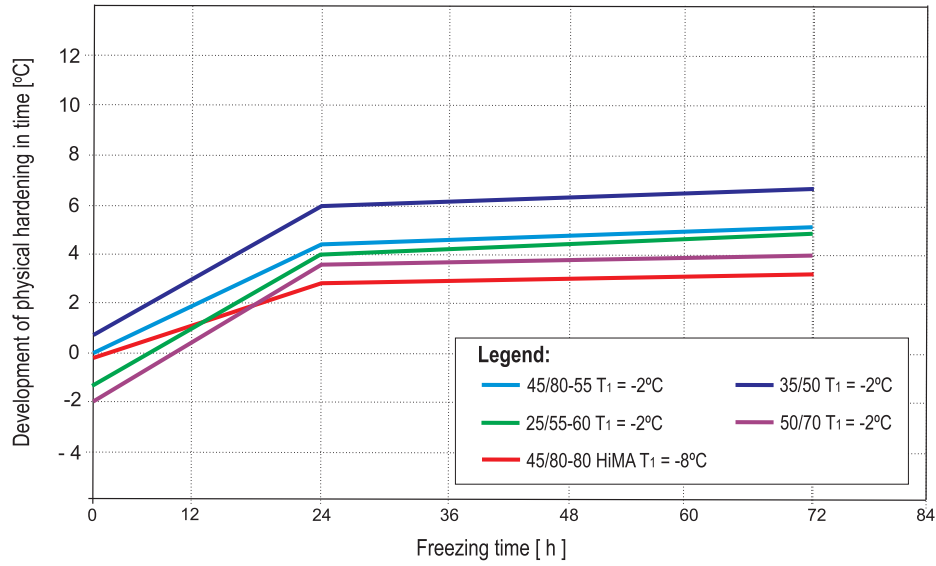


Fig. 8.2. Presentation of the results for physical hardening for all bitumen types in the freezing temperature of  $T_1 = 20^{\circ}\text{C} + YY$

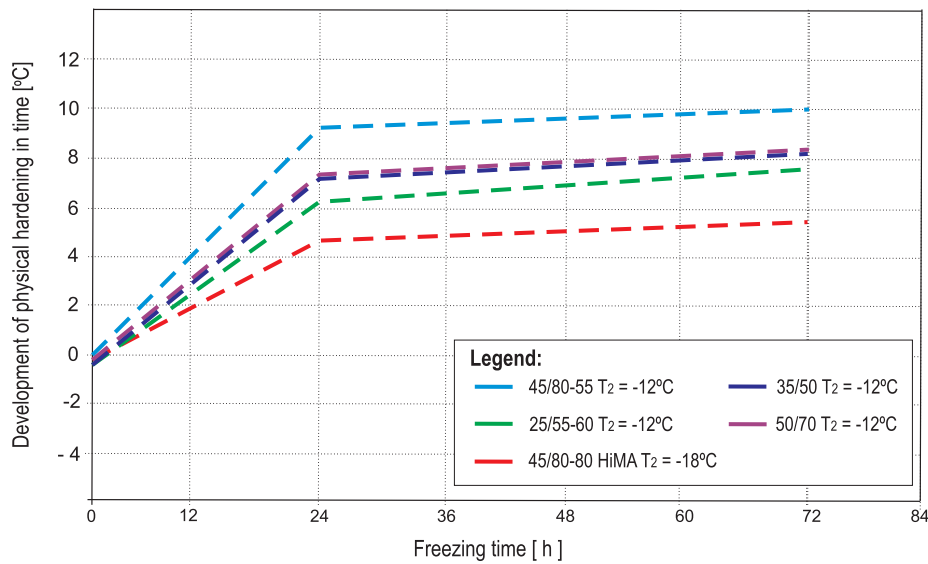


Fig. 8.3. Presentation of the results for physical hardening for all bitumen types in the freezing temperature of  $T_2 = 10^{\circ}\text{C} + YY$

For all tested bitumen types, the biggest increment of the value of physical hardening can be observed within the first 24 hours of exposure of samples to low temperatures. The next 48 hours does not affect the increase in physical hardening of the tested samples significantly, although an increment in stiffness does indeed exist. In the above charts it can also be clearly seen that the value of physical stiffness depends on the sample freezing temperature. **Lower conditioning temperatures are characterised by higher increments of physical hardening in time.**

Finally, the end result of the test can be presented in the form of the value of physical hardening increment in a specific type of bitumen, from the beginning of the test until the end of the freezing time of 72 hours.

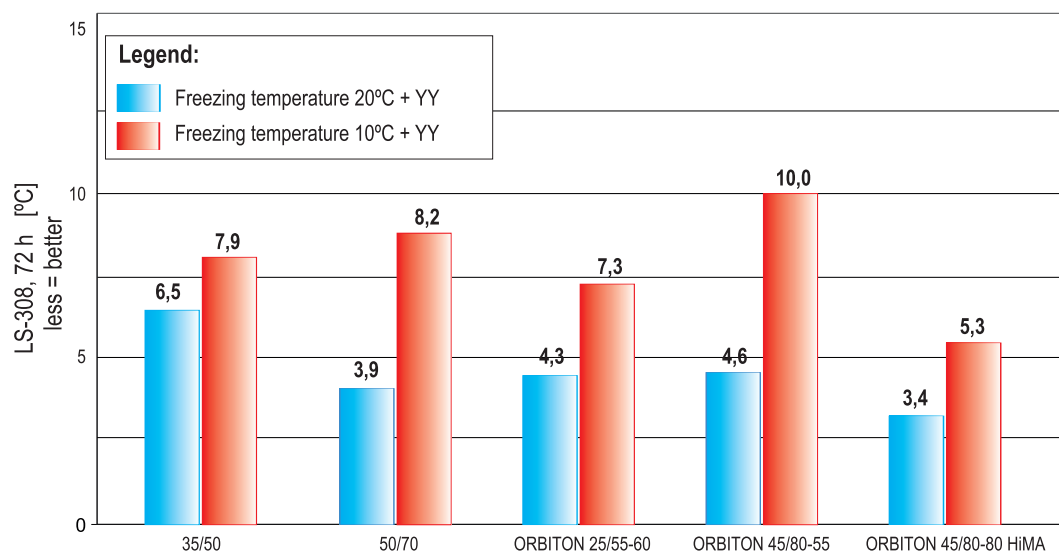


Fig. 8.4. Degree of physical hardening of tested bituminous binders after 72h of freezing, on the basis of  $S=300$  MPa

It must be noted that in spite of freezing of ORBITON 45/80-80 HiMA highly modified bitumen in lower temperatures than other binders, the level of stiffness represented by the physical hardening value was the lowest for this type of bitumen. This means that pavement made using this type of bitumen is the most resistant to long-term low temperatures.

## 8.5. SUMMARY

The analysis of LS-308 method test results essentially represents the evaluation of the degree of bitumen stiffening in low temperatures. The increase of bituminous binder stiffness leads to the worsening (increase) of "low PG", and has a consequence to the reduction of the functional scope of operation of the given type of bitumen.

Excessive stiffness of bituminous binder is undesirable, because it may lead to the generation of a higher number of cracks in wearing course in low-temperature periods. Therefore, **the lower the physical hardening value, the better**. This is confirmed by previous tests performed by ORLEN Asphalt, which have revealed the correlation between LCT ( $S=300$  MPa) and stiffness of binder in the temperature of  $-16^{\circ}\text{C}$ , and the temperature of cracking of asphalt mixture in the TSRST test [28].

The value of physical hardening is affected by temperature and the length of time of freezing the bitumen. **Lower temperature and longer time of freezing result in higher effect of physical hardening**. This means that bituminous pavement may be subject to excessive stiffening during lengthy periods of freezing temperatures, and the lower the long-term freezing temperature is, the higher the risk of cracking of pavement.



## Chapter 9

### RESISTANCE OF BINDER IN MASTIC ASPHALT (MA) TO SHORT-TERM AGEING

#### 9.1. INTRODUCTION

Short-term ageing of bituminous binders is a complex process. It depends on many factors, such as nature, structure and group composition of bitumen and on other external factors. It is a chemical process, which irreversibly changes the functional properties of bituminous binders.

Hardening of bituminous binders due to high temperatures is called *technological or short-term ageing*.

The degree of ageing of bituminous binders affects the duration of faultless operation of pavement, whether we are using conventional paving-grade bitumens, or polymer-modified bitumens. The most intense bitumen ageing processes take place when bitumen is mixed with hot aggregate in the asphalt plant's mixer. Temperature at that time is the highest, with free access to air oxygen, and the layer of bitumen on the aggregate is very thin. During mixing of aggregate with bitumen, the vaporisation of light fraction and oxidation of bitumen components is the fastest and most intense.

As a result of oxidation, the composition and chemical structure of bitumen undergoes irreversible changes, which result, among others, in:

- reduction of penetration,
- softening point increase,
- increase (worsening) of Fraass breaking point,
- increase of viscosity.

From the perspective of pavement durability, it is required to check the effects of short-term ageing on at least the primary bitumen properties, such as: penetration, softening point, viscosity and elastic recovery (for modified bitumen).

The significance of short-term bitumen ageing must not be excluded from this discussion on bitumen as a construction material. Importantly, **bitumen build-in in the pavement has already undergone short-term ageing**. Tests on bitumen sensitivity to ageing are therefore very relevant.



## 9.2. PURPOSE AND SCOPE OF TESTS

Changes in the chemical structure of bitumen, caused by oxidation, usually lead to the deterioration of its properties. Bitumen becomes more brittle and stiff, its viscosity and softening point increase, while penetration and ductility are reduced. However, hot mixtures are not equally susceptible to ageing. In the case of mechanically compacted mixtures, i.e. ones compacted by rolling (e.g. asphalt concrete AC or SMA), the temperature of production of mma usually does not exceed 170-180°C, and this is a temperature that is considered to be a relatively safe limit for the heating of bituminous binders.

In the case of mastic asphalt, the mixture production temperature may even reach 240°C, and the time of mixing/storage of mma may often amount to 6 hours. During this time, the bitumen experiences very intense ageing processes.

In order to verify the degree of destruction of bituminous binders used for the production of mastic asphalt MA, in 2015 ORLEN Asphalt carried out research intended to establish the maximum safe temperatures for the heating of bitumen, and the possible time of storage of this bitumen without losing its stable properties.

The tests of the resistance to short-term ageing were carried out on hard bitumens – usually used for the production of mastic asphalt:

- Paving-grade bitumen 35/50
- BITREX 35/50-57/69 multigrade bitumen
- ORBITON 25/55-60 modified bitumen

All tested binders were aged using the TFOT (*Thin Film Oven Test*) method, according to standard EN 12607-2 „*Bitumen and bituminous binders. Determination of the resistance to hardening under influence of heat and air. Part 2: TFOT method*“, for the duration time and temperature values which reflect the actual conditions of production of MA. Due to technical reasons, the RTFOT method was not used as reference for the evaluation of bitumen ageing. The research plan assumed the heating of binders in temperatures in excess of 200°C, which turned out to be impossible to perform in an RTFOT oven.

The degree of ageing of bituminous binders was evaluated on the basis of the analysis of the changes in primary properties, i.e.:

- Penetration at 25°C as per EN 1426
- R&B softening point as per EN 1427
- Fraass breaking point as per EN 12593
- Dynamic viscosity at 60°C as per EN 13702-1

Each binder was heated in the TFOT oven for 75, 120, 240, 360 and 480 minutes at the following temperature values: 163°C, 200°C, 220°C, 240°C, covering both the typical temperature range used for “hot” technologies (<200°C), as well as temperatures applied in Mastic Asphalt technology (>200°C).

Above all, the tests helped to obtain information specific for mastic asphalt, but some of the results may also be applied to conventional mixtures, i.e.: AC, SMA, PA.

## 9.3. RESULTS

### 9.3.1. Change of penetration at 25°C

The penetration test was carried out in accordance with standard EN 1426 „*Bitumen and bituminous binders. Determination of needle penetration*“.

Figures 9.1-9.3. demonstrate the change in penetration of tested bituminous binders as a function of heating time and temperature.

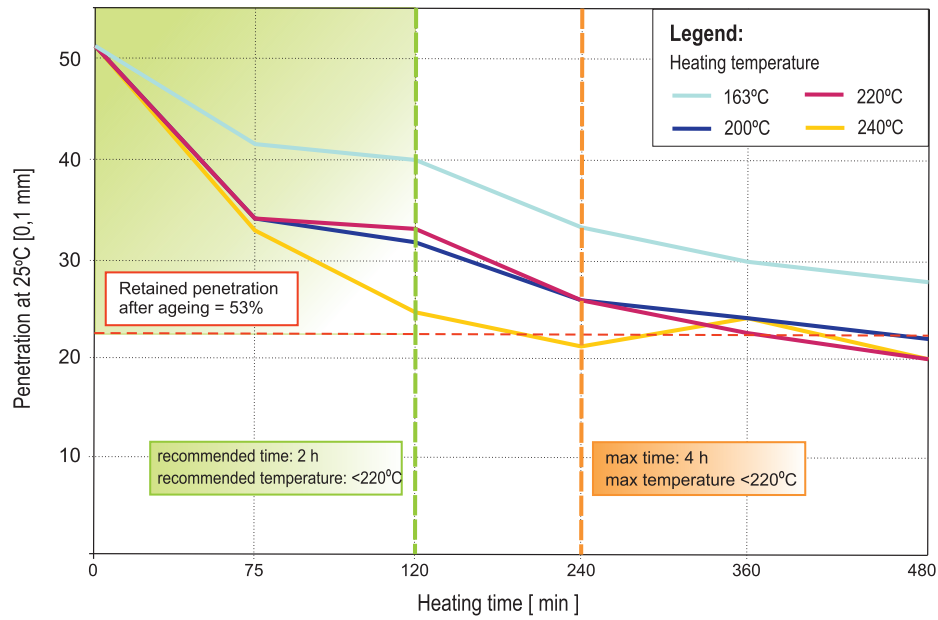


Fig.9.1. Change of the value penetration for 35/50 paving-grade bitumen

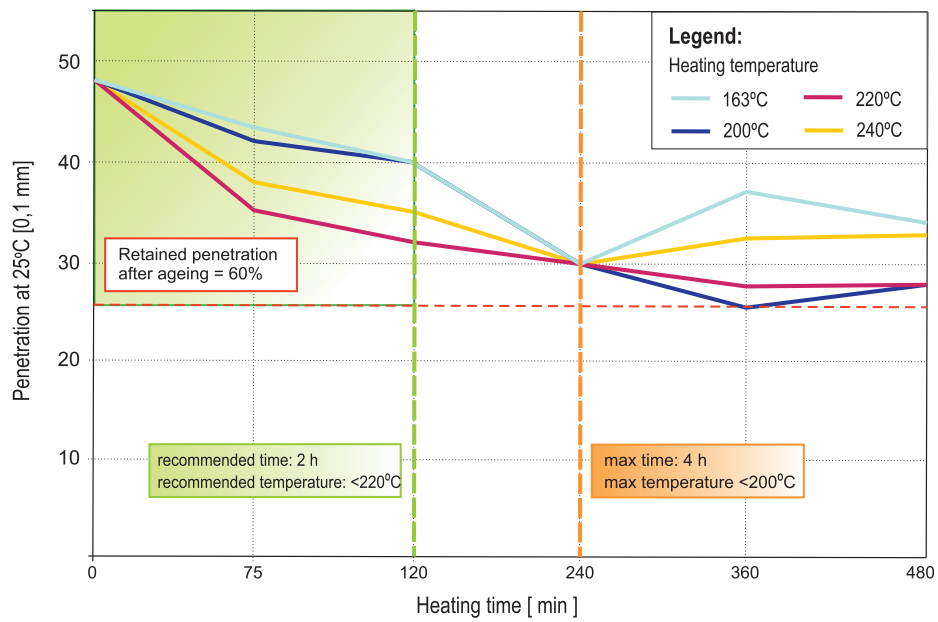


Fig.9.2. Change of the value penetration for BITREX 35/50-57/69 multigrade bitumen

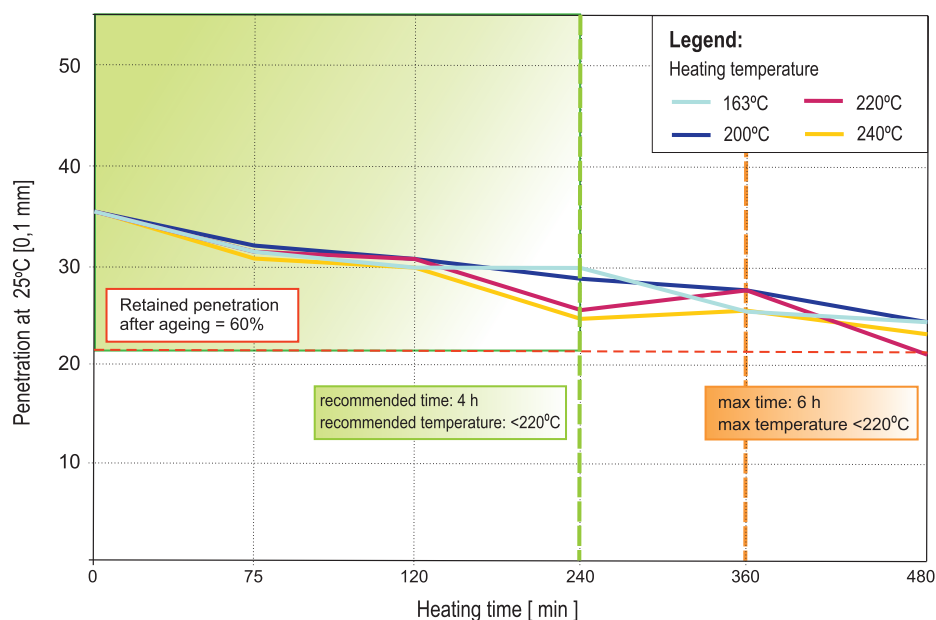


Fig. 9.3. Change of the value penetration for ORBITON 25/55-60 modified bitumen

As a result of short-term ageing, the value of penetration drops – this is directly connected with the process of bitumen hardening. *Retained penetration after RTFOT*, expressed in [%], is the standard parameter which defines the acceptable degree of ageing of binder on the basis of penetration test at 25°C.

In the above charts, the red line indicates the acceptable value of penetration that can be demonstrated by the specific bituminous binders, so that their parameters are still compliant with the standard after the hardening process. It must be noted, however, that the standard defines requirements for bitumen after RTFOT ageing, and that method is more aggressive than the TFOT method. The green area indicates the safe heating range (recommended by ORLEN Asphalt) for each bituminous binder.

### 9.3.2. Change of R&B softening point

The testing of the changes in bitumen softening point was carried out on the basis of standard EN 1427 „*Bitumen and bituminous binders. Determination of the softening point. Ring and Ball method*”.

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

Fig. 9.4-9.6 demonstrate the changes in the value of R&B softening point of tested bituminous binders as a function of heating time and temperature.

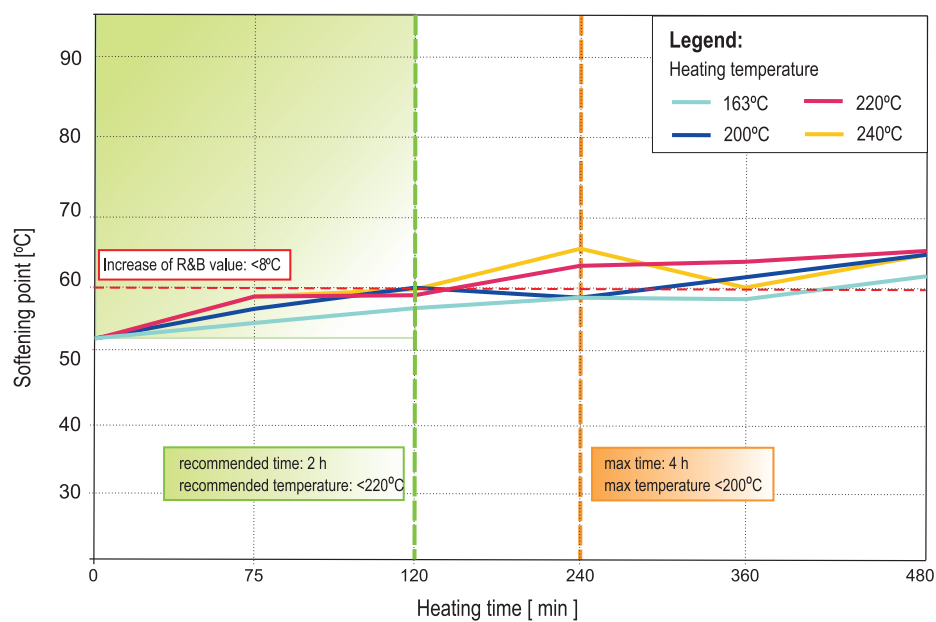


Fig. 9.4. Change of the value of R&B softening point for 35/50 paving-grade bitumen

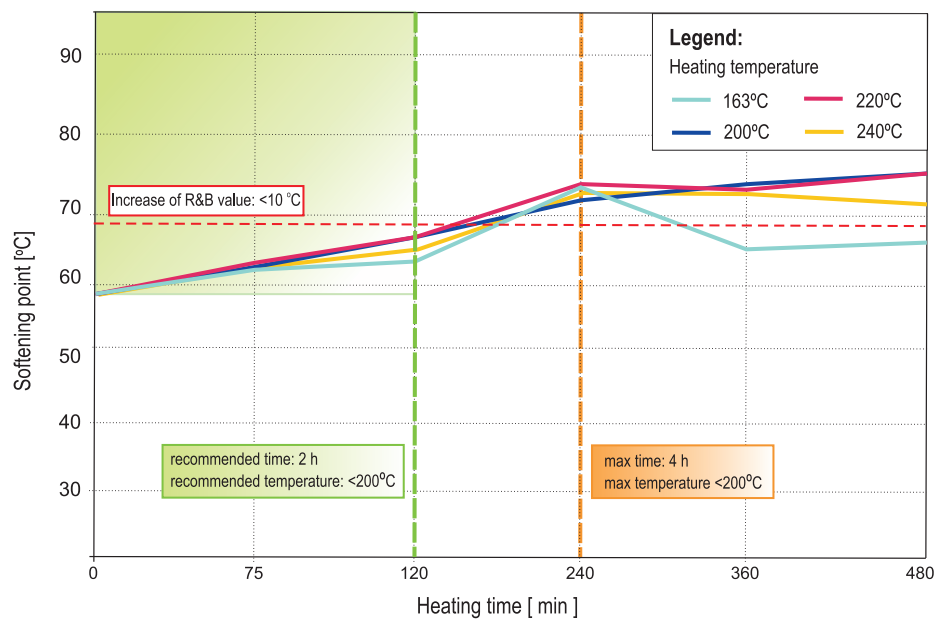


Fig. 9.5. Change of the value of R&B softening point for BITREX 35/50-57/69 multigrade bitumen

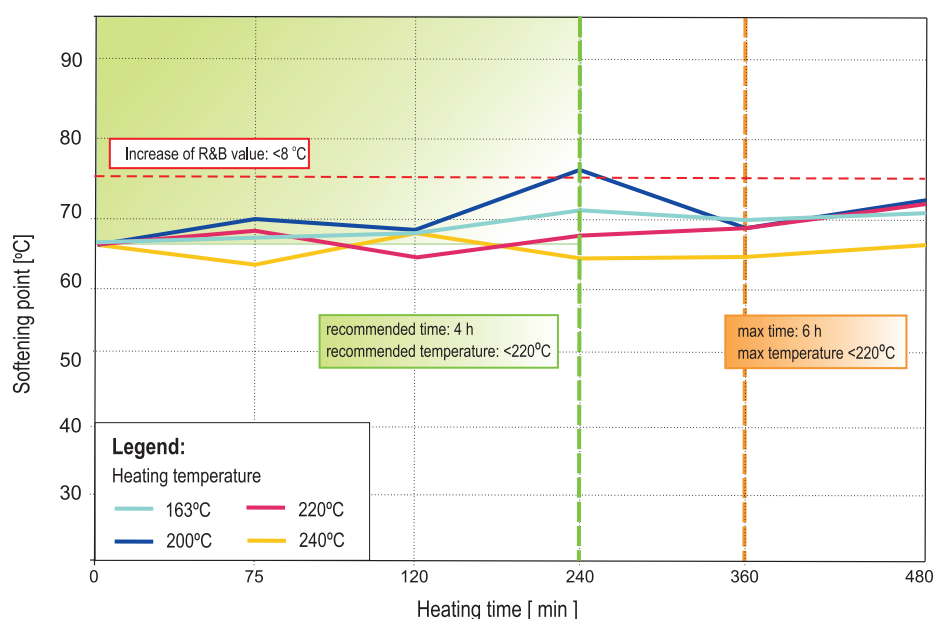


Fig. 9.6. Change of the value of R&B softening point for ORBITON 25/55-60 modified bitumen

As a result of the ageing of bituminous binders, the softening point rises, which is also the direct consequence of the hardening of bitumen in high temperatures.

The difference between the softening point of bitumen before and after RTFOT (acceptable increase of R&B) is the standard parameter which defines the acceptable increase of R&B after the oxidation process. As in the case of the analysis of penetration charts, a red line indicates the standard acceptable increase of the value of R&B temperature for each bitumen. Also in this case it must be noted that the standard defines this parameter for the RTFOT method. The green area indicated the safe time and short-term temperature that are recommended by ORLEN Asphalt for each type of bitumen during the production of MA.

### 9.3.3. Testing of Fraass breaking point

The Fraass breaking point is another test which was carried out to evaluate the properties of bituminous binders after short-term ageing.

The breaking point determines low-temperature bitumen properties and represents an approximate (conventional) lower limit of viscoelastic properties. The breaking point test was carried out in accordance with standard EN 12593 „Bitumen and bituminous binders. Determination of the Fraass breaking point”.

Figures 9.7.-9.9. show the changes in Fraass breaking point achieved for the tested bituminous binders.

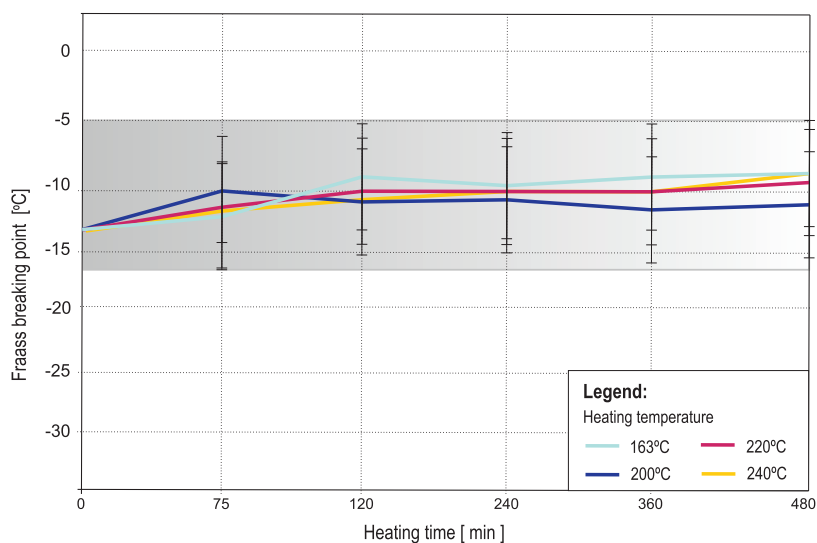


Fig. 9.7. Change of the value of Fraass breaking point for 35/50 paving-grade bitumen

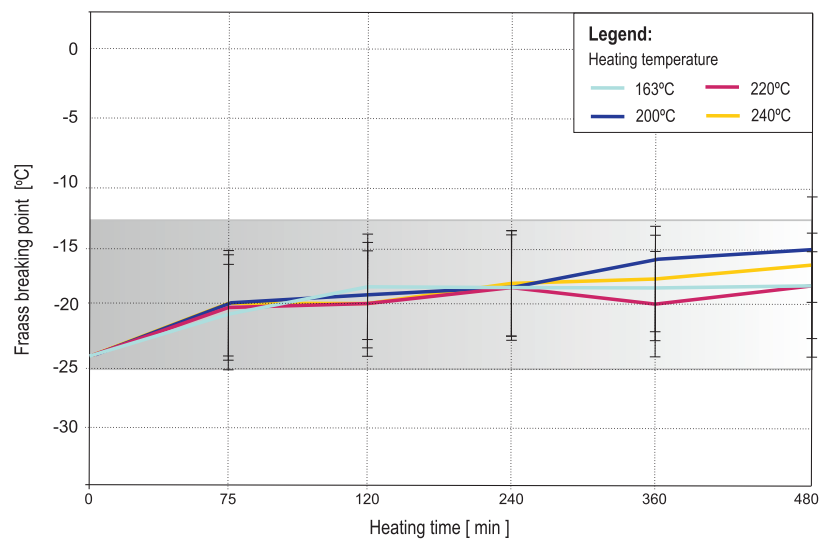


Fig. 9.8. Change of the value of Fraass breaking point for BITREX 35/50-57/69 multigrade bitumen

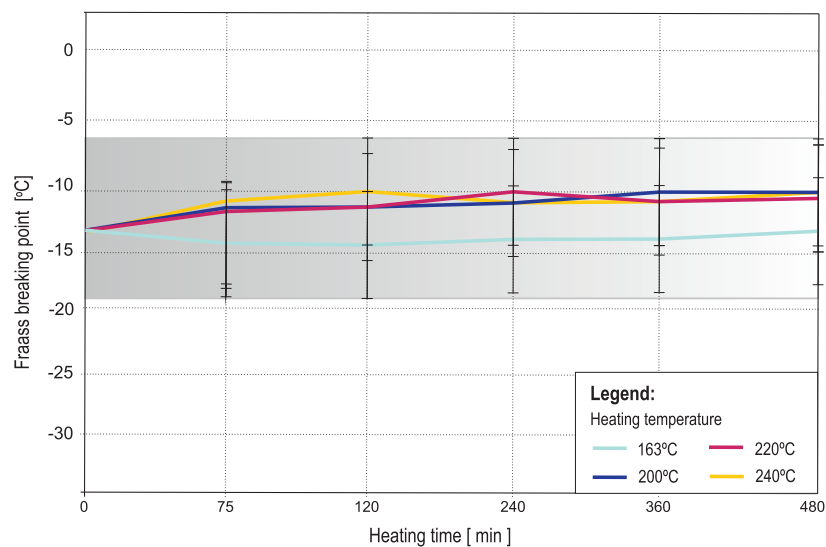


Fig. 9.9. Change of the value of Fraass breaking point for ORBITON 25/55-60 modified bitumen

The grey area in the above charts indicates the uncertainty of measurement, determined on the basis of the validation of the method at the testing laboratory. Please note that in fact each of the obtained results lies within the  $\pm 3^{\circ}\text{C}$  range. This means that in the case of bitumen with a determined Fraass breaking point of  $-10^{\circ}\text{C}$ , the correct result that lies within the limits of measurement uncertainty is within the  $-7^{\circ}\text{C} \div -13^{\circ}\text{C}$  range. The spread of the obtained results is therefore very wide.

Due to poor precision of the test, the practicality of the Fraass method has been questioned, as it arguably does not reflect the actual behaviour of bitumen in the pavement. It can be stated therefore that breaking point established as above is not a suitable method to verify low-temperature properties of bituminous binders after short-term ageing.

### 9.3.4. Change of dynamic viscosity at $60^{\circ}\text{C}$

The testing dynamic viscosity by cone and plate method was carried out according to standard EN 13702-1 „*Bitumen and bituminous binders. Determination of dynamic viscosity of modified bitumen. Part 1: Cone and plate method*”

The increase in viscosity represents one of the main changes of rheological properties of bituminous binders occurring during short-term ageing. The charts below demonstrate changes of the dynamic viscosity of tested bitumen types, depending on the heating time and temperature.

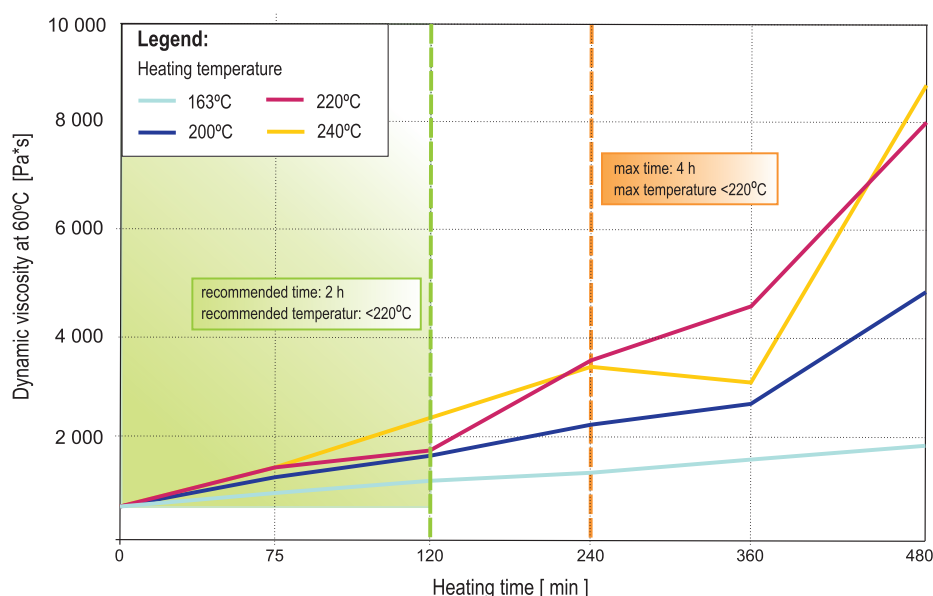


Fig. 9.10. Change of dynamic viscosity at  $60^{\circ}\text{C}$  for 35/50 paving-grade bitumen

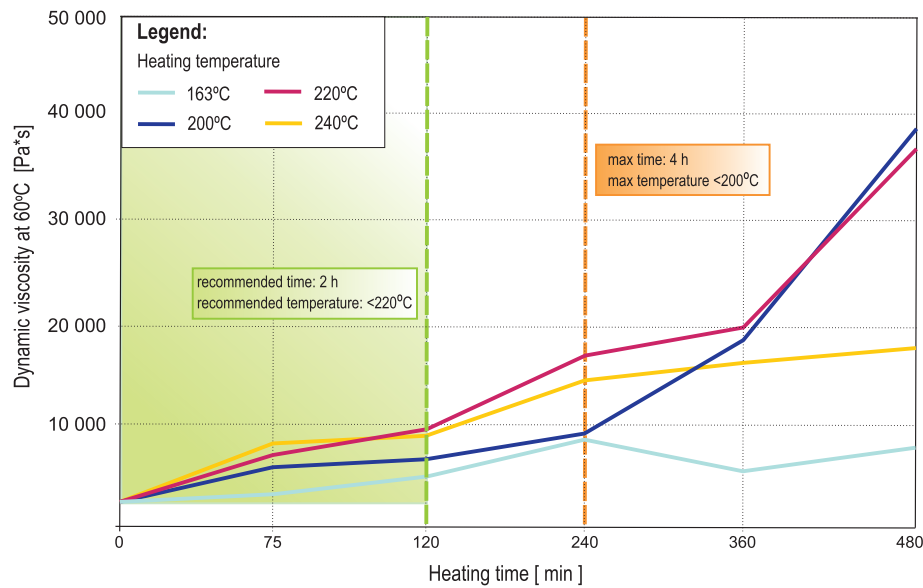


Fig. 9.11. Change of dynamic viscosity at 60°C for BITREX 35/50-57/69 multigrade bitumen

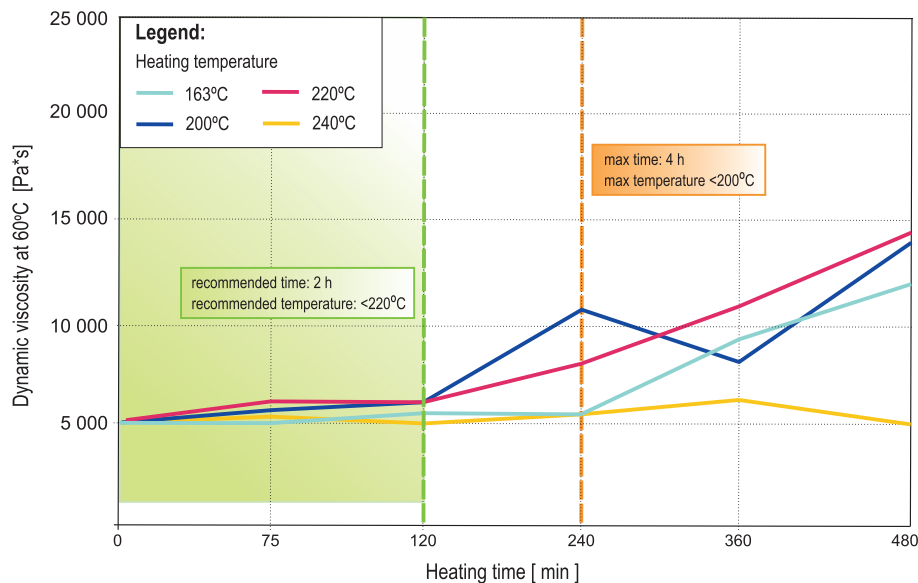


Fig. 9.12. Change of dynamic viscosity at 60°C for ORBITON 25/55-60 modified bitumen

Significant increase of viscosity of bitumen during heating indicates that this characteristic is very sensitive to the short-term ageing process.

EN standards for the specific types of bitumen do not define the acceptable level of increase of viscosity after ageing. Therefore there is no standard reference for the maximum level of increase of viscosity during heating. However, this characteristic must be seriously taken into account, because excessive increase of viscosity during ageing directly affects process temperatures and may significantly complicate e.g. the application of the mixture (especially in the case of manual application).

### 9.3.5. Ageing of polymer-modified bitumens

The complexity of the ageing process increases, when we are dealing with polymer modified bitumens. Although the analysis of the above tests clearly shows that these binders are most resistant to the effects of high temperatures, one must be very careful during the application of this bitumen group, because the bitumen polymer network



may be damaged during excessive heating. The properties of modified bitumens will be therefore dependant also on the degree of decomposition of the used polymer. Thus the analysis of only the primary parameters is insufficient to evaluate the properties of these binders after the oxidation process.

One of the methods which allows us to look into the chemical structure of binders is the test with the use of fluorescent microscope with a UV lamp, via image analysis in reflected light. The test is carried out on the basis of standard EN 13632 „Bitumen and bituminous binders. Visualisation of polimer dispersion in polymer-modified bitumen”.

The photographs below present the dispersion of SBS polymer in ORBITON 25/55-60 modified bitumen after ageing:

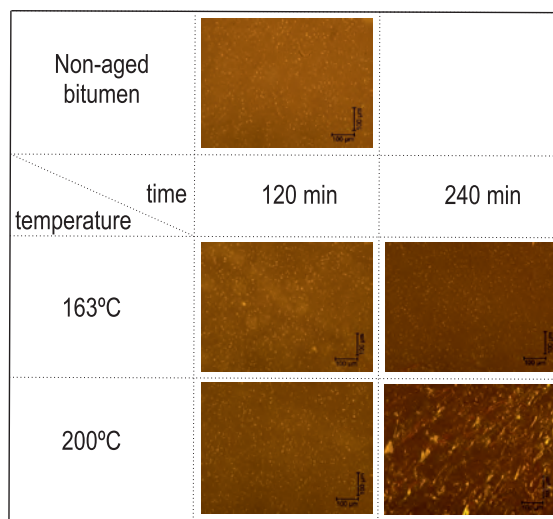


Fig. 9.13. Change of structure of SBS polymer in ORBITON 25/55-60 modified bitumen after ageing

At 163°C, during the heating time of up to 4 hours, no major changes in the polymer bitumen microstructure were observed. If the temperature is raised to 200°C, the microstructure of bitumen begins to change already after 240 minutes of heating.

## 9.4. SUMMARY

The main factor responsible for the ageing of bitumens is the reaction of its components with air oxygen. High temperatures, as well as the storage time of the mixture affect the degree of ageing of the binder. The higher the temperature and the longer the time of heating of bitumen, the faster and more intensive is the ageing process.

The charts shown above suggest that ORBITON 25/55-60 modified bitumen is the most resistant to the effects of high temperatures – its primary properties remain stable during the heating period for the longest time. Multigrade bitumen is the most sensitive to high temperatures and longer heating times, and its primary parameters deteriorate unpredictably after 240 minutes of heating, already in temperatures above 200°C. Paving-grade bitumen demonstrates intermediate properties between the multigrade and modified bitumens.

Finally, we recommend the implementation of the following technological conditions during the production of MA Mastic Asphalt:

35/50		BITREX MG 35/50-57/69		ORBITON PMB 25/55-60	
Time	Temperature	Time	Temperature	Time	Temperature
Max. up to 4h Recommended up to 2h	< 220°C	Max. up to 4h Recommended up to 2h	< 200°C	Recommended up to 4h	< 220°C

## Chapter 10

### BITUMEN APPLICATION TECHNOLOGY

The application of asphalt binders requires, most of all, expertise on optimum process temperatures and particular criteria for bitumen sample handling.

The following chapter sections provide technical details which may be of interest to laboratory staff and process/technology divisions of road engineering companies.

Table 10.2 summarises all key information about process temperatures for the application of bitumen manufactured by ORLEN Asphalt.

#### 10.1. LABORATORY GUIDELINES

##### 10.1.1. Determination of process temperatures

Bitumen types differ from one another in terms of their characteristic viscosities within 60-165°C temperature “window” (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 (see Chapter 9). The simulation of short-term ageing under laboratory conditions takes place in the RTFOT device, and in the PAV or RCAT vessel for long-term ageing simulation.

The 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 on-site 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. At the same time, 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 usually already undergone short-term ageing. Therefore we suggest taking the viscosity value measured after RTFOT to determine the compaction start and end temperatures.

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.

The viscosity-temperature relation curves for each type of bitumen have been shown in figures in Chapters 2-4.

In examination of process 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 temperature should correspond to the general conditions at the mixing plant and on the construction site. If the temperature adopted at the lab is too high, the volumetric density of the asphalt 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 compaction is significantly lower, this will in practical terms prevent the achievement of the required layer compaction indexes. 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 very important.

### 10.1.2. Bitumen samples in the lab

The road laboratory receives asphalt 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 handling of bitumen 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 be avoided. Therefore we suggest using a greater number of small samples (for one-off use) rather than a single, large bitumen-holding container.

If the bitumen sample is enclosed in one, large container (e.g. 10 kg), heating the container for the first time is recommended, its homogenisation through mixing, and subsequently pouring into a few smaller, labelled containers, to be used later.

The handling of bitumen samples for tests is specified in EN 12594 *"Bitumen and asphalt 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 up to the temperature exceeding 200°C,
- **containers of the volume of less than 1 litre**, heating time of up to 120 minutes, oven heating temperature: not more than 100°C above the expected bitumen softening point,
- **containers of the volume of 1–2 litres**, heating time of up to 3 hours, oven heating temperature: not more than bitumen softening point +100°C,
- **containers of the volume of 2–3 litres**, heating time of up to 3.5 hours, oven heating temperature: not more than bitumen softening point +100°C,
- **containers of the volume of 3–5 litres**, heating time of up to 4 hours, oven heating temperature: not more than bitumen softening point +100°C,
- **containers of the volume of more than 5 litres**, heating time of up to 12 hours, oven heating temperature: not more than bitumen softening point +50°C, temperature shall be raised adequately for the last 2 hours.

In the case of modified bitumen, the procedure provided by the sample supplier shall be implemented. If no information has been delivered regarding polymer-modified bitumen (accordant with EN 14023), the oven temperature should be in the range between 180°C and 200°C, disregarding the softening point.

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.

If the sample is contaminated with coke or with a fine mineral material, the sample may be sieved through a heated sieve (0.5 mm lacing) before the extraction of the sample for testing. The presence of coke or a fine mineral material and sieving (filtering) should be recorded in the test report.

Bitumen samples obtained from:

- the extraction of asphalt mixtures (as per EN 12697-1, EN 12697-2, and EN 12697-4),
- tests of the resistance to hardening or ageing (as per EN 12607-1, EN 12607-2 and EN 12607-3 or EN 14769 or any other relevant standard which concerns hardening or ageing)

should be prepared and tested in accordance with adequate extraction and testing methods.

### 10.1.3. 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”, “alkaline” and “intermediate” aggregates are used in road engineering, and the specific pH status results from high or low content of SiO<sub>2</sub> (silica) in the rock. The general rule is that “acidic” aggregates (as granite) bear little affinity to bitumen and require the application of additives that improve bitumen adhesion. “Alkaline” aggregates, such as limestone, are characterised 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, asphalt mixture resistance to water and frost:

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

In the case of weak affinity between bitumen and aggregate, adhesion promoters (agents) are used. Adhesion evaluation can follow e.g. the test described in EN 12697-11 method A on the selected aggregate mixture fraction. The adhesion of binder to aggregate should be at least 80% after 6 hours of testing (requirement in Poland).

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 asphalt mixture resistance to water and frost is the ITSr test as per EN 12697-12.

## 10.2. 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 *Asphalt mixtures – Requirements – Part 21: Factory Production Control*) state 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 build-up 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 build-up 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.

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 tank walls or heating coils. This causes an additional coke buildup at the bottom of the tank.

In the course of bitumen mixing with aggregate at the asphalt mixing plant, 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 the production of mixtures without continuous granulation (SMA mix or PA mix), 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*).

Table 10.1. Bitumen ageing in storage tanks

Reasons for bitumen ageing in the tank	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	<p>Bitumen circulation is commonly used to homogenise it in the tank. 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.</p> <p>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.</p>
Tank structure	The most desirable situation is when the ratio of bitumen surface and its volume in the silo is small, and that is why storage tanks for bitumen should be vertical, because then the tank height-to-diameter ratio is high.

Detailed information on the storage of all bituminous binder types has been presented in Chapters 2-4.

### Other recommendations

Before changing the type or grade of bitumen in the tank, the user must make sure 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 silo and laboratory control. Binders from different manufacturers should not be mixed.

Multiple heating and cooling cycles for ORBITON modified bitumens and ORBITON HiMA highly modified bitumens 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. Binder properties must be necessarily tested after heating. Note – not every type and sort of bitumen can be stored in this way (see Chapters 2-4), especially polymer modified binders shouldn't be stored over winter time.

Bitumen temperature in the course of storage should not exceed values indicated in Table 10.2.

### 10.3. ASPHALT MIXTURE PRODUCTION

The viscosity of bitumen supplied to the hot mix plant should be small enough to enable its unloading from the road tanker. Since bitumen viscosity is strictly related to its temperature (the higher the temperature of bitumen, the lower its viscosity), 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.

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 asphalt 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 10.2 do not apply to asphalt mixtures containing an agent for production and placement temperature reduction.**

The storage period in the silo 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 or modified bitumen),
- presence of additives such as stabilisers, modifiers or adhesion agents,
- silo condition and equipment (thermal insulation, heat tracing),
- asphalt mixture quantity in the silo.

### 10.4. TRANSPORT OF ASPHALT MIXTURES

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 asphalt binder.

**Diesel oil or any other mineral oils 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 (temperature  $< +5^{\circ}\text{C}$ , strong wind  $> 10\text{ m/s}$ , 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 could be considered [1]:

- **blue smoke** – present above 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 “melts”** 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 compactability on the site; typically the mixture should form a dome-like shape after loading,
- **aggregate is not entirely coated 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,
- **coarse aggregates are covered with bitumen bubbles** – this effect looks as if the bitumen is boiling/foaming on the surface of the aggregate. The reason for this is excessive dampness of aggregate, which the mixing plant dryer was not able to eliminate. This effect occurs more frequently with highly-absorbable aggregate types and after long-lasting rainfall.

## 10.5. PLACEMENT

Asphalt concrete mixes with high stiffness modulus (AC EME) 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 (infrared) 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 (MA) mix sometime can’t be placed manually due to its high viscosity. Mechanical equipment is recommended, along with additives to reduce the placement temperature. Attention must be paid to the temperature and time of storage of mastic asphalt mixture, detailed guidelines can be found in Chapter 9.



## 10.6. PROCESS TEMPERATURES

Table 10.2. Minimum and maximum temperature of bitumen and asphalt mixtures depending on bitumen type

Bitumen type	Paving Grade Bitumen				Polymer-modified bitumen					Bitumen highly modified with polymers		
	EN 12591 annex NA				EN 14023 annex NA					EN 14023 annex NA		
	Modified bitumen 20/30	Modified bitumen 35/50	Modified bitumen 50/70	Modified bitumen 70/100	ORBITON 10/40-65	ORBITON 25/55-60	ORBITON 45/80-55	ORBITON 45/80-65	ORBITON 65/105-60	ORBITON 25/55-80 HiMA	ORBITON 45/80-80 HiMA	ORBITON 65/105-80 HiMA
	Temperature [°C]											
Laboratory												
Marshall sample compaction temperature/gyratory press	155-160	140-145	135-140	130-135	150-155	145-150	145-150	150-155	145-150	150-160	150-155	145-150
Component temperature at the mixing plant												
Pumping of bitumen	> 145	> 140	> 130	> 120	> 150	> 150	> 150	> 150	> 150	above 160	above 150	above 140
Short-term bitumen storage at the mixing plant	up to 190	up to 190	up to 190	up to 180	up to 190	up to 190	up to 190	up to 190	up to 190	up to 200 (<3 days)	up to 190 (<3 days)	up to 190 (<3 days)
										up to 160 (>3 days)	up to 160 (>3 days)	up to 160 (>3 days)
Ready asphalt mixture temperature in the mixing plant's mixer												
Asphalt concrete (AC)	<185	<180	<175	<170	<185	<185	<185	<185	<185	max. 195	max. 195	max. 185
SMA	—	—	<175	<170	—	<185	<185	<185	<185	max. 195	max. 195	max. 185
Porous asphalt (PA)	—	—	—	—	—	—	<185	<185	<185	max. 195	max. 195	max. 185
Mastic asphalt (MA)	<220 <sup>a</sup>	<220 <sup>a</sup>	—	—	<230 <sup>c</sup>	<230 <sup>c</sup>	<230 <sup>c</sup>	—	—	max. 220	max. 220	--
Temperature on site												
Minimum temperature of the supplied asphalt mixture in the spreader's hopper	150	145	140	135	160	155	155	160	160	180	180	175
End of effective compaction temperature	>120	>115	>110	>100	>125	>125	>120	>125	>120	>150	>145	>120
a) mastic asphalt (MA) residence time in the bitumen boiler of up to 6 h, at the specified temperature; higher temperature of mastic asphalt, up to 230°C, is permitted if boiler residence time does not exceed 2 h b) mastic asphalt (MA) residence time in the bitumen boiler of up to 4 h, at the specified temperature c) mastic asphalt residence time in the bitumen boiler of up to 4 h, at the specified temperature; higher temperature of mastic asphalt, up to 230°C, is permitted if boiler residence time does not exceed 2 h												



## Chapter 11

### OCCUPATIONAL HEALTH, SAFETY AND ENVIRONMENT PROTECTION

#### 11.1. INTRODUCTION

General aspects of occupational health, safety and environment discussed below apply to petroleum-derived bitumens used in road construction, supplied by ORLEN Asphalt. Standard-defined properties of bitumen and current test results have been presented and discussed in the first part of the Handbook, (Chapters 2-5).

Although bitumen is not classified as a hazardous substance, MSDSs for bitumen are broadly available to the users in order to ensure maximum application safety and full product information.

Extensive ecological and toxicological information as well as risk identification, acting in the case of fire or inadvertent environmental release for all ORLEN Asphalt products are provided in MSDSs, which are available on the company web page [13].

All MSDSs provided to the clients are compliant with the latest EU regulations, i.e. with the REACH (*Registration Evaluation and Authorisation of Chemicals*) resolution and the CLP (*Classification, Labelling, Packaging*) resolution.

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.

It must be emphasised that the mixing of paving-grade bitumens with other substances or additives should be considered for the identification of hazards and risk assessment. Such mixtures may generate additional hazards. However, the manufacturers of those mixtures are responsible for the changes that may cause bitumen to become a substance which is hazardous to human life or to the environment.

#### 11.2. POTENTIAL HEALTH HAZARDS DURING PRODUCTION, STORAGE, TRANSPORT AND APPLICATION OF BITUMINOUS BINDERS

##### 11.2.1. Transport of bitumen

Bitumen transport is governed by international rules on hazardous substance transport. **Bitumens are classified as hazardous due to their high transport temperature.**

The vast majority of ORLEN Asphalt's products are 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.

### 11.2.2. 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 occur in different situations: during routine work actions (e.g. when 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 protection equipment must be used at all times when working with hot bitumen, such as e.g.:**

- helmet with face shield and neck protection. It should be remembered that safety glasses protect only your eyes!
- safety clothes and shoes,
- heat-resistant safety gloves (it must be ensured that hot bitumen cannot get into the gloves!)

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.

National regulations must be checked and appropriate equipment is to be selected adequately.

### 11.2.3. Fire (preventive actions)

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 flash point. Importantly, flash point (Cleveland open cup method) of paving-grade bitumens discussed in this Handbook is over 310°C (Chapter 2-4 of the Handbook, property tables). The latest 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 are not considered as explosive on the basis of structural considerations and oxygen balance [6]. In order to minimise the risk of vapour generation, 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 may build up on the walls and decks of tanks, and self-combust if oxygen is present.

### 11.2.4. Suppression of bitumen fire

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 may cause the very dangerous and sudden splattering of hot bitumen. Water can only be used for cooling down hot surfaces.

Appropriate extinguishing agents include carbon dioxide, dry chemical, foam and sand. For details contact to relevant Fire Brigade Dept.

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.

### 11.2.5. 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. Bitumen foaming may be accompanied by hot bitumen splattering.

An important consideration for the loading procedure of hot bitumen is to check whether the tanker contains water, and for the unloading operation – whether hoses do not contain water or moisture.

The bitumen storage tank should be dry at all times. An empty and cold tank should be initially filled with a small quantity of bitumen 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.

### 11.2.6. 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. Additional research and production process monitoring are being continued 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), there is no proof that bitumen vapours are hazardous to personal health of the employees.

Research carried out in July 2009 regarding the cases of lung cancer among employees who had contact with bitumens, performed by IARC (*The International Agency for Research on Cancer*) have not revealed any relation between the risk of lung cancer and the exposure to bitumen vapours [6].

Although bitumen vapours have not been classified as hazardous to human health, nevertheless contact with this material should be avoided when working with hot bitumen, and the same applies to the inhalation of vapours or mist generated by the heated 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. Emission of bitumen vapours should therefore be minimised.

Employees' exposure to bitumen vapours/smoke should be minimised through the application of the so-called best practices [6]:

- keep process temperatures as low as possible (use warm-mix asphalt techniques),
- 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.

### 11.2.7. Hydrogen sulfide

The elemental composition of bitumens varies depending on the chemical properties of petroleum oil used for their production and on the production methods [9]. The elemental composition of the majority of bitumens also includes a small quantity of sulphur. Therefore, if hot bitumen is stored in closed tanks over a long time, hydrogen sulphide may be released which concentration – in extreme cases – may reach dangerous levels. Before entering empty bitumen tanks, they should be ventilated beforehand, and then left with a constant air inflow and in lowered

temperature. After the preparation of a tank as above before the entrance of any employee inside, additional analyses of the atmosphere inside must be performed to examine the presence of oxygen or potential concentration of explosive or toxic substances inside. The analysis should be performed not before 1 hour ahead of the planned entrance. The employee entering the tank should be adequately equipped with personal protection means.

Latest national regulations must always be checked and appropriate course of action is to be selected adequately.

#### **11.2.8. Polycyclic Aromatic Hydrocarbons (PAH)**

The presence of minor quantities of Polycyclic Aromatic Hydrocarbons – in short PAH in bitumen vapours raises concerns over their potential influence on the health of employees exposed to the effects of bitumen or bitumen vapours. It has been stated that some polycyclic aromatic hydrocarbons demonstrate carcinogenic properties. The US Environmental Protection Agency has drawn up a list of fifteen hydrocarbons deemed to be toxic. Out of those hydrocarbons, benzo(a)pyrene demonstrates the strongest carcinogenic properties. Carcinogenic effects of this compound are in effect when its content in the binder exceeds 50 mg/kg. The maximum content of benzo(a)pyrene in bitumen is 4 mg/kg, while the total PAH content does not exceed 40 mg/kg [2].

ORLEN Asphalt additionally conducts the tests of its products in terms of ecological and toxicological aspects. During the recent years, a series of tests were carried out to define the content of Polycyclic Aromatic Hydrocarbons (PAH) in mg/kg in samples of the commonly used 35/50 pavement-grade bitumen using the gas chromatography (GC) method. In all tested samples, the content of benzo(a)pyrene did not exceed 0.5 mg/kg, and the total for PAH was approx. 4.5 mg/kg.

Despite the existence of small amounts of PAH in bitumen, there is no proof that contact with bitumen or bitumen vapour may increase the risk of lung cancer.

### **11.3. SUMMARY**

In conclusion, it is worth recalling the final conclusion from the detailed paper “Clinical-control examination of the cases of lung cancer embedded in the cohort of employees of the European bitumen industry. Final report of July 2009” to the report published in 2009 by the International Agency for Research on Cancer (IARC). The paper was developed by the Occupational Medicine Institute of J. Nofera in Łódź, Poland [21]. It was concluded that no cohesive proof was found for the existence of a causal connection between the (inhalable and dermal) exposure to bitumen and the risk of actual lung cancer incidence. Above-average number of lung cancer incidents reported during the cohort study for workers exposed to bitumen can be rather attributed to the intensity of smoking and possible exposure to coal tar. The remaining tested professional factors did not play any significant role in the generation of the risk of lung cancer.

One must also be aware that bitumen present in the asphalt mixtures that makes up the asphalt pavement (roads, airports, other pavements) is in solid form and does not represent a hazard to health or natural environment. What's more, bitumen is one of very few road construction products that is considered as ecological, because it can be 100% – recyclable and then re-used for road pavement construction.

## Chapter 12

### BITUMEN LABORATORIES OF THE ORLEN CAPITAL GROUP

#### 12.1. INTRODUCTION

The importance of having an efficient, effective and reliable laboratory is an essential issue that no-one needs reminding of. Regular laboratory control allows the company to fully monitor the faultless progress of ongoing production processes and to efficiently react to any existing problem.

Within the ORLEN Capital Group, production control tests and some of the research programs are carried out at ORLEN Laboratorium (Poland) and at Unipetrol Centre of Research and Education (UniCRE) (Czech Republic).

The ORLEN Laboratorium company has been present on the Polish market for almost 15 years. The main office of the company, together with the Central Laboratory, is located in the area of the PKN ORLEN refinery in Plock.

The Unipetrol Centre of Research and Education (UniCRE) chemical laboratory is located in Litvinov in the Czech Republic, in the area of the Česka Rafinerska refinery, which also belongs to the ORLEN CG.

The ORLEN Capital Group Laboratories are mainly responsible for analytical activities, and concentrate on regular laboratory control of the ongoing processes performed by various manufacturing systems. The other area of activity for the laboratories are product implementation and research and development tasks performed for the entire ORLEN CG.

#### 12.2. ORLEN LABORATORIUM

##### 12.2.1 General information

ORLEN Laboratorium S.A., which forms a part of the ORLEN CG, is one of the foremost Polish company which carries out analyses of fuels, petroleum products (including bituminous binders), water, sewage, soil, air, fertilizers and plastics.

In order to meet our customers' needs and to confirm the high standard of our services ORLEN Laboratorium company operate in the system compatible with international standard EN ISO/IEC 17025. This standard includes both the guidelines governing the quality management in the laboratory and the technical requirements influencing the flawless performance of the tests.

The flawless functioning of the management system and the acknowledgement and recognition of technical competences were confirmed by the positive assessment by the independent auditors of the Polish Centre of Accreditation and by the Certificate of Accreditation No AB 484 which company obtained on the 9th of April 2004.

The quality and credibility of services provided by ORLEN Laboratorium is confirmed by the implemented and constantly improved Integrated Management System, which covers: quality management (acc. to standard EN ISO/IEC 17025:2005), environmental management (acc. to standard EN ISO 14001:2005), OHS management (acc. to standard EN 18001:2004) and information security management (acc. to standard PN-ISO/IEC 27001:2014-12).

This system covers therefore all areas of company activity, which affect the quality of provided services, and ensures that the quality and environmental requirements of our Clients are met, while Occupational Health and Safety of our employees is compliant with legal requirements and is constantly improved.

Because of the implementation of the latest, modern testing techniques and methods, and as a result of constant investment in the latest laboratory equipment, ORLEN Laboratorium is able to carry out analyses in accordance with Polish, European and American standards.

### 12.2.2. Central Laboratory of ORLEN Laboratorium

Until 2015, at the area of PKN ORLEN in Płock, there were 8 laboratories that served the purposes of production processes taking place in the Main Plant.

During the development of the programme of: *"The construction and development of technological and scientific-research component of the Industrial-Technological Park in Płock for the purposes of interregional innovative activities"* it was decided to centralise laboratory services in the area of the refinery in Płock. As a consequence of investment in the Industrial-Technological Park (PPP-T) in Płock and co-financing from the European Regional Development Fund, the **Central Laboratory** was opened in 2015, becoming one of the most modern laboratory centres in Poland.



Fig. 12.1. Central Laboratory in Płock, external view (photo with permission of PPP-T)

Six ORLEN Laboratorium laboratories, which previously operated in the area of the Płock refinery, were relocated to the Central Laboratory. The idea behind this project was to optimise analytical processes, more effective use of laboratory equipment and logistical improvement through the concentration of analytical activities for different test objects in one location.

The Central Laboratory performs analytical tests which monitor production processes, and carries out product development and implementation works. The complex consists of 45 laboratory rooms, which accommodate over 1100 research stations. The Logistics Department, specialising in the extraction and transport of samples, is also based here.

The Central Laboratory is responsible for the performance of test-related tasks that were previously performed by the transferred laboratories, namely: analysis of fuels, refinery products and petrochemicals, minerals, in-process streams, bituminous binders, waste water, deposits.



The scope of services provided by ORLEN Laboratorium is therefore very extensive, and also includes:

- laboratory services related to the qualification of the quality of tested products,
- laboratory services related to quality control of fuels distributed via petrol stations and storage terminals,
- laboratory services related to the professional extraction of samples of fuel, petroleum products, water, sewage, deposits and other utilities,
- physico-chemical analysis of fuel, biofuel, LPG, engine oil and crude oil,
- testing of bituminous binders,
- testing of used oil,
- testing of biocomponents,
- microbiological analysis of fuel,
- laboratory services related to physico-chemical tests of oil-contaminated soil,
- physico-chemical tests of fertilizers,
- physico-chemical and mechanical tests of plastics,
- corrosion process tests,
- measurements of the concentration and intensity of agents harmful to health at workstations,
- evaluation of professional exposure necessary for the evaluation of professional risks,
- analysis related to environmental protection,
- extraction and analysis of samples taken from observation holes (piezometers),
- monitoring and analysis of water, sewage, deposits and soils,
- services related to technical and technological consultancy.

The opening of this centre was an enormous task for ORLEN Laboratorium, and required the coordination of many areas and involved many months of intensive work. The employees of the company were constantly involved in the designing works related to work station technology.



*Fig. 12.2. Measurement equipment – general view (photo by ORLEN Asphalt Sp. z o.o., with the permission of ORLEN Laboratorium Sp. z o.o.)*

The features that definitely distinguish the Central Laboratory in Płock are:

- very high levels of infrastructural systems,
- fire alarm systems and local chemical warning systems,
- external and internal monitoring systems,
- equipment of laboratory rooms with special laboratory furniture, e.g. automatic fume closets with electrically-raised screens and movement sensor-operated closing system, with large work chambers, safe also with low air flow levels,

- completely controllable and balanced system of air inflow and outflow (VAV), energy-efficient and adequate in terms of efficiency and functionality to the quantity and requirements of ongoing chemical analyses, operated automatically or manually in hazardous situations, i.e. in cases of abnormal levels of concentration of hazardous or flammable substances.

The Central Laboratory of ORLEN Laboratorium is the biggest analytical-chemical centre in Poland, and the sixth biggest laboratory of this type in Europe.

### 12.2.3. Crude Oil Processing Laboratory

The Crude Oil Processing Laboratory, which operates for the purposes of the bitumen production system, is in operation within the structures of ORLEN Laboratorium since 2003. From July 2015 it operates as part of the Refinery Laboratory, which is located in the Central Laboratory.

The main task of this laboratory is the analytical control of ongoing refinery processes, such as: production of fuel, oil, grease, bituminous binders and other products resulting from the processing of crude oil.

The photographs shown below present examples of modern testing equipment used for the analysis of bituminous binders.



*Fig. 12.3. Dynamic Shear Rheometer  
(photo by ORLEN Asphalt Sp. z o.o., with the permission  
of ORLEN Laboratorium Sp. z o.o.)*



*Fig. 12.4. Fluorescent microscope with UV lamp  
for the testing of bitumen microstructure  
(photo by ORLEN Asphalt Sp. z o.o., with the permission  
of ORLEN Laboratorium Sp. z o.o.)*

Laboratory control of the bitumen production process is performed constantly (24/7), and the analyses are carried out according to the developed and approved Analysis Schedule, which is compliant with the requirements of the Factory Production Control. The methodology of testing of bituminous binders includes analyses performed in accordance with standards EN, ASTM and in-house methods. Most of the performed analyses are covered by the scope of AB 484 accreditation. All performed tests involve the use of highly-advanced instrumental analyses,



which allow the laboratory to precisely characterise the physico-chemical and rheological parameters, as well as other conventional techniques. The Crude Oil Processing Laboratory has very extensive and modern measurement facilities, which allow the laboratory to obtain very sensitive and precise results of performed tests.

The Crude Oil Processing Laboratory, which has been testing bituminous binders constantly since 2004, has received, from the national accreditation unit – the Polish Centre of Accreditation, a confirmation of its competency for the performance of tests as defined in the Accreditation No AB 484, and of the compliance of the laboratory with the requirements of reference standard EN ISO/IEC 17025:2005 “Quality Management Systems in Research Laboratories”.

The Research and Development Department of ORLEN Asphalt cooperates with ORLEN Laboratorium from the moment of its inception.

### **12.3. UNIPETROL CENTRE OF RESEARCH AND EDUCATION – UNICRE, (IN CZECH: UNIPETROL VÝZKUMNĚ VZDĚLÁVACÍ CENTRUM, A.S.)**

#### **12.3.1. General information**

The Unipetrol Centre of Research and Education (UniCRE), located in Litvínov in the Czech Republic, in the area of the Česka Rafinerska refinery, is the second analytical-research centre operating within the ORLEN CG.

The chemical laboratory in the Czech Republic was opened in 1952. Initially it operated as a laboratory with the main focus on analyses related to analytical and non-organic chemistry. Currently, the laboratory is also responsible for research activities related to organic chemistry, and concentrates mainly on issues related to the control of petrochemical and refinery processes.

Because of European Union funding between 2010 and 2015, the laboratory has been completely modernised. Currently, UniCRE is the biggest and best equipped research-development centre in the Czech Republic. The main occupation of the laboratory is regular laboratory control of processes ongoing at the production systems. The UniCRE research centre also participates in and coordinates a series of research programmes, and cooperates with many prestigious research units in scientific and educational projects.

Currently, the technical facilities and human resources of the laboratory are available for its partners and clients for cooperation in the following fields:

- production process optimisation
- testing of the synthesis technology of primary and / or special non-organic compounds,
- testing of the technology of fertilizer production,
- neutralisation of solid, liquid and gas waste products
- synthesis of catalysts including the optimisation of catalytic processes,
- technology of crude oil processing – production and quality control of fuel, oil and bitumen.

The UniCRE chemical laboratory has implemented a quality management system that is compliant with the international standard ISO 9001:2008. Positive assessment by independent auditors from the Bureau Veritas Certification has confirmed faultless functioning of the implemented system and the technical competencies of the employees of the Company.

UniCRE research centre offers its clients a wide scope of accredited and in-house methods, which cover chemical, as well as physico-chemical analyses of petroleum, petrochemical products, and others.

### 12.3.2. Bituminous binder research-analytical laboratory

The bituminous binder analytical laboratory was established in 1993. Initially the scope of tests performed in the laboratory covered only conventional analyses related to the evaluation of conformity of produced bitumens, on the basis of Czech and European standards.

In 2006 it began to implement modern testing methods, based on the American *Superpave* system, which enables the performance of functional tests of bituminous binders.

Between 2010 and 2015, the bituminous binder analytical laboratory has undergone complete renovation. Currently it contains modern testing equipment, which enables the laboratory to perform tests of the highest quality.

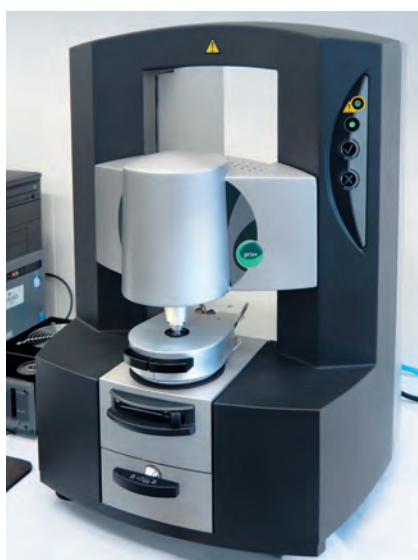


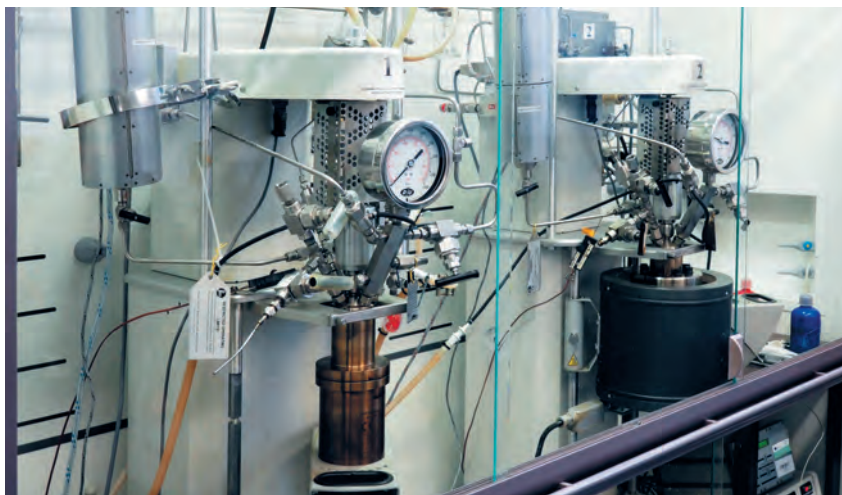
Fig. 12.5. Dynamic Shear Rheometer (photo by ORLEN Asphalt Sp. z o.o. with the permission of UniCRE)



Fig. 12.6. Gas chromatographs, general view (photo by ORLEN Asphalt Sp. z o.o. with the permission of UniCRE)

The analytical control of bitumen production process is performed constantly, in order to guarantee adequate quality of the products. The methodology of bituminous binder tests is based on the requirements of European standards, which serve as the basis for the release of documents that confirm the properties of produced bitumen products, and on the basis of requirements found in American ASTM or AASHTO standards.

The laboratory's areas of interest also include rheological tests of bitumen, the effect of other factors on the final properties of bituminous binders and production optimisation. The laboratory rooms also contain testing systems, which are used for tests related to the use of new polymers, new catalysts in laboratory scale, which after the completion of necessary tests can be then implemented in production scale.



*Fig. 12.7. Testing system for the performance of production trials in laboratory scale, general view (photo by ORLEN Asphalt Sp. z o.o. with the permission of UniCRE)*

The employees of the UniCRE research centre cooperate with many scientific centres in the Czech Republic, participate in product implementation and research-development projects. They are also involved in scientific and educational activities, and publish the results of their work in many prestigious professional magazines.



*Fig. 12.8. Examples of publications by employees of UniCRE Laboratory – posters of publications (photo by ORLEN Asphalt Sp. z o.o. with the permission of UniCRE)*

The Research and Development Department of ORLEN Asphalt cooperates with Unipetrol Centre of Research and Education since 2009.

## 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 No78 (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). WKŁ 2004
- [5] Wymagania Techniczne GDDKiA „Nawierzchnie Asfaltowe na drogach krajowych. WT-2 2014 – część I Mieszanki mineralno-asfaltowe” (GDDKiA Technical Requirements. Bituminous pavements on national roads. WT-2 2014 – part I Mineral-bituminous mixtures)
- [6] CONCAWE: Chemical Safety Report. Part B. "Bitumen" and "Oxidized Asphalt"
- [7] Development in Asphalt Binder Specifications. Transportation Research Circular E-C147. Transportation Research Board, December 2010, ISSN 0097-8515, pp. 39-40
- [8] Encyklopedia fizyki (Encyclopedia of Physics). Państwowe Wydawnictwo Naukowe. 1972
- [9] Gawel I., Kalabińska M., Piłat J. Asfalty drogowe (Paving-grade bitumens). Wydawnictwa Komunikacji i Łączności, edition 2, 2014
- [10] Gust J., Pawłowski K. Systemy oceny zgodności wyrobów budowlanych. Kryteria dobor systemu. (Construction product conformity assessment systems. System selection criteria) Warszawa-Miedzeszyn 2003
- [11] <http://pl.wikipedia.org>
- [12] <http://www.eurobitume.eu/hse>
- [13] [http://www.orlen-asfalt.pl/informacje\\_techiczne.php](http://www.orlen-asfalt.pl/informacje_techiczne.php)
- [14] Kossowicz L., Polskie asfalty naftowe (Polish petroleum-based bitumens), Kraków 1968, Zjednoczenie Przemysłu Rafinerii Nafty
- [15] Habib N.Z., Kamaruddin I., Napiah M., Tan I.M., Effect of thermoplastic copolymers on microstructure and viscoelastic behavior 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
- [16] Physical differentiation between air-rectified and oxidised bitumens. Technical Committee Task Force. Eurobitume, 15.04.2011
- [17] 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.
- [18] Chemical Safety Report by CONCAWE. Part B. "Oxidised bitumen" and "Distilled bitumen"
- [19] Regulation of the Minister of Labour and Social Policy of 06 June 2014 on maximum permitted concentrations and rates of agents harmful to health in work environments
- [20] Schramm G.: "Reologia. Podstawy i zastosowania" (Reology. Fundamentals and applications), Ośrodek Wydawnictw Naukowych, Poznań 1998
- [21] 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
- [22] Szydłowski C., Judycki J., Badania odporności na pękanie mieszanek mineralno-asfaltowych na próbkach półwałcowych (Testing the resistance to cracking of mineral/bituminous mixtures on semi-cylindrical samples), Drogownictwo 10/2015, pp. 348-353.
- [23] 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



- [24] Soenen H., Ekblad J., Lu X., Redelius P., Isothermal Hardening in Bitumen and in Asphalt Mixes, Proceedings of the 3rd Euroasphalt and Eurobitume Congress, Vienna, May, 2004, vol. 2, pp. 1364-1375
- [25] Hesp S.A.M., Genin S.N., Scafe D., Shurvell H.F., Subramani S., Five Years Performance Review of a Northern Ontario Pavement Trial: Validation of Ontario's Double-Edge-Notched Tension (DENT) and Extended Bending Beam Rheometer (BBR) Test Methods, Proceedings of the Canadian Technical Asphalt Association, Vol. 54, 2009, pp. 99-126
- [26] Zhao M.O., Hesp S.A.M., Performance Grading of the Lamont, Alberta C-SHRP Pavement Trial Binders, International Journal of Pavement Engineering, Vol. 7, No. 3, September, 2006, pp. 199-211
- [27] Lu X., Isacsson U., Laboratory Study on the Low Temperature Physical Hardening of Conventional and Polymer Modified Bitumens, Construction and Building Materials, No. 14, 2000, pp. 79-88
- [28] Błażejowski K., Olszacki J., Peciakowski H. The influence of functional binder properties on asphalt mixtures performance, XIV-th Romanian National Congress of Roads and Bridges, 2014
- [29] LS-308 – „Method of test for determination of performance grade of physically aged asphalt cement using extended beam rheometer (BBR) method”
- [30] Baglieria O., Dalmazzoa D., Baraziao M., Tabatabaeeb H.A., Bahia H.A., Influence of Physical Hardening on the Low-Temperature Properties of Bitumen and Asphalt Mixtures, SIIV – 5th International Congress – Sustainability of Road Infrastructures, 2012
- [31] Judycki J. Twardnienie fizyczne asfaltów i mieszanek mineralno-asfaltowych oraz jego wpływ na spękania niskotemperaturowe (Physical Hardening of Bitumen and Mineral/Bituminous Mixtures and Its Effect on Cracking in Low-Temperatures), Drogownictwo 12/2013, pp. 368-373.
- [32] AASHTO TP 70: Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)
- [33] Anderson R. M. (2011), „Understanding the MSCR Test and its Use in the PG Asphalt Binder Specification”, Asphalt Institute
- [34] Kluttz R., Willis R., Molenaar A., Scarpas T., Scholten E., Fatigue Performance of Highly Modified Asphalt Mixtures in Laboratory and Field Environment, 7th RILEM International Conference on Cracking in Pavements, 2012
- [35] Kluttz R. Q., Molenaar A., Van de Ven M. F. C., Poot M.R., Liu X., Scarpas A., Scholten E.J., Modified Base Courses for Reduced Pavement Thickness and Improved Longevity. Proceedings of the International Conference on Perpetual Pavement, October, 2009, Columbus, OH.
- [36] Kluttz R. Q., Jellema E., Woldekidan M.F., Huurman M., Highly Modified Bitumen for Prevention of Winter Damage in OGFCs, Am Soc. Civil E., 2013.
- [37] Timm, D., Robbins M., Kluttz R., Full-Scale Structural Characterization of a Highly Polymer-Modified Asphalt Pavement, Proceedings of the 90th Annual Transportation Research Board, Washington, D.C., 2011.
- [38] Timm D.H., Robbins M.M., Willis J.R., Tran N., Taylor A.J., Field and Laboratory Study of High-Polymer Mixtures at the NCAT Test Track. Draft Report, National Center for Asphalt Technology, Auburn University, 2013.
- [39] Timm D., Powell R., Willis J., Kluttz R., Pavement Rehabilitation Using High Polymer Asphalt Mix, submitted for the Proc. 91st Annual Transp. Res. Board, Washington D.C., 2012.
- [40] West R., Timm D., Willis R., Powell B., Tran N., Watson D., Brown R., Robbins M., Vargas-Nordbeck A., Nelson J., Phase IV NCAT Pavement Test Track Findings, Draft Report, National Center for Asphalt Technology, Auburn University, February 2012.
- [41] Willis J., Timm D., Kluttz R., Taylor A., Tran, N. Laboratory Evaluation of a High Polymer Plant-Produced Mixture, submitted for the Assoc. Asphalt Paving Technology, Annual Meeting, Austin, TX, 2012.
- [42] Paczuski M., Przedlacki M., Lorek A. Technologia produktów naftowych (Technology of Petroleum-Based Products), Oficyna wydawnicza Politechniki Warszawskiej, Warszawa 2015
- [43] Cortioza M.S., Larsenb D.O., Bianchetob H., Alessandrini J.L., Effect of the thermal degradation of SBS copolymers during the ageing of modified asphalts, Polymer Degradation and Stability, Volume 86, Issue 2, November 2004, pp. 275-282

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Company department at ORLEN Asphalt. Active from the company's foundation in 2003. The R&D performs research and development work related to bituminous binders, asphalt mixtures, technical marketing and new product development. It offers technical consultancy to customers related to the application of bituminous binders manufactured by the company.

The R&D Department achievements include patent applications, gold medal received at the International Warsaw Invention Exhibition IWIS 2007 for BITREX multigrade bitumens, and the prize awarded by the Polish Minister of Science and Higher Education for achievements in the area of inventions (2007). In 2016 ORLEN Asphalt received the Leader of Innovation prize in the Diamonds of Polish Infrastructure competition for highly-modified ORBITON HiMA bitumen, developed by the Department of Research and Development.

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