

2021 BITUMEN HANDBOOK

Bitumen Handbook 2021
ORLEN Asphalt sp. z o.o., Poland

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Bitumen Handbook 2021 contains extracts from the texts and diagrams that constitute the content of 2009 to 2018 editions.

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LETTER FROM THE MANAGEMENT BOARD

DEAR READERS!

It is with great satisfaction that we present you with the seventh edition of Bitumen Handbook, the only publication of its kind in Poland, which is a structured collection of knowledge on bituminous binders and information on their effective application.

The publication comprehensively describes the properties of bitumens, their functionality and many issues related to their production, logistics or use in the production of asphalt mixtures. The knowledge contained in the Handbook can therefore be useful at any stage of road construction – from the design phase, through research and development, to execution.

The authors of Bitumen Handbook explain the basic issues related to the use of bitumens, as well as more complex issues concerning the testing of their properties. Therefore the Handbook constitutes a valuable resource for both beginners and experts in the field of bituminous binders.



Marek Pietrzak
President of the Management Board

ORLEN Asphalt Management Board



Alan Steinbarth
Member of the Management Board

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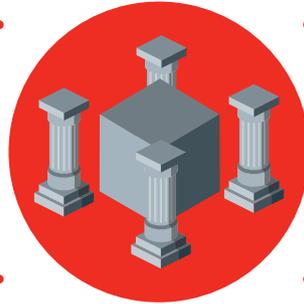
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ABOUT ORLEN ASFALT



ORLEN Asfalt – General Information

The company is a part of the ORLEN Group and one of the leading suppliers of bituminous binder used for paving the roads in Poland, Central and Eastern Europe. Since October 2012, ORLEN Asfalt is the owner of the Czech company ORLEN Asfalt Česká republika which sells bitumen produced in Litvinov and Pardubice. In 2019, ORLEN Group sold 1.56 million tons of bitumen, which is the best result in the history of the company's activity on the market. Almost half of these sales were achieved by ORLEN Asfalt.

We are a leader in implementing innovative bitumen technologies. In 2014, ORLEN Asfalt was the first company to introduce to the Polish market a group of products highly modified bitumen under the trade name ORBITON HiMA. Today, it is one of the fastest-growing directions of road pavement technology. Our research confirms that in terms of functional properties these are the best products for use on heavy traffic roads.



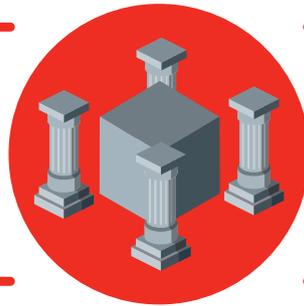
PRODUCTS

As part of ORLEN Group's bitumen segment, we supply products from five production centres located in Poland (Płock, Trzebinia), Czech Republic (Pardubice, Litvinov) and Lithuania (Mažeikiai).

| PRODUCTS | | |
|---------------------------|--------------------------------------|-----------------------|
| PŁOCK, TRZEBINIA (POLAND) | PARDUBICE, LITVÍNŮV (CZECH REPUBLIC) | MAŽEIKIAI (LITHUANIA) |
| PAVING GRADE BITUMEN | PAVING GRADE BITUMEN | PAVING GRADE BITUMEN |
| POLYMER MODIFIED BITUMEN | HARD PAVING GRADE BITUMEN | V6000 BITUMEN |
| HIGHLY MODIFIED BITUMEN | MULTIGRADE BITUMEN | V12000 BITUMEN |
| INDUSTRIAL BITUMEN | POLYMER MODIFIED BITUMEN | |
| | HIGHLY MODIFIED BITUMEN | |
| | INDUSTRIAL BITUMEN | |
| | OXIDISED BITUMEN | |
| | BITUMEN FOR RECYCLING | |

1. BITUMINOUS BINDERS – REQUIREMENTS AND APPLICATION

1.1. HISTORY OF BITUMEN



Bitumen as a material has been known since ancient times [1]. At first, it was extracted from rock deposits in a form of a thick, dark liquid. Natural bitumen can occur as bitumen-saturated rocks as well as pitch lakes, asphaltites, bituminous sands or pyrobitumen [2]. The first use of natural bitumen dates back to around 3800 BC. It was used by the Sumerians to seal boats made of reeds. As a binder for bonding stone blocks of floors and walls, it was then used for the first time near Baghdad around the 30th century BC [2]. The first documented use of bitumen in road construction is attributed to the constructors of the Processional Road in Babylon during the reign of King Nabopolassar around 615 BC. According to discovered inscriptions, the road was made of burnt bricks bonded with bitumen [3]. Egyptians used the preservative properties of bitumen for the mummification of corpses. In the Roman Empire, it was used to seal pools, reservoirs and aqueducts. Many centuries later, in 1595, Sir Walther Raleigh, sailing to the New World, discovered a natural pitch lake on the island of Trinidad, off the coast of Venezuela. Using its resources, he was able to seal ships damaged during navigation.

Despite its long history of use, bituminous binders became widespread as a road construction material in the 19th century. The first bituminous pavement was placed in Paris on the Place de la Concorde in 1837 and 21 years later, also in Paris, another one, made of crushed compacted bitumen rocks [2]. In the USA, the first bituminous pavement was constructed in 1870 by the Belgian chemist Edmund J. DeSmedt. Following the model of the pavement made in Paris, he laid a course of a bituminous mixture in Newark, New Jersey.

The late 19th century was crucial for the development of bituminous pavements. Firstly, the technology of processing crude oil was developed. Secondly, pneumatic tyres – invented by John Dunlop in 1888 – were becoming increasingly popular, forcing road network managers to build better surfaces that were even and reduced dust behind ever faster-moving vehicles. This led to a real breakthrough in the development of pavements made with petroleum bitumen. In Poland and Europe, Ignacy Łukasiewicz is regarded as a pioneer in the field of petroleum distillation. He presented the obtained petroleum products – including petroleum bitumen – for the first time at the exhibition in Jasło in 1858 [2]. In the USA, industrial production of bitumen obtained from oxidation of residues from the distillation of crude oil started in 1894 [2]. Already in 1902, the production of bituminous binders in the USA reached the level of about 20 thousand tons. In Poland, asphalt pavements made with petroleum bitumen started to be used after World War I. However, asphalt pavements were already known in Poland in the first half of the 19th century. Stanisław Wysocki had been trying to use tar mixtures (gravel mixed with tar) since 1837 and as early as in 1838, he successfully used this material for pavements and roofing in Warsaw (e.g. the pavement at Bank Square and around the Royal Castle, the covering of the porch of St. John's Cathedral). The first tar factory in Poland, where binders were produced using vegetable tar, marls and lime, was opened by him in Augustów in 1838 [12].

Petroleum bitumen for road construction at the beginning of the 20th century came mainly from the Polmin refinery in Drohobych [2]. In the interwar period, several thousand tons of binders

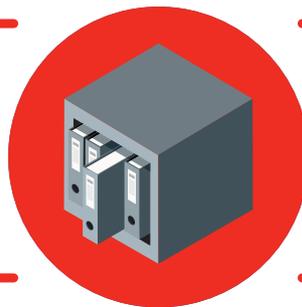
were produced in Poland annually, which now corresponds to the average annual demand of three asphalt mix plants, each of which produces about 100,000 tons of asphalt mixes.

Since its invention, crude oil processing technology and bitumen production technology have been greatly improved. A significant step in the development of road pavement technology was the start of modifying bitumen with natural and synthetic polymers. In the 1980s, the SBS-type polymer was produced for the first time [13]. The use of polymer modified binders on a larger scale in Europe and the USA started in the 1980s

[14]. In Poland, the first use of modified binders in a refinery took place in the first half of the 1990s.

Currently, the bitumen market in Poland exceeds 1 million tons of binders annually, reaching even 1.5 million in the years of peak demand. At the same time, this means that more than 20 million tons of asphalt mixtures are produced in Poland annually, and in the peak years of road works even up to 30 million tonnes. About 20–25% of all binders sold in the country are polymer modified bitumens.

1.2. REFERENCE DOCUMENTS AND REQUIREMENTS



Bituminous binders used in road pavements are construction products, the marketing and production of which is governed by Regulation (EU) No 305/2011 of the European Parliament and of the Council of 9 March 2011, and in Poland by the Construction Products Act and a number of implementing regulations.

The system of requirements related to construction products does not apply to oxidised bitumen which is usually used as a raw material for the production of other materials (sealants, roofing paper, etc.). In the case of binders for paving applications, requirements have been introduced for each type of bitumen that must be met in order for the product to be placed on the market.

The conformity of the properties of paving grade binders with the requirements of the relevant standard and values specified therein (including grades) should be demonstrated by:

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

The bitumen standards oblige 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. Chapter ZA of every standard contains requirements related to the verification and maintenance 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, be documented and ensure that the properties remain essentially similar to those defined during the initial type tests.

Binders used for paving roads, airports and other pavements which carry traffic are covered by the conformity assessment system “2+”, which requires every manufacturer to implement

the Factory Production Control system certified by an FPC Certificate issued by a notified body. Annex ZA also includes the procedure for product conformity assessment, division of responsibilities between the manufacturer and the notified body, a chapter on the certification and a declaration of performance, CE marking and labelling.

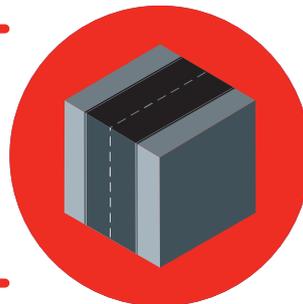
Table 1.1. shows currently produced bituminous binders with reference documents in force. Product groups offered by ORLEN Asphalt are marked in bold; they are described in the following sections with regard to properties, requirements and recommended application.

Table 1.1.

Attribution of European Standards to various types of binders and numbers of FPC Certificates for production sites within ORLEN Group

| TYPE OF BITUMINOUS BINDER | REFERENCE DOCUMENT | NO. OF FPC CERTIFICATE |
|---|--------------------------|--|
| Paving grade bitumen | EN 12591 | 1434-CPR-0183 in Płock 1434-CPR-0185 in Trzebinia 1020-CPR-090030197 in Pardubice 1020-CPR-090031819 in Litvinov 1567-CPD-0064 in Mažeikiai |
| Hard paving grade bitumen Multigrade bitumen | EN 13924-1 EN 13924-2 | 1020-CPR-090033991 14 0300SV/ITC/a in Pardubice |
| Polymer modified bitumen Highly modified bitumen | EN 14023 | 1434-CPR-0184 in Płock 1434-CPR-0186 in Trzebinia 1023-CPR-1055F in Pardubice |
| Fluxed and cut-back bitumen | EN 15322 | – |
| Cationic bituminous emulsions | EN 13808 | 1023-CPR-0629F in Pardubice |
| Oxidised (industrial) bitumen | EN 13304 | Not applicable |
| Hard grade industrial bitumen | EN 13305 | Not applicable |

1.3. PAVING GRADE BITUMEN



Paving grade bitumens offered by ORLEN Asphalt are produced in accordance with standard EN 12591:2009

Standard EN 12591:2009 *Bitumen and bituminous binders. Specifications for paving grade bitumen* are a partially classification standard, i.e. most of the requirements 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 maintenance of roads, airports and other road traffic-bearing pavements. It also includes all requirements regarding the assessment of conformity

1.3.1. CLASSIFICATION OF PAVING GRADE BITUMEN

Paving grade bitumen, manufactured based on the requirements of standard EN 12591:2009, are marked according to classification provided in table 1.2

Table 1.2.

Classification of paving grade bitumen manufactured acc. to EN 12591

| TYPE OF BITUMINOUS BINDER | PAVING GRADE BITUMEN |
|---|---|
| Reference document | EN 12591:2009 |
| Standard designation of bituminous binder | XX/YY |
| Type of bituminous binder supplied by ORLEN Group | 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]

1.3.2. REQUIREMENTS FOR PAVING GRADE BITUMEN

Table 1.3. shows general requirements regarding paving grade bitumen, according to information provided in standard EN 12591:2009.

Table 1.3.

Requirements regarding all types of paving grade bitumen of the penetration range from 20 × 0.1 to 220 × 0.1 mm, according to the Polish National Annex to standard EN 12591:2009

| | PROPERTY | TEST METHOD | UNIT | PAVING GRADE BITUMEN TYPE | | | | | |
|---|-------------------------------------|------------------------------|---------|---------------------------|--------|--------|--------|---------|---------|
| | | | | 20/30 | 35/50 | 50/70 | 70/100 | 100/150 | 160/220 |
| Properties applying to all paving grade bitumen listed in this table | 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 | | | – | – | – | – | – | – |
| | Retained penetration | EN 12607-1 (RTFOT method) | % | ≥ 55 | ≥ 53 | ≥ 50 | ≥ 46 | ≥ 43 | ≥ 37 |
| | Increase in Softening point | | °C | ≤ 8 | ≤ 8 | ≤ 9 | ≤ 9 | ≤ 10 | ≤ 11 |
| | Change of mass* (absolute value) | | % | ≤ 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 |

* Change of mass may be a positive or negative value

Table 1.3. (continued)

Requirements regarding all types of paving grade bitumen of the penetration range from 20×0.1 to 220×0.1 mm, according to the Polish National Annex to standard EN 12591:2009

| PROPERTY | TEST METHOD | UNIT | PAVING GRADE BITUMEN TYPE | | | | | | |
|---|------------------------------|------------------|---------------------------|-------|-------|--------|---------|---------|-------|
| | | | 20/30 | 35/50 | 50/70 | 70/100 | 100/150 | 160/220 | |
| Properties adapted to country-specific conditions | 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 ² /s | NR | NR | NR | NR | NR | NR |

NR – (No Requirement) – indicates that there are no requirements for a specific property

Work is currently underway to update the EN 12591 standard. The draft standard prEN 12591 prepared by CEN was released in 2017. Like the current version of the standard, it contains rules for the determination of properties and relevant test methods for paving grade bitumen. Additionally, it provides information on the System of Assessment and Verification of Constancy of Performance (AVCP). The draft also takes into account changes necessary to comply with the Construction Products Regulation (CPR).

The most significant changes in relation to the current version of the standard are introduced in Annex B, where new test methods for paving grade bitumen are indicated, i.e. bending beam rheometer (BBR) and dynamic shear rheometer (DSR) tests. The tests will be used in the future to assess the performance of paving grade bitumen. Presently, these are "Informative Properties", which the binder supplier should make available as a single value RV (Reported Value). The publication date of the new edition of the standard is currently unknown.

1.3.3. DESCRIPTION OF PAVING GRADE BITUMEN

Paving grade bitumens are the most popular binders for hot mix asphalts used for road pavement construction.

All paving grade bitumens offered by ORLEN Asphalt are classified as paving grade bitumens with the penetration range of 20–220 [0.1 mm], tested at 25°C.

Fig. 1.1. presents a schematic comparison of primary bitumen properties for the two most popular bituminous binder parameters, namely penetration at 25°C and softening point $T_{R\&B}$.

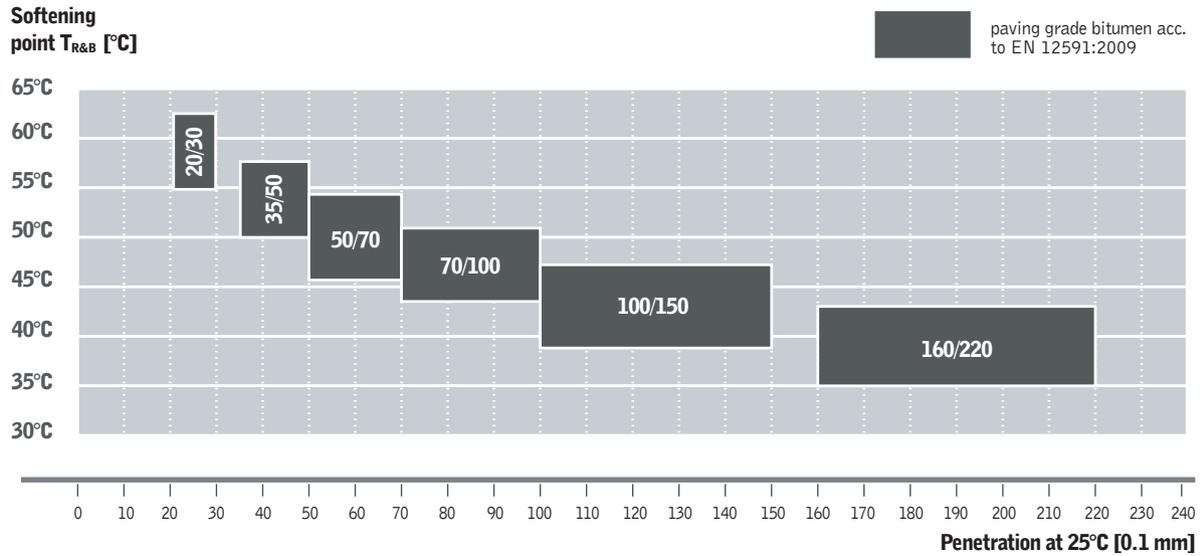


Fig. 1.1. Schematic comparison of paving grade bitumen types acc. to EN 12591:2009 as a function of penetration at 25°C and softening point T_{R&B}

1.3.4. APPLICATION OF PAVING GRADE BITUMEN

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 offered by ORLEN Group. Its high softening point and high sensitivity to low-temperature cracks renders it recommendable solely for the binder and base courses of high-stiffness modulus asphalt concrete (AC EME) in regions with suitable climate. Courses with bitumen 20/30 should not be left over the winter period without being covered with the next course. Additionally, for main roads, in WT-2 part I of 2014 [6] are given special requirements and climate zones where specific types of binders (paving grade 20/30 or polymer modified) are used in AC EME, Fig. 1.2

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. The 35/50 bitumen should not be used in wearing courses made of asphalt concrete (AC) or stone mastic asphalt (SMA).



Fig. 1.2. Map of Poland – climatic zones for the use of bitumen 20/30 in high-stiffness modulus asphalt mixtures AC EME – zones I and II only [6]

Paving grade bitumen 50/70 can be used primarily for asphalt concrete (AC) and stone mastic asphalt (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 heavy, 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 (AC) and stone mastic asphalt (SMA) in wearing courses on roads designed for light traffic, on the assumption that the mix resistance to rutting is confirmed.

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.

Table 1.4. summarises the recommendations for the use of paving grade bitumen for road construction in Poland

Table 1.4.

Recommended application of paving grade bitumens from ORLEN Asphalt depending on the road surface course and traffic load category

| COURSE | TRAFFIC CATEGORY*** | | |
|---------|------------------------|----------------------|---------------|
| | KR 1-2 | KR 3-4 | KR 5-7 |
| Base | 50/70 | 20/30*, 35/50, 50/70 | 20/30*, 35/50 |
| Binder | 50/70 | 20/30*, 35/50, 50/70 | 20/30*, 35/50 |
| Wearing | 35/50**, 50/70, 70/100 | 35/50**, 50/70 | – |

* for AC EME only

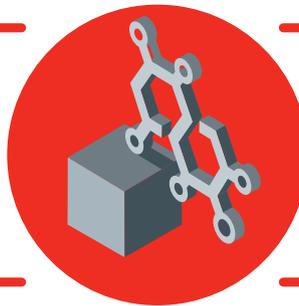
** for mastic asphalt MA only

*** Traffic categories (KR) are specified in million axles 100 kN carried in service life, single tire is used as standard. KR1-2 (below 0.5), KR3-4 (0.5 - 7.3), KR 5-7 (7.3 - 52+)

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

apparatus, in the air, temperature +60°C, 10 000 cycles). The above refers especially to road sections located within crossroad zones, slow traffic zones, parking zones, etc.

1.4. POLYMER MODIFIED BITUMEN



The properties of bitumen can be improved by the introduction of various additives. ORLEN Asphalt offers binders modified with SBS elastomer (styrene-butadiene-styrene) which widens the temperature range in which the binder retains its viscoelastic properties. The production of this type of binder is based on a physical method involving mechanical mixing of the bitumen with the polymer, with the possible use of various additives. The primary feedstock for modified bitumen production comprises special bitumens, so-called base bitumens, of properties adequate for modification. The production of polymer modified bitumen involves the introduction of the polymer into the hot bitumen, the grinding of the mixture in a high shear mill and its final dissolution and homogenisation.

The polymer modified binders offered by ORLEN Asphalt are produced in accordance with the EN 14023:2010 standard at one of the three production facilities – in Płock, Trzebinia (Poland) and Pardubice (Czech Republic).

Standard EN 14023:2010 *Bitumen and bituminous binders. Specification framework for polymer modified bitumen* is a classification standard. It means that it provides a set of properties and assigns to them various requirement levels (classes). It assumes that each Member State of CEN selects the properties and the related requirement levels. This enables each Member State to specify its own requirements to be met by bituminous binders used on its territory. The differentiation results from diverse climate conditions in various parts

of Europe, different permissible vehicle axle loads and many other technological factors.

EN 14023:2010 standard contains a set of basic and additional properties, which are contained in three separate tables:

TABLE 1

properties required for polymer modified bitumen (In the National Annex Table 1 is divided into two tables: NA.1 – Polymer modified bitumen requirements, NA.2 – Highly polymer modified bitumen requirements)

TABLE 2

properties related to legal regulations or other country-specific conditions

TABLE 3

additional properties

The standard also includes all requirements regarding the assessment of conformity. CEN is currently working on an amended revision of EN 14023 – it is discussed in detail in chapter 7.

1.4.1. CLASSIFICATION OF POLYMER MODIFIED BITUMEN

Modified bitumen manufactured according to the requirements of standard EN 14023:2010 are marked according to classification provided in table 1.5.

Table 1.5.

Classification of polymer modified bitumen manufactured acc. to EN 14023

| TYPE OF BITUMINOUS BINDER | MODIFIED BITUMEN |
|---|---|
| Reference document | PN-EN 14023:2011/Ap2:2020 |
| Standard designation of bituminous binder | PMB X/Y-Z |
| Type of bituminous binder supplied by ORLEN Group | ORBITON 10/40-65 ORBITON 25/55-60 ORBITON 45/80-55 ORBITON 45/80-65 ORBITON 65/105-60 |

Notes to designations:

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] acc. to EN 1427.

PMB – abbreviation stands for Polymer Modified Bitumen (usually replaced by the manufacturer's trade name)

This Handbook contains a detailed description of polymer modified bitumens ORBITON manufactured for the application in the road engineering in Poland. ORLEN Asphalt also offers polymer modified bitumens ORBITON adapted to local requirements of countries to which these PMB are exported (e.g. Romania, Lithuania, Latvia, Czech Republic, Slovakia, Germany, Hungary). In Table 1.6, they are marked as EXP.

The types of PMB produced by ORLEN Group are presented in Table 1.6.

Table 1.6.

Types of modified bitumen supplied by ORLEN Group

| TYPES OF ORBITON MODIFIED BITUMEN ACC. TO ANNEX NA FOR POLAND | TYPES OF ORBITON MODIFIED BITUMEN FOR OTHER EU STATES |
|---|---|
| 10/40-65 25/55-60 45/80-55 45/80-65 65/105-60 | 25/55-55 EXP 25/55-60 EXP 25/55-65 EXP 45/80-75 SK |

1.4.2. REQUIREMENTS FOR POLYMER MODIFIED BITUMEN

The National Annex to standard EN 14023:2010 published with an amendment Ap2:2020-02 in 2020 is a set of properties and requirement levels for these properties for six polymer modified bitumens used in Poland. The requirements given in the Polish National Annex have been established by the Subcommittee

for Bitumen of Technical Committee 222 of Polish Committee for Standardization.

A division by type and requirements for polymer modified bitumen is shown in Table 1.7.

Table 1.7.

Division based on types and requirements for polymer modified bitumen in Poland acc. to National Annex NA to standard PN-EN 14023:2011/Ap2:2020-02

| PROPERTY | TEST METHOD | UNIT | POLYMER MODIFIED BITUMEN TYPE | | | | | | | |
|---|--|---|-------------------------------|-------------------|--------------|-------|--------------|-------|-----------|---|
| | | | PMB 10/40-65 | | PMB 25/55-60 | | PMB 45/80-55 | | | |
| | | | RANGE | CLASS | RANGE | CLASS | RANGE | CLASS | | |
| Basic properties | Penetration at 25°C | EN 1426 | 0.1 mm | 10-40 | 2 | 25-55 | 3 | 45-80 | 4 | |
| | Softening point | EN 1427 | °C | ≥65 | 5 | ≥60 | 6 | ≥55 | 7 | |
| | Cohesion** | Force ductility tested using ductilometer (50 mm/min) | EN 13589 EN 13703 | J/cm ² | ≥2 at 10°C | 6 | ≥2 at 10°C | 6 | ≥3 at 5°C | 2 |
| | | Tensile test at 5°C (100 mm/min) | EN 13587 | J/cm ² | NR | 0 | NR | 0 | NR | 0 |
| | | Vialit pendulum (impact test) | EN 13588 | J/cm ² | NR | 0 | NR | 0 | NR | 0 |
| | Change of mass after hardening* | EN 12607-1 | % m/m | ≤0.5 | 3 | ≤0.5 | 3 | ≤0.5 | 3 | |
| | Retained penetration at 25°C after hardening | EN 12607-1 EN 1426 | % | ≥60 | 7 | ≥60 | 7 | ≥60 | 7 | |
| | Softening point increase after hardening | EN 12607-1 EN 1427 | °C | ≤8 | 2 | ≤8 | 2 | ≤8 | 2 | |
| | Flash point | EN ISO 2592 | °C | ≥235 | 3 | ≥235 | 3 | ≥235 | 3 | |
| | Additional properties | Fraass breaking point | EN 12593 | °C | ≤-5 | 3 | ≤-10 | 5 | ≤-15 | 7 |
| Elastic recovery at 25°C | | EN 13398 | % | ≥60 | 4 | ≥60 | 4 | ≥70 | 3 | |
| Elastic recovery at 10°C | | EN 13398 | % | NR | 0 | NR | 0 | NR | 0 | |
| Plasticity range | | EN 14023 | °C | NR | 0 | NR | 0 | NR | 0 | |
| Drop in softening point after EN 12607-1 | | EN 12607-1 EN 1427 | °C | TBR | 1 | TBR | 1 | TBR | 1 | |
| Elastic recovery at 25°C after EN 12607-1 | | EN 12607-1 EN 13398 | % | ≥50 | 4 | ≥50 | 4 | ≥50 | 4 | |
| Elastic recovery at 10°C after EN 12607-1 | | EN 12607-1 EN 13398 | % | NR | 0 | NR | 0 | NR | 0 | |
| Storage stability – difference in softening point | | EN 13399 EN 1427 | °C | ≤5 | 2 | ≤5 | 2 | ≤5 | 2 | |
| Storage stability – difference in penetration | | EN 13399 EN 1426 | 0.1 mm | NR | 0 | NR | 0 | NR | 0 | |

* – change of mass may be a positive or negative value

** – depending on the final use, only one method for determining cohesion should be chosen. The cohesion determination by the Vialit method (EN 13588) should only be chosen for bitumen for surface treatment

NR – No Requirement

TBR – To Be Reported

Table 1.7. (continued)

Division based on types and requirements for polymer modified bitumen in Poland acc. to National Annex NA to standard PN-EN 14023:2011/Ap2:2020-02

| PROPERTY | TEST METHOD | UNIT | POLYMER MODIFIED BITUMEN TYPE | | | | | | |
|--|---|------------------------|-------------------------------|------------|---------------|-----------|---------------|-------|---|
| | | | PMB 45/80-65 | | PMB 65/105-60 | | PMB 90/150-45 | | |
| | | | RANGE | CLASS | RANGE | CLASS | RANGE | CLASS | |
| Penetration at 25°C | EN 1426 | 0.1 mm | 45-80 | 4 | 65-105 | 6 | 90-150 | 8 | |
| Softening point | EN 1427 | °C | ≥65 | 5 | ≥60 | 6 | ≥45 | 9 | |
| Basic properties Cohesion** | Force ductility tested using ductilometer (50 mm/min) | EN 13589 EN 13703 | J/cm ² | ≥2 at 10°C | 6 | ≥3 at 5°C | 2 | NR | 0 |
| | Tensile test at 5°C (100 mm/min) | EN 13587 | J/cm ² | NR | 0 | NR | 0 | NR | 0 |
| | Vialit pendulum (impact test) | EN 13588 | J/cm ² | NR | 0 | NR | 0 | ≥0,7 | 2 |
| Change of mass after hardening* | EN 12607-1 | % m/m | ≤0.5 | 3 | ≤0.5 | 3 | ≤0.5 | 3 | |
| Retained penetration at 25°C after hardening | EN 12607-1 EN 1426 | % | ≥60 | 7 | ≥60 | 7 | ≥50 | 5 | |
| Softening point increase after hardening | EN 12607-1 EN 1427 | °C | ≤8 | 2 | ≤10 | 3 | ≤10 | 3 | |
| Flash point | EN ISO 2592 | °C | ≥235 | 3 | ≥235 | 3 | ≥235 | 3 | |
| Additional properties | Fraass breaking point | EN 12593 | °C | ≤-15 | 7 | ≤-15 | 7 | ≤-18 | 8 |
| | Elastic recovery at 25°C | EN 13398 | % | ≥80 | 2 | ≥70 | 3 | ≥50 | 5 |
| | Elastic recovery at 10°C | EN 13398 | % | NR | 0 | NR | 0 | NR | 0 |
| | Plasticity range | EN 14023 | °C | NR | 0 | NR | 0 | NR | 0 |
| | Drop in softening point after EN 12607-1 | EN 12607-1 EN 1427 | °C | TBR | 1 | TBR | 1 | TBR | 1 |
| | Elastic recovery at 25°C after EN 12607-1 | EN 12607-1 EN 13398 | % | ≥60 | 3 | ≥60 | 3 | ≥50 | 4 |
| | Elastic recovery at 10°C after EN 12607-1 | EN 12607-1 EN 13398 | % | NR | 0 | NR | 0 | NR | 0 |
| | Storage stability – difference in softening point | EN 13399 EN 1427 | °C | ≤5 | 2 | ≤5 | 2 | ≤5 | 2 |
| | Storage stability – difference in penetration | EN 13399 EN 1426 | 0.1 mm | NR | 0 | NR | 0 | NR | 0 |

* – change of mass may be a positive or negative value

** – depending on the final use, only one method for determining cohesion should be chosen. The cohesion determination by the Vialit method (EN 13588) should only be chosen for bitumen for surface treatment

NR – No Requirement

TBR – To Be Reported

1.4.3. DESCRIPTION OF POLYMER MODIFIED BITUMEN

Polymer modified bitumens represent a group of binders for paving application designed specifically to improve the durability of asphalt pavements and to counteract the most frequent road problems, such as plastic deformations of pavements carrying heavy traffic, low-temperature cracking of wearing courses during winter, and fatigue-related cracks. The application of the SBS elastomer (Styrene-Butadiene-Styrene) 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 classical 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 $T_{R\&B}$, are schematically shown in Figure 1.3.

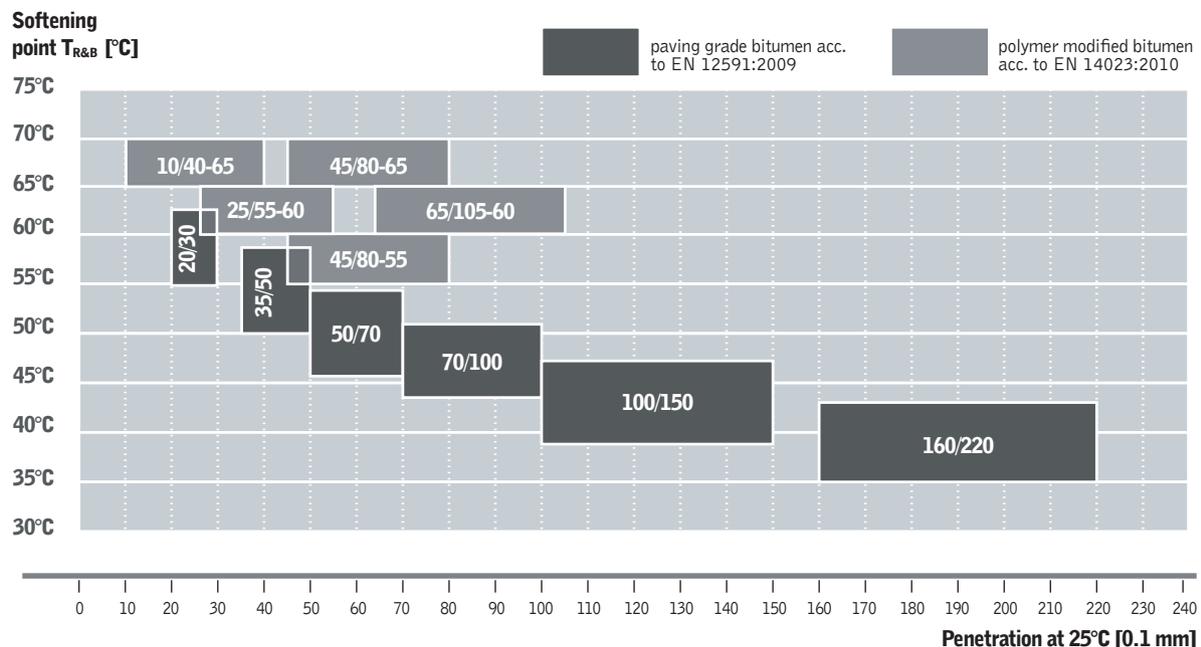


Fig. 1.3. Schematic comparison of paving grade and polymer modified bitumen as a function of penetration at 25°C and softening point $T_{R\&B}$

1.4.4. APPLICATION OF POLYMER MODIFIED BITUMEN

ORBITON polymer 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 much better properties than their paving grade counterparts of similar hardness.

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

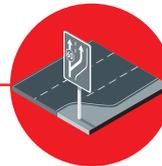
ORBITON 10/40-65 modified bitumen modified bitumen is the hardest modified bitumen in the range of modified bitumens currently manufactured by ORLEN Group. Its very high softening point renders it suitable for high-stiffness-modulus mixture AC EME base and binder courses. It can also be used for conventional asphalt concrete mixtures. 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 parking lots, slow traffic lanes and crossroads. This type of bitumen is not recommended for use in wearing courses.

suitable for

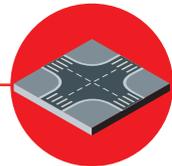
pavements carrying slow and heavy traffic



parking lots



slow traffic lanes

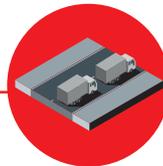


crossroads zones

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-stiffness modulus asphalt concrete AC EME¹ (at the required stiffness modulus of 14 000 MPa). It can also be used in wearing, binder and base courses made of SMA at sections carrying slow and heavy traffic and in mastic asphalt MA.

suitable for

asphalt concrete base and binder courses



sections carrying slow and heavy traffic

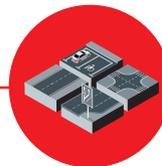


in mastic asphalt MA

ORBITON 45/80-55 modified bitumen is one of the most popular modified bitumen types. It was designed for application in all types of asphalt mixtures for wearing courses: AC, SMA, PA, BBTM, AUTL.

suitable for

all types of asphalt mixtures for wearing courses



AC, SMA, PA, BBTM, AUTL

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 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.

suitable for

locations where high tensile strength and fatigue resistance are necessary



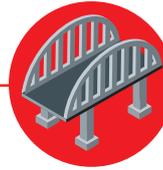
wearing courses, including porous asphalt PA

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

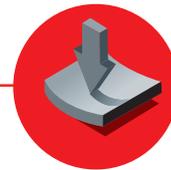
ORBITON 65/105-60 modified bitumen is designed for use in hot mix wearing courses, within good mineral skeleton mixtures. It is produced using soft base bitumen with a high polymer content, which allows to obtain a product with excellent low-temperature properties and elasticity. ORBITON 65/105-60 is characterised by higher penetration at 25°C in comparison to modified bitumen 45/80-65, and at the same time demonstrates high levels of cohesion and elasticity. The combination of those features renders the product a very good binder for thin-layered mixtures of non-continuous granulation. Such applications include PA, BBTM and AUTL mixes for thin wearing courses and SMA. Those are primarily special wearing courses and wearing courses used at low-temperature locations. This binder can also be used in bridge deck mixtures, whenever excellent elasticity of the binder is required.

suitable for

special wearing courses and courses used at low-temperature locations



on bridge decks



high elasticity and cohesion of the binder

Figure 1.4 shows application examples of ORBITON polymer modified bitumen for particular pavement courses.

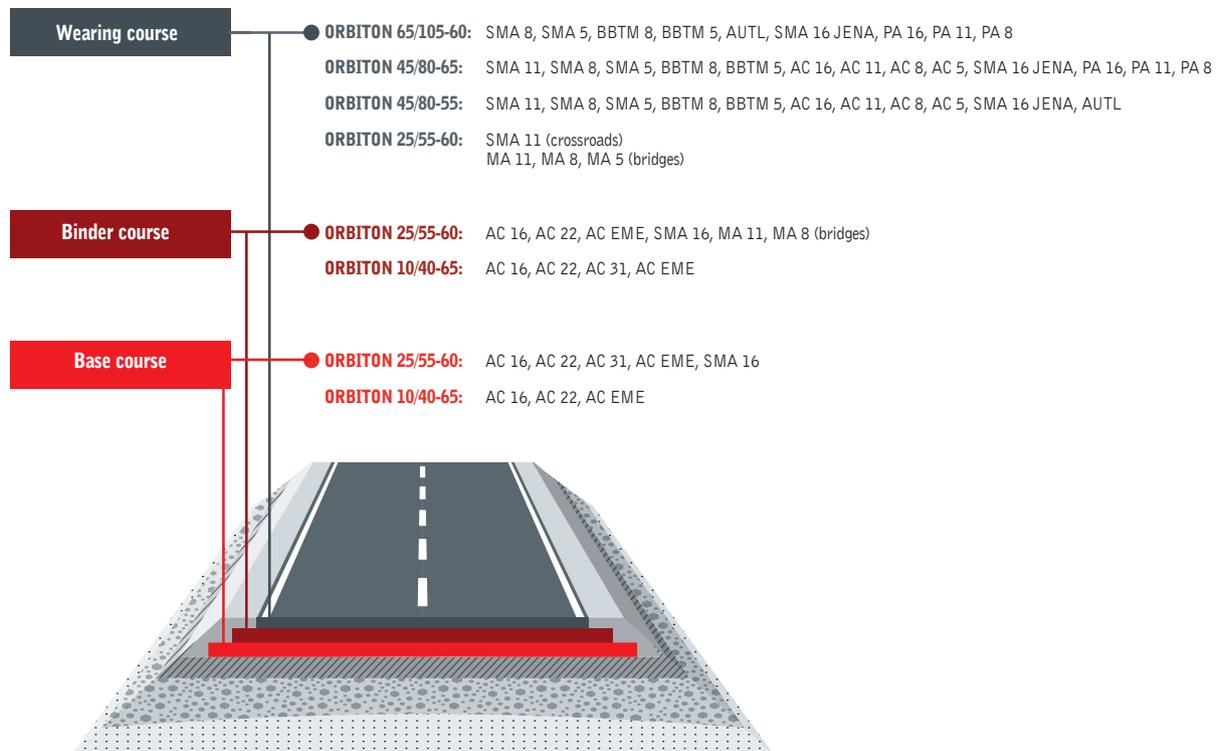


Fig. 1.4. Proposed use of ORBITON modified bitumen for conventional flexible pavement construction [own elaboration]

Additionally, based on experience, Tables 1.8 and 1.9 present recommendations for the use of polymer modified bitumen for the construction of road and bridge pavements in Poland.

Table 1.8.

Recommended application of polymer modified bitumen from ORLEN Asphalt depending on the **road surface course** and traffic load category

| COURSE | TRAFFIC CATEGORY* | | |
|---------|---|---|---|
| | KR 1-2 | KR 3-4 | KR 5-7 |
| Base | – | ORBITON 10/40-65 ORBITON 25/55-60 | ORBITON 10/40-65 ORBITON 25/55-60 |
| Binder | ORBITON 45/80-55 | ORBITON 10/40-65 ORBITON 25/55-60 | ORBITON 10/40-65 ORBITON 25/55-60 |
| Wearing | ORBITON 45/80-55 ORBITON 45/80-65 ORBITON 65/105-60 | ORBITON 25/55-60 ORBITON 45/80-55 ORBITON 45/80-65 ORBITON 65/105-60 | ORBITON 45/80-55 ORBITON 45/80-65 ORBITON 65/105-60 |

* Description in table 1.4

Table 1.9.

Recommended application of polymer modified bitumen from ORLEN Asphalt depending on the **bridge pavement course**

| COURSE | BINDERS |
|---------|--|
| Binder | ORBITON 25/55-60 |
| Wearing | ORBITON 25/55-60* ORBITON 45/80-55 ORBITON 45/80-65 ORBITON 65/105-60 |

* for mastic asphalt MA

1.5. HIGHLY POLYMER MODIFIED BITUMEN



The main idea of highly modified bitumens HiMA (am. Highly Modified Asphalt) is to prevent pavement cracking, permanent deformation (rutting), and to increase fatigue life of asphalt courses. 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. In structural terms, HiMA courses retain very high tolerance for an increased tensile deformation (so-called fatigue).

Highly modified bitumens are also particularly suitable for long-service-life pavements such as perpetual pavements. Application of ORBITON HiMA in the special anti-fatigue bottom layer allows to achieve a very long pavement life cycle.

Highly modified bitumens are classified according to European Standard EN 14023:2010 *Bitumen and bituminous binders. Specification framework for polymer modified bitumen.*

1.5.1. CLASSIFICATION OF HIGHLY MODIFIED BITUMEN

Classification of highly modified bitumen has been presented in Table 1.10.

Table 1.10.

Classification of highly modified bitumen types manufactured acc. to EN 14023

| TYPE OF BITUMINOUS BINDER | HIGHLY MODIFIED BITUMEN |
|---|--|
| Reference document | PN-EN 14023:2011/Ap2:2020-02 |
| Standard designation of bituminous binder | PMB X/Y-Z |
| Type of bituminous binder supplied by ORLEN Group | ORBITON 25/55-80 HiMA ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA |

Notes to designations:

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] acc. to EN 1427.

PMB – abbreviation stands for Polymer Modified Bitumen (usually replaced by the manufacturer's trade name)

ORBITON HiMA – bitumen trade name

1.5.2. REQUIREMENTS FOR HIGHLY MODIFIED BITUMEN

In February 2020, the Polish Committee for Standardisation issued a new National Annex to EN 14023, establishing updated requirements for highly polymer modified bitumen.

The division into types, classes and requirements according to the new National Annex NA, Table NA.2 to PN-EN 14023:2011 is presented in Table 1.11

Table 1.11.

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/ Ap2:2020-02

| PROPERTY | TEST METHOD | UNIT | ORBITON 25/55-80 HIMA | | ORBITON 45/80-80 HIMA | | ORBITON 65/105-80 HIMA | | |
|---|---|-------------------|-----------------------|-------|-----------------------|-------|------------------------|-------|---|
| | | | REQUIREMENT | CLASS | REQUIREMENT | CLASS | REQUIREMENT | 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 ² | ≥ 0.5 at 15°C | 8 | ≥ 2 at 10°C | 6 | ≥ 1 at 5°C | 4 | |
| Resistance to hardening | Change of mass | % | ≤ 0.5 | 3 | ≤ 0.5 | 3 | ≤ 0.5 | 3 | |
| | Retained penetration | EN 12607-1 | % | ≥ 60 | 7 | ≥ 60 | 7 | ≥ 60 | 7 |
| | Increase in softening point | °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 | |
| Plasticity range | EN 14023 Item 5.2.8.4 | °C | NR | 0 | NR | 0 | NR | 0 | |
| Drop in softening point after EN 12607-1 | EN 1427 | °C | TBR | 1 | TBR | 1 | TBR | 1 | |
| Elastic recovery at 25°C after EN 12607-1 | EN 13398 | % | ≥ 50 | 4 | ≥ 60 | 3 | ≥ 70 | 2 | |
| Storage stability Difference in softening point | EN 13399 EN 1427 | °C | ≤ 5 | 2 | ≤ 5 | 2 | ≤ 5 | 2 | |
| Storage stability Difference in penetration | EN 13399 EN 1426 | 0.1 mm | NR | 0 | NR | 0 | NR | 0 | |

* change of mass may be a positive or negative value
NR – No Requirement
TBR – To Be Reported

1.5.3. DESCRIPTION OF HIGHLY MODIFIED BITUMEN

Many research works and implementations of highly modified binders have demonstrated that they are products with above-standard functional properties. They are characterised, among others, by a very good resistance to rutting and excellent fatigue performance

and resistance to cracking. Figure 1.5. shows a chart of Pen25-SP R&B (Penetration at 25°C vs Softening Point Ring&Ball) which presents how the highly modified bitumen are positioned relative to the paving grade and conventional modified bitumen

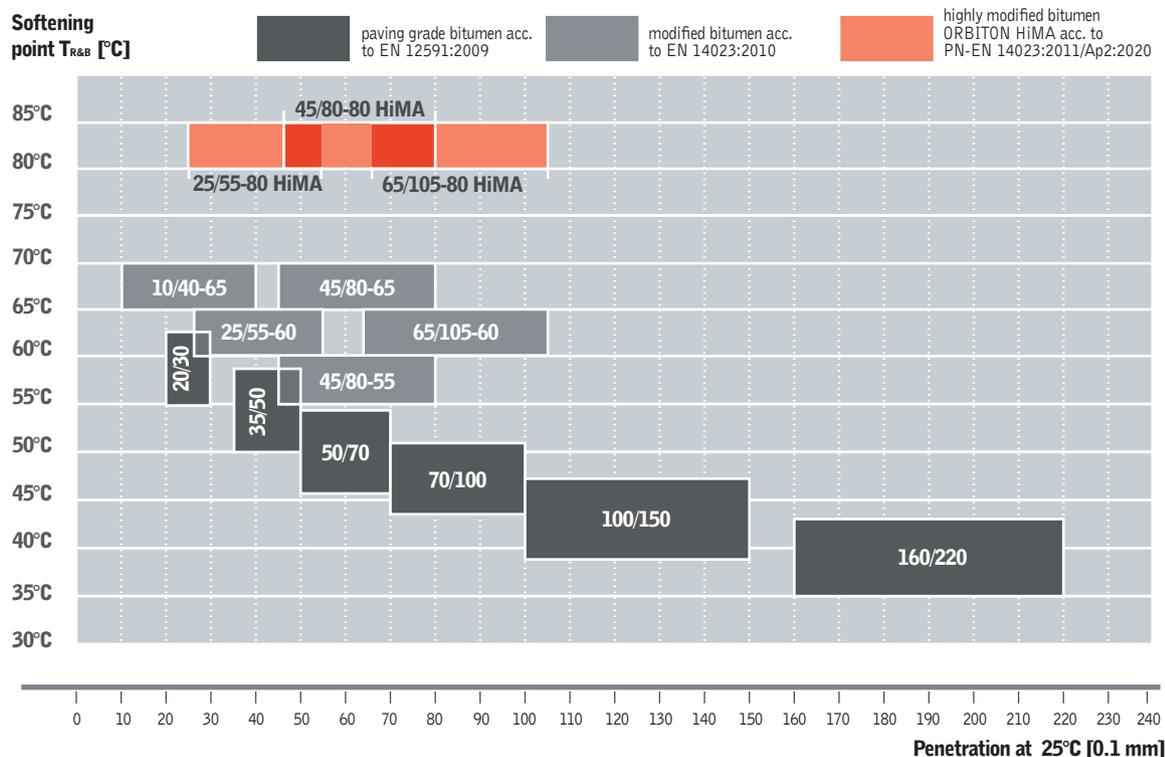


Fig. 1.5. Positioning of highly modified bitumens ORBITON HiMA relative to paving grade and conventional polymer modified bitumen in the Pen25-SP R&B chart

Highly modified binders have a R&B softening point above 80°C, regardless of hardness. This means that whether ORBITON HiMA with the penetration value of 50 or 100 is used, the properties

at a high temperature will be similar. This is due to the reversed polymer phase and the dominant influence of the polymer on the properties of this type of bitumen.

1.5.4. APPLICATION OF HIGHLY MODIFIED BITUMEN

ORBITON HiMA highly modified binders are particularly suitable for applications requiring very high durability, such as:

- asphalt pavements subjected to very high stress and strain,
- asphalt base courses with very good fatigue performance,
- courses with high resistance to low temperature

Highly modified bitumens are also particularly suitable for long-service-life pavements such as perpetual pavements. Application of ORBITON HiMA in the special anti-fatigue layer allows to achieve a very long pavement service life.

Recommended applications of particular types of highly modified bitumen are shown below.

ORBITON 25/55-80 HiMA ("hard HiMA") is designed for special courses requiring exceptional deformation resistance (parking lots for heavy vehicles, container terminals, etc.) and where very heavy, slow traffic takes place. Since this binder is very hard, it should be used only in justified cases and suitable site conditions need to be ensured. It should be clearly stated that ORBITON 25/55-80 HiMA is not a direct replacement for PMB 25/55-60 and is not intended for AC EME. For typical pavement constructions, ORBITON 45/80-80 HiMA is recommended instead of ORBITON 25/55-80 HiMA.

ORBITON 45/80-80 HiMA ("medium HiMA") is suitable for all asphalt layers of flexible pavements: base courses, including perpetual pavements, binder courses and wearing courses subject to very high loads. This universal binder combines elasticity with a very good rutting resistance.

ORBITON 65/105-80 HiMA ("soft HiMA") is mainly suitable for wearing courses made of BBTM, AUTL, DSH, PA, SMA and special technologies, e.g. SAMI courses ("hot spray" application). Due to its top fatigue properties, it can be a component of anti-fatigue (AF) layers in the perpetual pavements concept. Another application of this binder is the production of bituminous

emulsions intended for the slurry seal or surface dressing. Where high water tightness and elasticity is needed, ORBITON 65/105-80 HiMA may be used for the mastic asphalt MA.

Figures 1.6 and 1.7 can be used to choose the right type of ORBITON HiMA for a flexible pavement construction.

The designation of mixtures as "AF" in Fig. 1.7 means "anti-fatigue" layers which refers to asphalt mixes with special properties other than those typical of a base course. These may be regular mixtures with modified properties (e.g. reduced voids content) or special mixtures according to separate specifications.

The data in Figures 1.6 and 1.7 may differ from the information given in WT-2 2014 – the decision on the specific use of a given binder is up to the Investor/Designer.

Tables 1.12 and 1.13 show recommended application of highly modified bitumen depending on the road surface course. Well-designed asphalt mixtures produced using highly modified bitumens (HiMA) allow to achieve much better properties compared to their counterparts of similar hardness (paving grade and polymer modified bitumens). More details about designing asphalt mixtures and pavements with ORBITON HiMA can be found in the ORLEN Asphalt publication: Asphalt mixtures and pavements with ORBITON HiMA [7].

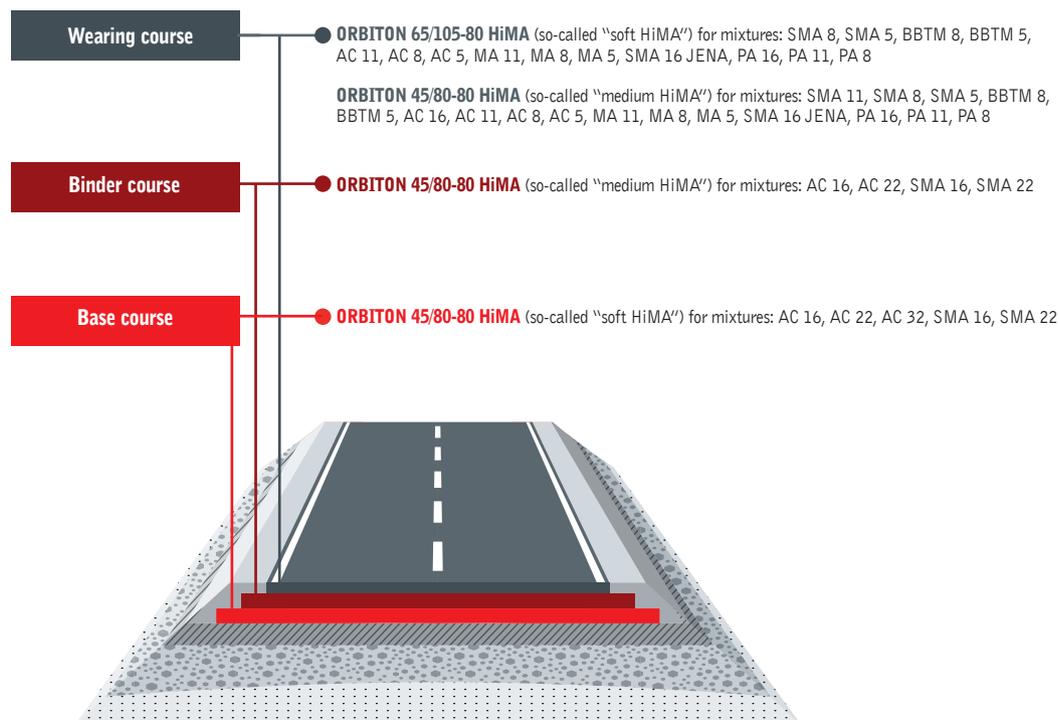


Fig. 1.6.

Proposed use of ORBITON HiMA binders for conventional flexible pavement structure (note: mastic asphalts MA for special applications and engineering structures) [own elaboration]

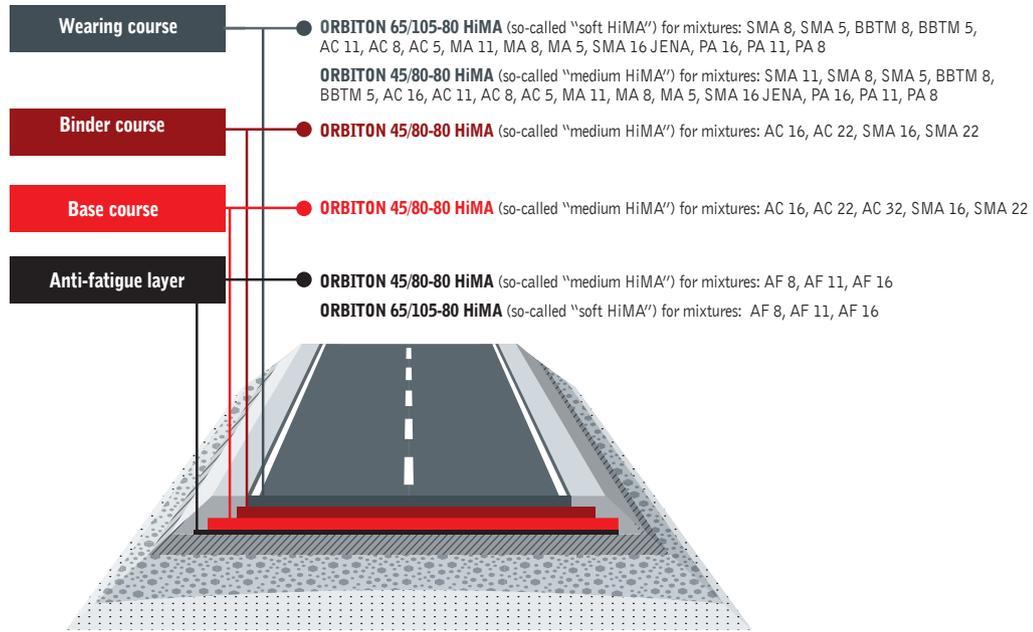


Fig. 1.7. Proposed application of ORBITON HiMA binders for a modern perpetual-type flexible pavement structure [own elaboration]

Table 1.12. Recommended application of highly modified bitumens depending on the **road surface** course

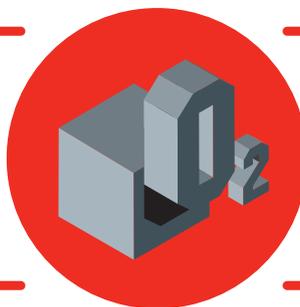
| COURSE | TRAFFIC CATEGORY* | | |
|---------|-------------------|---|--|
| | KR 1-2 | KR 3-4 | KR 5-7 |
| Base | – | ORBITON 45/80-80 HiMA | ORBITON 25/55-80 HiMA ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA |
| Binder | – | ORBITON 45/80-80 HiMA | ORBITON 25/55-80 HiMA ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA |
| Wearing | – | ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA | ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA |

* Description in table 1.4.

Table 1.13. Recommended application of highly modified bitumens depending on the **bridge surface** course

| COURSE | BINDERS |
|---------|---|
| Binder | ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA |
| Wearing | ORBITON 45/80-80 HiMA ORBITON 65/105-80 HiMA |

1.6. OXIDISED (INDUSTRIAL) BITUMEN ACC. TO EN 13304



Oxidised bitumen is produced according to EN 13304 *Bitumen and bituminous binders. Principles of classification for oxidised bitumen*.

Oxidised bitumen, commonly referred to as industrial bitumen or abbreviated as PS (in Poland), is produced in the technology of oxidation of vacuum residue using flux fractions. Usually, the Penetration Index of such binders is greater than 2.0, so they have a very low thermal sensitivity. A characteristic feature of these bitumens is a very high softening point in relation to penetration.

Oxidised bitumens are not construction products, they are not covered by Regulation 305/11 of the European Parliament and of the Council and they are not CE marked and no declaration of performance is issued.

The only formal document is a specification of product properties agreed between the producer and the customer and a Quality Certificate as a control document issued for each production batch.

1.6.1. CLASSIFICATION OF OXIDISED BITUMEN

Classification of oxidised bitumens manufactured in accordance with EN 13304 is shown in Table 1.14.

Table 1.14.

Classification of industrial bitumen manufactured in accordance with EN 13304

| TYPE OF BITUMINOUS BINDER | INDUSTRIAL BITUMEN |
|---|--|
| Reference document | EN 13304 |
| Standard designation of bituminous binder | XX/YY |
| Type of bituminous binder supplied by ORLEN Group | 80/15, 85/25, 85/40, 95/35, 100/40, 105/15 |

Notes to designations:

XX – softening temperature as the midpoint of the interval $\pm 5^{\circ}\text{C}$

YY – penetration at 25°C as the midpoint of the interval $\pm 5 \times 0.1 \text{ mm}$

e.g. 85/25 means that the softening point of the product, determined by the Ring and Ball method is between 80°C and 90°C and the penetration between $20 \times 0.1 \text{ mm}$ and $30 \times 0.1 \text{ mm}$.

1.6.2. REQUIREMENTS FOR OXIDISED BITUMEN

EN 13304 standard basically gives only two classification properties and specifies the requirements for three properties, leaving the rest to be agreed between the producer and

the customer. Table 1.15. shows the classification and required properties for industrial bitumen.

Table 1.15.

Specification of oxidised bitumen acc. to EN 13304

| PROPERTY | TEST METHOD | UNIT | VALUE |
|--|-------------|-------------------|-------|
| CLASSIFICATION PROPERTIES | | | |
| Ring and ball softening point | EN 1427 | °C | ±5 |
| Penetration at 25°C | EN 1426 | 0.1 mm | ±5 |
| OBLIGATORY PROPERTIES | | | |
| Solubility in toluene | EN 12592 | % | ≥ 99 |
| Loss in mass after heating | EN 13303 | % | ≤ 0.5 |
| Flash point | EN ISO 2592 | °C | > 250 |
| PROPERTIES SUBJECT TO AGREEMENT BETWEEN THE PRODUCER AND THE CUSTOMER (NR – NO REQUIREMENT) | | | |
| Fraass breaking point | EN 12593 | °C | NR |
| Staining properties | EN 13301 | mm | NR |
| Dynamic viscosity | EN 13302 | Pa·s | NR |
| Density | EN 15326 | kg/m ³ | NR |

It is worth noting unusual properties such as “staining properties”, used when the customer is planning to use bitumen for production of, for example, asphalt shingles. Tested attribute determines whether there is a risk that stains will appear on the sprinkle on the surface of the shingles. In contrast,

“loss in mass after heating” is not the same test as a rolling thin film ageing resistance (as RTFOT is for bituminous binders for paving application) as it is performed in a much thicker layer, without rotation.

As stated earlier, no European specification with requirements has been created for oxidised bitumen, leaving most requirements to be agreed between the customer and the manufacturer.

Table 1.16. shows the most commonly specified requirements for oxidised bitumen.

Table 1.16.

Most frequent requirements for oxidised bitumen

| PROPERTY | TEST METHOD | UNIT | 80/15 | 85/25 | 85/40 | 95/35 | 100/40 | 105/15 |
|-------------------------------|-------------|-------------------|-------|-------|-------|--------|--------|---------|
| Ring and ball softening point | EN 1427 | °C | 75-85 | 80-90 | 80-90 | 90-100 | 95-105 | 100-110 |
| Penetration at 25°C | EN 1426 | 0.1 mm | 10-20 | 20-30 | 35-45 | 30-40 | 35-45 | 10-20 |
| Solubility in toluene | EN 12592 | % | ≥ 99 | ≥ 99 | ≥ 99 | ≥ 99 | ≥ 99 | ≥ 99 |
| Loss in mass after heating | EN 13303 | % | ≤ 0.5 | ≤ 0.5 | ≤ 0.5 | ≤ 0.5 | ≤ 0.5 | ≤ 0.5 |
| Flash point | EN ISO 2592 | °C | > 250 | > 250 | > 250 | > 250 | > 250 | > 250 |
| Fraass breaking point | EN 12593 | °C | ≤ -7 | TBR | TBR | ≤ -20 | TBR | TBR |
| Staining properties | EN 13301 | mm | TBR | NR | NR | NR | NR | NR |
| Dynamic viscosity | EN 13302 | Pa·s | TBR | NR | NR | NR | NR | NR |
| Density | EN 15326 | kg/m ³ | TBR | NR | NR | NR | NR | NR |

NR – No Requirement
TBR – To Be Reported

NOTE

Oxidised bitumen is not suitable for use in road construction! Due to their specific properties, it is not possible to achieve a durable asphalt pavement.

2. BASIC PROPERTIES OF BITUMINOUS BINDERS

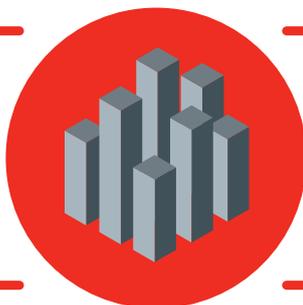
During the production season, bituminous binders are manufactured in numerous batches. Each batch must be tested for specific properties according to the product standards and the Factory Production Control principles before it is released for sale. An important feature of products from various production batches, both for the manufacturer and the user, is the homogeneity of their properties.

The observation of the properties of individual batches is subject to a random error each time and does not allow to formulate any conclusions regarding their significance. Only the analysis of as large a number of results as possible using appropriate statistical tools allows to discover and understand certain regularities

that characterise a given set of results and to observe relations between them [1].

This chapter contains information on the properties of bituminous binders specified in EN 12591 and EN 14023, as well as data on the density of bituminous binders and the microstructure of polymer modified bitumen. Statistical parameters of the results of laboratory control of selected properties of bituminous binders produced by ORLEN Group (installations in Płock and Trzebinia) in the period from January 2018 to April 2020 are shown in the tables and figures below. The tests were performed in accredited units of ORLEN Laboratorium S.A.

2.1. INTRODUCTION TO STATISTICS



The term 'statistics' has been derived from the Latin word 'status' meaning state, condition. The origins of statistics reach the ancient times, when general population censuses were conducted. Nowadays, the term statistics has a much broader meaning and is used in numerous fields¹ [2]. Statistics, which we are interested in, is defined as an independent scientific discipline dealing with quantitative methods of

studying regularities in statistical populations and providing reliable information about these regularities characterised by numbers [3]. The description of surrounding phenomena and objects and the evaluation of relations between them, including the assessment of quality and homogeneity of individual batches of bituminous binders, is possible with the use of appropriate tools such as statistical methods.

1) Did you know that...with the help of statistics, it is possible to create a mathematical formula for good beer? One of the most famous statisticians in the world – William Gosset – brewed beer professionally, more specifically Irish Guinness, and developed one of the most popular statistical tests, the so-called Student's t-test. This test was designed to develop a mathematical method to ensure that every beer that leaves the brewery is as good as the reference beer. And why the name Student's t-test? Gosset had to remain anonymous and adopt a certain pseudonym, due to the fact that his management did not want to reveal to competitors that they had the mathematical formula for a perfect beer [2].

Three basic areas of activities can be distinguished in statistics [4]:

- **informative** – presents a complete and objective picture of the studied phenomena,
- **analytical** – allows to determine factors that shape particular processes and phenomena,
- **prognostic** – enables the prediction of the development direction of the studied phenomena.

Statistics as a scientific discipline can be divided into two main fields [1, 5, 6]:

- **descriptive statistics**, or statistical description, dealing with methods of collecting, processing and presenting data with their summary description;
- **mathematical statistics**, called statistical inference, which covers methods of investigating dependencies and regularities in an analysed set of data.

Statistical surveys is a set of activities carried out on a specific statistical population in order to find out the properties that characterize the studied set [4].

Statistical population is a set of any elements e.g. people, objects, events, etc. characterised by at least one common

feature with different values. This population must be uniquely defined in terms of material, spatial and temporal [1, 5].

Statistical unit is the smallest element of a statistical population that is included in a statistical survey [5].

Statistical characteristic (variable) is a property of the statistical units constituting a defined statistical population [4].

For example, in statistical testing of bituminous binder performance:

- **statistical population** = all production batches of paving grade bitumen 50/70 in 2020,
- **statistical unit** = a single production batch of paving grade bitumen 50/70 e.g. on 25.09.2020,
- **statistical characteristic** = penetration of the paving grade bitumen 50/70.

Statistics is an excellent tool for searching answers to questions concerning the world around us. However, this tool should be used reasonably and skilfully [7], otherwise it may lead to erroneous or different interpretations of the obtained results.

2.1.1. STATISTICAL PARAMETERS, DEFINITIONS

Statistical parameters can be called numerical quantities that make it possible to describe the structure of the statistical population under study in a systematic manner.

Statistical parameters can be divided into several general categories [1, 5]:

- **measures of position** – also known as average measures describing the statistical population without taking into account the differences between individual units of this population. These measures characterise the similarity of the population taking into account the analysed variable. Position measures are divided into classical (e.g. arithmetic mean) and positional (e.g. modes, quartiles) measures;
- **measures of variability** – called dispersion measures, describe a statistical population taking into account differences

between particular units belonging to the population. Measures of variability characterise the degree of variation in the population taking into account the variable trait under analysis. Measures of variability can be divided into classical (variance, standard deviation, typical range, classic coefficient of variation) and positional (e.g. range, quartile deviation) measures;

- **measures of asymmetry** – measures of skewness specifying the distribution direction of the variables in the population and to what extent the distribution of a given characteristic deviates from the symmetric distribution;
- **measures of concentration** – describe the concentration degree of particular observations around the mean value (kurtosis) or the degree of the distribution unevenness of a phenomenon in a population.

The most common statistical measures used to describe bituminous binders produced by ORLEN Group are:

Arithmetic mean, x_{mn} – a conventional measure of position, the sum of the values of a variable characteristic divided by the number of units of a finite statistical population:

$$x_{mn} = \frac{1}{n} \sum_{i=1}^n x_i$$

where:

x_i – value of the variable trait/value of a single determination result

n – size of the population/number of obtained results

The main disadvantage of the arithmetic mean is its sensitivity to extreme values of a characteristic.

The lower quartile (first quartile), Q_1 – positional measure of position; the value of a variable dividing the tested population in such a way that 25% of the elements of the population ordered in a non-decreasing sequence are below this value. Graphical representation of the value is shown in Fig. 2.1a.

Median (second quartile), Me or Q_2 – a positional measure of position; the value of a variable dividing the studied population in half, in such a way that below and above its value there are respectively half of the units of the population (Fig. 2.1b.).

For an n -odd non-decreasing sequence of observed values, the median is the middle value in the series. For an n -even non-decreasing sequence, the median is the arithmetic mean of the two middle values. It can be determined graphically or by calculation.

The Upper quartile (third quartile), Q_3 – a positional measure of position; it is the value of observation dividing the examined population in such a way that 75% of the units of the population ordered in a non-decreasing sequence are below this value. Graphical representation of the value is shown in Fig. 2.1c.

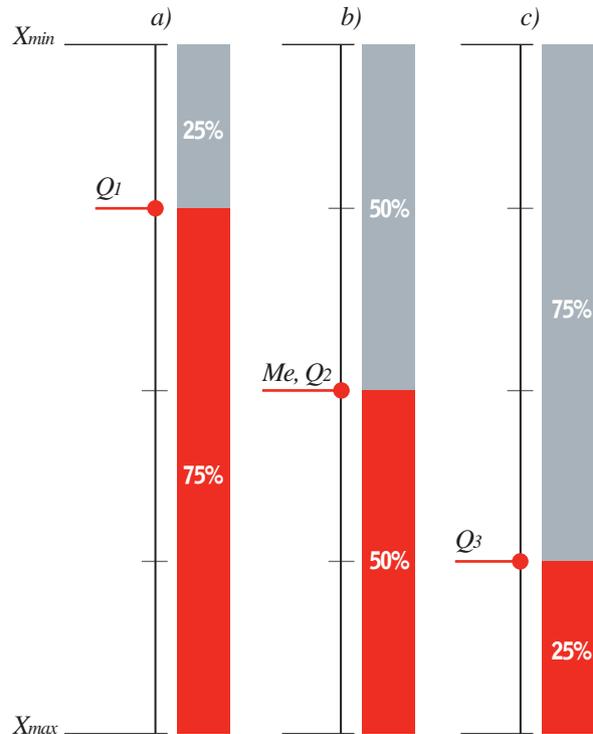


Fig. 2.1. Graphical representation of the median and other quartiles

Standard deviation, σ – defined as a conventional measure of variation indicating the extent to which the values are dispersed around the mean value. If all the results were the same, the standard deviation would be zero. In other cases, this number is a positive value. Hence, the greater the value of the standard deviation, the greater the dispersion of the results around the mean [7].

Standard deviation is calculated according to the formula:

$$\sigma = \sqrt{\frac{\sum_{i=1}^n (x_i - x_{mn})^2}{n}}$$

in which:

x_i – value of the variable trait/value of a single determination result

x_{mn} – arithmetic mean of the variable/arithmetic mean of the obtained results

n – size of the population/number of obtained results

The standard deviation for normal distribution is connected with the **so-called three-sigma** rule of thumb, which states that practically the whole statistical population is contained in the interval $x_{mn} \pm 3\sigma$. At the same time "practically the whole" means 99.73%, thus 3 out of 1000 results of determinations may be outside the interval [8].

Typical range x_{type} , is the interval containing units of a statistical population that differ from the mean by maximum one standard deviation.

$$x_{mn} - \sigma < x_{type} < x_{mn} + \sigma$$

This interval contains about 68% of the units of the tested statistical population.

Range, R – positional measure of variation, which is the difference between the highest and the lowest value of a variable:

$$R = x_{max} - x_{min}$$

where:

x_{max} – maximum value of the variable

x_{min} – minimum value of the variable

Quartile range, IQR – a measure of variation, which is the difference between the third and the first quartile. It defines the range containing 50% of units of the tested population

$$IQR = Q_3 - Q_1$$

2.1.2. WAYS OF PRESENTATION OF STATISTICAL DATA

There are various forms of presentation of the statistical material. Tables are the basic way of publishing data but it also can be done using descriptive characteristics, analytical formulas or in graphical form. Graphical presentation of data helps to analyse and observe regularities that cannot be seen only on the basis of calculated statistical parameters.

One of the most popular forms of presenting data is a **histogram** (Fig. 2.2.) – a graph of the distribution (frequency of occurrence) of a studied characteristic in the form of a bar chart. The width of each bar corresponds to a certain range of the examined characteristic – a class interval. The height of the bars on the histogram, on the other hand, can represent:

- **frequency of determination results** – the ratio of the number of results obtained with the same value to the total size of the tested statistical population. For example: during the production season "penetration at 25°C" of bitumen 35/50 was determined 200 times (for 200 production batches), the result of 45 [0.1 mm] was obtained 86 times, therefore frequency of results in class 45 is $86/200 = 0.43$. The frequency can also be expressed as a percentage, in which case it means the percentage share of the particular result in the tested statistical population (in the example, it is 43%);

- **the size of determination results** – the number of results obtained with the same value or in the same class.

Another way of presenting the results is a box plot (Fig. 2.2.) commonly referred to as a box-and-whisker plot [9]. It contains information on the position, dispersion and shape of the distribution of the analysed data. A box plot can describe various statistical parameters. In the most common case, the length of the box is equal to the quartile range (IQR or the difference $Q_3 - Q_1$, i.e. the range between 25% and 75% of the sequence of results). The minimum and maximum values are determined by characteristic lines called whiskers. Inside the box, a vertical line marks the median value. If the median is in the middle of the box, it can be concluded that the distribution of a given characteristic is **symmetric** (the case presented in Fig. 2.2.). In a situation where the median divides the box into two unequal parts and the whiskers are of different lengths, it may be dealt with an **asymmetric distribution** means that the results of a given variable are not arranged symmetrically around the meaning. Depending on the length of the whiskers – longer on the right or left – it is encountered a right-side or a left-side asymmetry respectively.

The histogram together with a box plot for the exemplary tested parameter is presented in Fig. 2.2, while the graphical interpretation of the histogram bars is presented in Fig. 2.3. To avoid misunderstandings that may arise during the interpretation

of class intervals on histograms, the following interpretation of Fig. 2.3. should be adopted: the given class interval (60, 65> denotes a right-closed interval, i.e. it includes numbers 61, 62, 63, 64, 65.

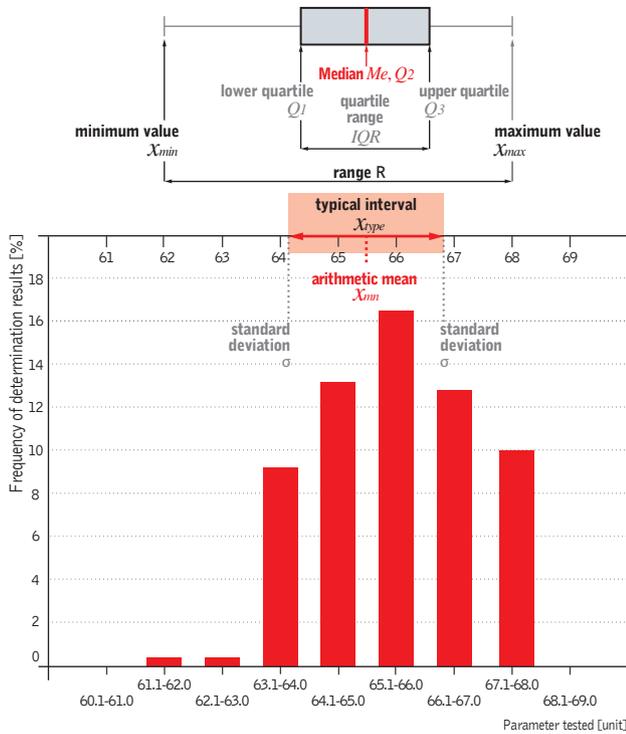


Fig. 2.2. Histogram and box-and-whisker plot for the exemplary tested parameter

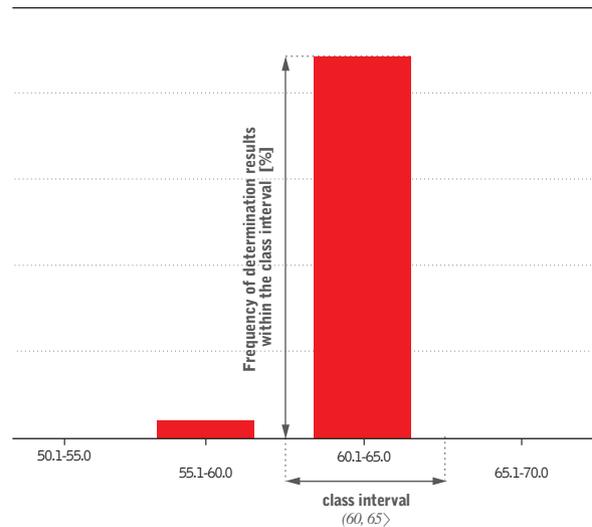


Fig. 2.3. Graphical interpretation of the histogram bars

2.1.3. INTERPRETATION OF TEST (ACCEPTANCE) RESULTS

Occasionally, there is a dispute between a customer and a bituminous binder supplier over the quality of the delivered product. Take for example the requirement for penetration at 25°C for 35/50 paving grade bitumen. When the result of Pen25 = 34 [0.1mm] is received from the laboratory, does it really mean that the delivered product does not conform to the specification?

The result of testing any parameter, such as the aforementioned penetration at 25°C of the 35/50 paving grade bitumen obtained in a given laboratory will always differ from the actual value of that feature.

The actual value of a given parameter is an abstract notion and, obviously, it is not known to the person performing the test (if it

were known then the measurement would be unnecessary) [10].

The limited accuracy of the measurement equipment, the influence of variable external conditions on the tested sample and the measurement system, as well as insufficient knowledge of all the circumstances influencing the given test, cause that the obtained result will differ from the real (i.e. true) value of the measured parameter by a certain amount.

Therefore, a measurement of a given property allows only for estimation of its approximate value. The result of a measurement obtained in the laboratory should never be expected strictly equal to the real value of the tested characteristic. It should only be assumed that it is within a certain range oscillating around the true value of the tested parameter.

2.1.3.1 DEFINITIONS RELATED TO CORRECT INTERPRETATION OF TEST RESULTS

Two concepts are inherent in any laboratory test: measurement error and uncertainty of measurement.

Measurement error is the discrepancy between the result obtained and the true value of the feature under test, which is usually unknown. It should be understood as an inherent part of the measurement process and not as an error resulting from a mistake alone. Measurement error is directly related to the given measurement method.

In laboratory work, the following types of measurement errors are distinguished [11, 12]:

- **systematic errors** – errors that remain constant when performing a series of measurements of a given characteristic under the same conditions (the same apparatus, the same operator, etc.). They result from imperfections of measurement instruments and methods. An example of a systematic error is e.g. the error of indication of a measuring instrument, provided on the calibration certificate.
- **random errors** – errors that vary in an unpredictable and random way when making a large number of measurements of the same property under practically unchanging conditions. The main causes of random errors include:
 - imperfect senses of the operator and lack of sufficient concentration during the test,
 - scattered readings of measuring instruments,
 - short-term changes in various external factors (e.g. increase in room temperature, change in mains voltage, etc.).

Reduction of the impact of random errors is achieved by repeated measurements of the same characteristic and taking the arithmetic mean as the final result.

- **excessive errors** – errors called mistakes or coarse errors; they result from incorrectly performed measurements and cause open distortion of the measurement result.

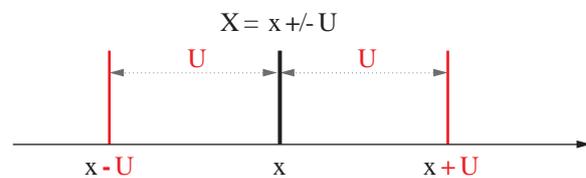
If several different measurement errors are superimposed on the result of a given test, the obtained result will be completely different from the true one.

Uncertainty of measurement is a parameter closely related to the obtained result. It characterises the dispersion of values that can be attributed to a given measured value [13]. When reporting the result of a test for a given parameter, quantitative information (“uncertainty” is always a number) about the

accuracy of the test should also be given, i.e. the previously calculated uncertainty of measurement [13, 14]².

The uncertainty of measurement of a given parameter is determined by many factors, e.g. the accuracy of the measuring instruments, external factors, such as temperature, pressure, humidity, shakes, vibrations, as well as various kinds of random errors, errors of the method or errors resulting from the evaluation of the result.

Graphically, uncertainty of measurement can be represented as follows:



X – test value, e.g. Penetration at 25°C
 x – obtained value, e.g. Pen25 = 34 [0.1 mm]
 U – uncertainty of measurement (expanded)

Fig. 2.4.

Graphical interpretation of uncertainty of measurement [12]

Thus, it can be stated that the uncertainty of measurement is the interval within which the true value of the tested parameter (variable) falls with a given probability (confidence interval).

For the example discussed earlier, the result of the penetration test together with the estimated measurement uncertainty should therefore be written as follows:

$Pen_{25} = 34 \pm U [0.1mm]$, for the confidence interval $p = \dots$

where:

U – the determined uncertainty of measurement for the laboratory

p – the adopted confidence interval.

A confidence interval is used to indicate the reliability of finding a particular result within a certain specified framework. It is always defined by an adopted confidence interval, usually expressed as a percentage; for example a “95% confidence interval”.

The extreme points of this interval are called confidence limits. Formally, a 95% confidence interval means that when repeating a given study under unchanging conditions, the interval will contain the true values of the results 95% of the time.

2) Different ways of calculating the uncertainty of measurement can be found in publications [13] and [14].

Graphical interpretation of the different confidence intervals for a normal distribution are shown in Figure 2.5.

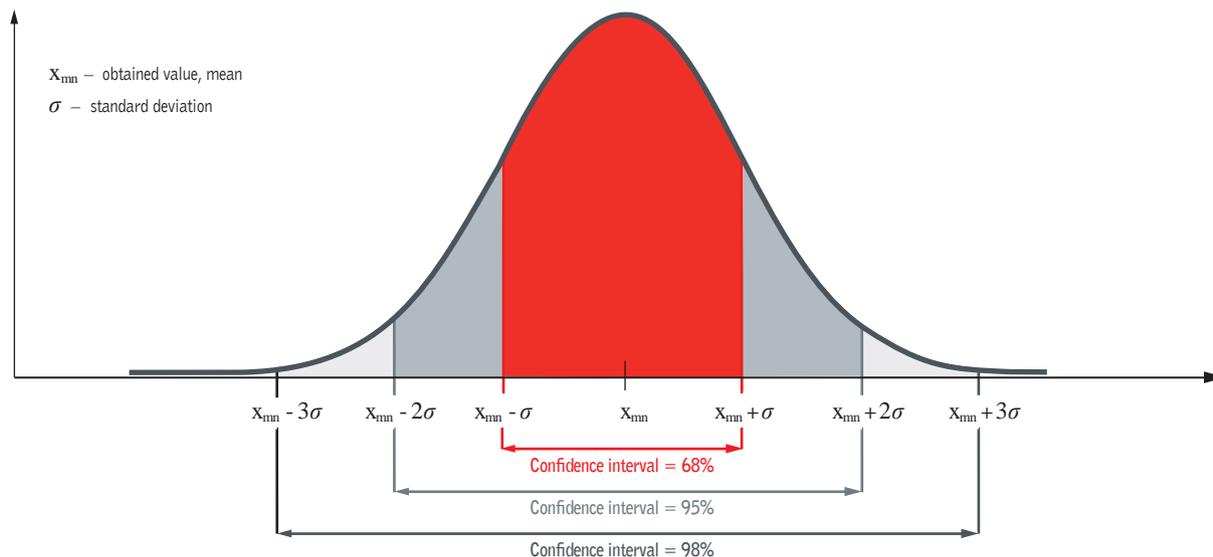


Fig. 2.5. Graphical interpretation of different confidence intervals for a normal distribution [12]

Two further terms that are inextricably linked to the tests performed in the laboratory are **precision and accuracy**. Although in everyday language these two words mean more or less the same thing, in the context of scientific testing methods, they carry distinctive meanings.

A measurement system can be precise but inaccurate, accurate but imprecise, accurate and precise, or inaccurate and imprecise. Explanation of the meaning of the combination of these terms is shown in Fig. 2.6.

A measurement is considered valid if it is both accurate and precise – option 3 in Fig. 2.6.

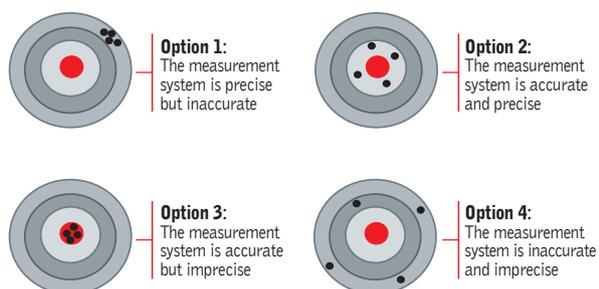


Fig. 2.6. Graphical interpretation of test precision and accuracy

Test accuracy is characterised indirectly by the opposite property: measurement error or uncertainty of measurement. It defines the degree of approximation of the measurement result to the true value of the measured characteristic.

Test precision, on the other hand, is the degree of agreement between individual results of a given analysis (in other words – the scatter of results), when a given test procedure is applied to repeated independent determinations of a given sample.

The most common measure of test precision is standard deviation, relative standard deviation or coefficient of variation.

Two further concepts are also inseparably connected with precision (scatter of results) of a given test: repeatability and reproducibility.

Repeatability (r) – the precision of results obtained under the same measurement conditions (same laboratory, analyst, measuring instrument, reagents, etc.).

Reproducibility (R) – the precision of results obtained in different laboratories using a given analytical procedure.

Repeatability and reproducibility are parameters specific to a given measurement method and they are usually given in the relevant functional standards.

The values of repeatability and reproducibility for the parameter of penetration of paving grade bitumen, specified in the functional standard of bitumen penetration EN 1426, are shown in Table 2.1:

Table 2.1.

Repeatability and reproducibility values for the penetration parameter of paving grade bitumen

| MEASUREMENT CONDITIONS | PENETRATION IN 0.1 mm | REPEATABILITY, r | REPRODUCIBILITY, R |
|---|-----------------------|-----------------------|-----------------------|
| Temperature: 25°C Needle weight: 100 g Load time: 5 s | < 50 ≥ 50 | 2 4% of mean value | 3 6% of mean value |

2.1.3.2 INTERPRETATION OF TEST RESULTS ACC. TO EN ISO 4259

EN ISO 4259 *Petroleum products and related products. Precision of measurement methods and results.* covers issues related to the correct interpretation of test results for bituminous binders.

This standard is referenced in every standard containing requirements for bituminous binders and is binding for both the manufacturer and the customer.

Limits of the characteristic

As previously written, the real value of the tested parameter can never be determined accurately in a laboratory. The parameter is measured in the laboratory using a standardised test method, the results of which may show a certain scatter defined by its repeatability and/or reproducibility.

When evaluating a test result, it is important to determine the limit or limits of the true value of the tested characteristic. The limit may be unilateral (no less than/no more than) or bilateral:

- bilateral limit (upper and lower) – **for example: penetration at 25°C from 35 to 50 [0.1 mm],**
- **unilateral limit** (upper or lower) – e.g. Fraass braking point = not higher than -18°C or elastic recovery at 25°C = not lower than 80%. Sometimes, there is an additional implicit limit, e.g. in the case of solubility with a unilateral requirement of "not lower than 99%" there is logically an additional limit of 100% – in such cases the unilateral limit becomes bilateral. The same is true for the elastic recovery, which also cannot be greater than 100%.

In EN ISO 4259, the upper limit is designated as A_1 and the lower limit as A_2 .

Determination of limits in specifications

At this point, it is necessary to add a few words about creating specifications (requirements). The rules given in EN ISO 4259 clearly indicate that the limit value of the tested characteristic should take into account the reproducibility of the adopted test method, as follows:

- for a bilateral limit (A_1 and A_2), the specified range should not be smaller than four times the reproducibility value R:

$$(A_1 - A_2) \geq 4 \cdot R$$

- for a unilateral limit (A_1 or A_2), the specified range should not be smaller than twice the reproducibility value R:

$$A_1 \geq 2 \cdot R \quad \text{lub} \quad A_2 \geq 2 \cdot R$$

If the condition $(A_1 - A_2) \geq 4 \cdot R$ is not fulfilled, then either the limits of the requirement should be widened or a test method with better precision should be sought. This means that the requirements for a characteristic in the specification must take into account the precision of the test method. Otherwise, conflicts between the supplier and the customer will be inevitable.

Evaluation of the measurement result for compliance with the specification

If the only source of information about a product parameter is a single result³⁾, then it should be assumed that the characteristics of that product are within the requirement range with a 95% confidence level only if the test result (denoted here as Y) is as follows:

- for unilateral upper limit A_1 :

$$Y > A_1 + 0,59 \cdot R$$

- for unilateral lower limit A_2 :

$$Y > A_1 + 0,59 \cdot R$$

- for bilateral limit – respectively one of the requirements should be met (one, since a result outside the lower or upper limit of the requirement range is usually questioned).

3) A single result in the case of the penetration test according to EN 1426 should be understood as the arithmetic mean of at least three measurements taken under the repeatable conditions of the method.

Returning to the example with the penetration value of 35/50 paving grade bitumen, let's consider a situation where the customer has received a control test result from their own laboratory:

Pen25 = 34 [0.1mm].

Knowing the above principles of interpretation and statistical analysis of test results, let's ask a question: is the obtained result consistent or inconsistent with the specification given in EN 12591? Can the customer conclude that they received a 35/50 paving grade bitumen or should they file a quality complaint because the penetration value is too low?

The result, Pen25 = 34 [0.1 mm] designated as Y. The standard limits for 35/50 bitumen are

- lower limit: $A_2 = 35$ [0.1 mm],
- upper limit: $A_1 = 50$ [0.1 mm],

therefore the result Pen25 = 34 [0.1mm] is just outside the lower specification limit A_2 .

The standard for penetration testing of bituminous binders (EN 1426) specifies the method reproducibility of $R = 3$ [0.1 mm] for bitumen with a penetration at 25°C of less than 50 [0.1 mm].

Let's calculate if the supplier has delivered bitumen compliant with the standard:

$$35 - 0,59 \cdot 3 < Y < 50 + 0,59 \cdot 3$$

$$33,2 < Y < 51,8$$

In this case, the test result of $Y = 34$ [0.1 mm] is within the specification limits extended by the uncertainty of the penetration measurement. To reject the delivery, the customer would have to find that the result is less than 33.2 [0.1 mm] or greater than 51.8 [0.1 mm] (Figure 2.7).

Considering the above, it should be concluded that the supplied 35/50 paving grade bitumen with Pen = 34 [0.1 mm] is accordance with the standard, so there is no formal basis for an official quality complaint.

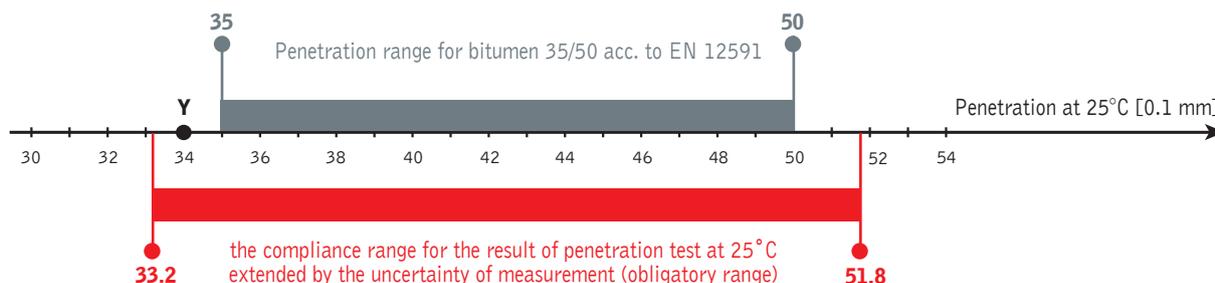


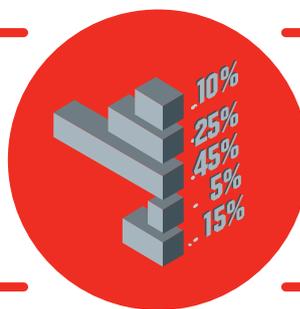
Fig. 2.7.

Illustration to the example – ranges of compliance for the penetration result extended by the value uncertainty of measurement [own elaboration]

Contentious cases

If the customer and the product supplier cannot agree on the quality of the delivered product, the procedure described in pt. 7 and in Annex B of EN ISO 4259-2 on acceptance and rejection of results in cases of dispute should be applied.

2.2. RESULTS OF SELECTED PROPERTIES OF BITUMINOUS BINDERS



This part of the chapter presents the results of laboratory control of selected parameters of bituminous binders produced by ORLEN Group from January 2018 to April 2020. The analysed data were presented by individual properties of bituminous binders.

Statistical data for all bituminous binders were included in tables and for selected characteristics (properties) of the most popular bituminous binders, the data were also presented in graphical form using histograms.

2.2.1. PENETRATION AT 25°C

Penetration is a basic test for evaluating the consistency of bituminous binders, conventionally expressed as the depth to which a standardised steel needle penetrates vertically into the bitumen sample at a given temperature.

Penetration test is carried out in accordance with EN 1426. The measurement can be performed at various temperatures but measurement at 25°C is the basic way to classify bituminous binders according to the European standardisation.

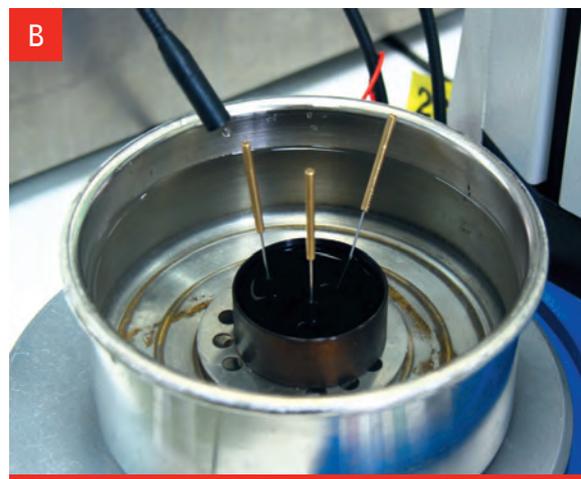


Fig. 2.8.

(A) General view of the penetration testing apparatus with a bitumen sample, (B) view of a bitumen sample after the test (photo by ORLEN Asphalt sp. z o.o., with the permission of ORLEN Laboratorium S.A.)

Statistical parameters of the results of the determination of penetration at 25°C for bitumens produced between 2018 and 2020 are shown in Table 2.2.

Table 2.2.

Statistical parameters of the results of the determination of penetration at 25°C for bitumens produced between 2018 and 2020*

| TYPE OF BINDER | REQUIREMENT** [0.1 mm] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUARTILE | THIRD QUARTILE | MIN-MAX VALUE |
|------------------------|---------------------------|-----------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | \bar{x}_{min} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| 20/30 | 20 ÷ 30 | 27.0 | 1.5 | 25.5 ÷ 28.5 | 27 | 26 | 28 | 24 ÷ 29 |
| 35/50 | 35 ÷ 50 | 43.7 | 2.4 | 41.3 ÷ 46.1 | 44 | 43 | 45 | 39 ÷ 48 |
| 50/70 | 50 ÷ 70 | 61.4 | 3.0 | 58.4 ÷ 64.4 | 61 | 59 | 63 | 53 ÷ 70 |
| 70/100 | 70 ÷ 100 | 89.8 | 4.8 | 85.0 ÷ 94.6 | 90 | 87 | 92 | 78 ÷ 100 |
| 100/150 | 100 ÷ 150 | 125.9 | 7.4 | 118.5 ÷ 133.3 | 124 | 122 | 131 | 112 ÷ 140 |
| 160/220 | 160 ÷ 220 | 182.3 | 6.4 | 176.9 ÷ 189.7 | 183 | 178 | 185 | 166 ÷ 198 |
| ORBITON 25/55-60 | 25 ÷ 55 | 38.5 | 4.2 | 34.3 ÷ 42.7 | 38 | 37 | 40 | 31 ÷ 52 |
| ORBITON 45/80-55 | 45 ÷ 80 | 66.6 | 3.5 | 63.1 ÷ 70.1 | 67 | 64 | 70 | 60 ÷ 73 |
| ORBITON 45/80-65 | 45 ÷ 80 | 57.5 | 3.4 | 54.1 ÷ 60.9 | 57 | 56 | 59 | 52 ÷ 64 |
| ORBITON 25/55-80 HiMA | 25 ÷ 55 | 49.7 | 3.1 | 46.6 ÷ 52.8 | 50 | 47 | 52 | 44 ÷ 53 |
| ORBITON 45/80-80 HiMA | 45 ÷ 80 | 67.0 | 5.6 | 61.4 ÷ 72.6 | 65 | 63 | 70 | 59 ÷ 80 |
| ORBITON 65/105-80 HiMA | 65 ÷ 105 | 86.8 | 7.3 | 79.5 ÷ 94.1 | 88 | 82 | 90 | 74 ÷ 100 |

* test results refer to the period: January 2018–April 2020

** for paving grade bitumen according to EN 12591; for ORBITON and ORBITON HiMA according to EN 14023

Additionally, Figures 2.9. to 2.13. show histograms with box plots for penetration results of selected bitumens produced in the 2018–2020 period.

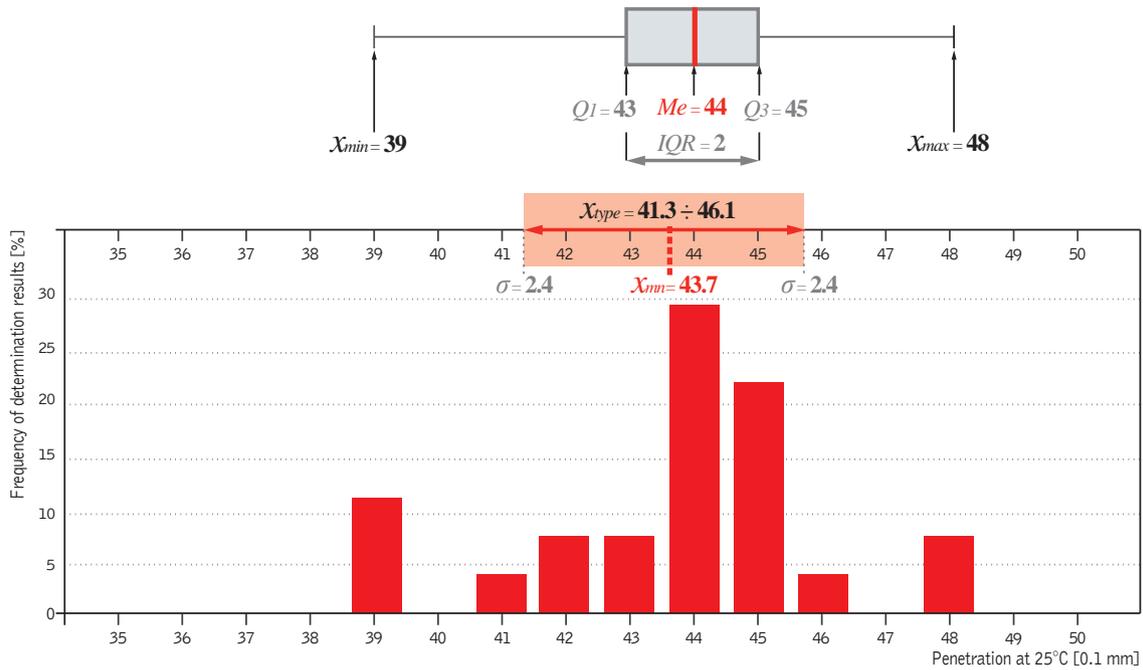


Fig. 2.9.

Histogram and box plot presenting the results of penetration determinations at 25°C of **paving grade bitumen 35/50** produced in the 2018–2020 period (standard range: 35–50 [0.1 mm])

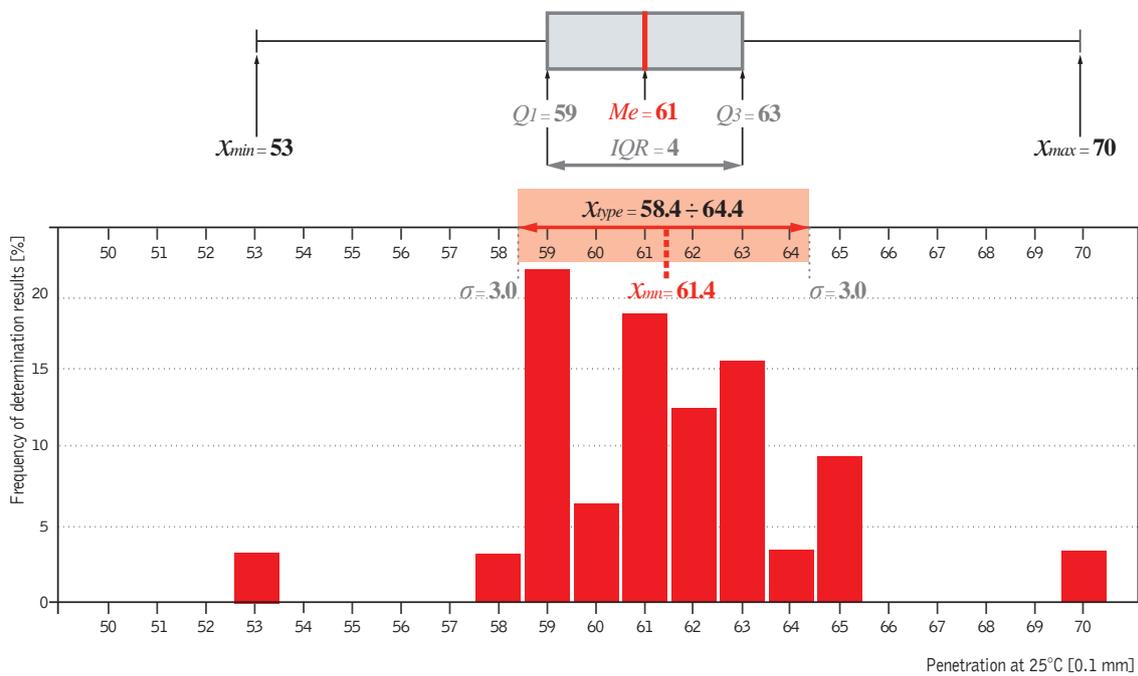


Fig. 2.10.

Histogram and box plot presenting the results of penetration determinations at 25°C of **paving grade bitumen 50/70** produced in the 2018–2020 period (standard range: 50–70 [0.1 mm])

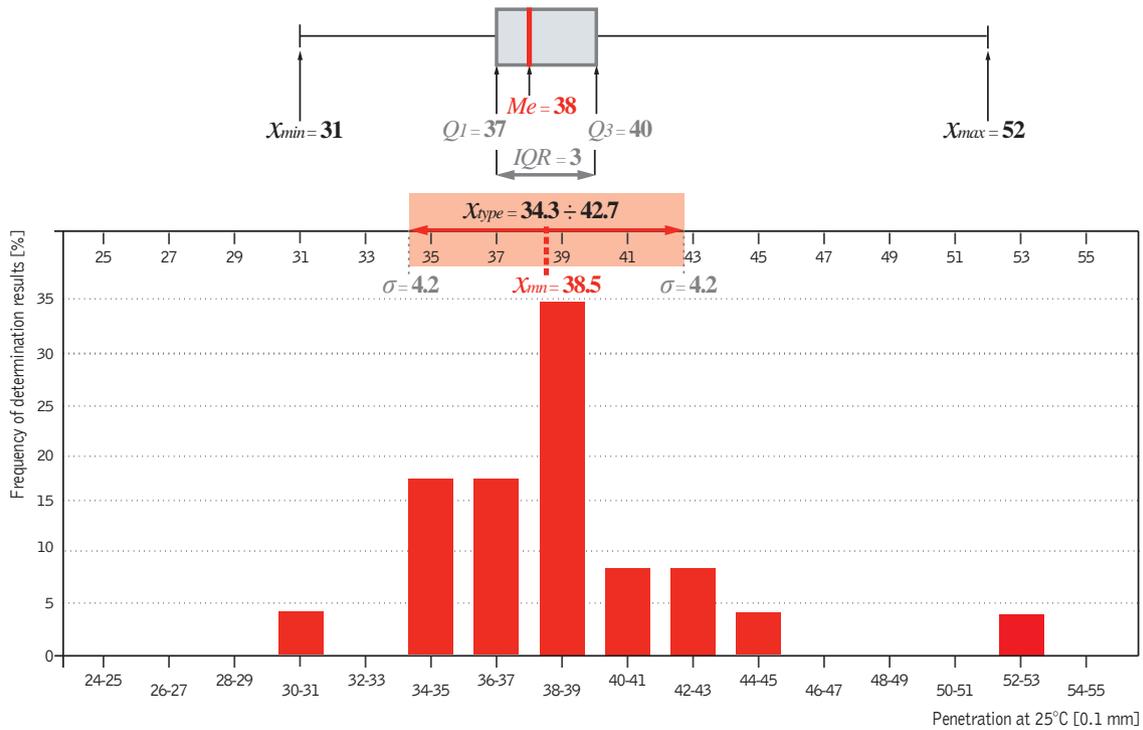


Fig. 2.11. Histogram and box plot presenting the results of penetration determinations at 25°C of polymer modified bitumen **ORBITON 25/55-60** produced in the 2018–2020 period (standard range: 25–55 [0.1 mm])

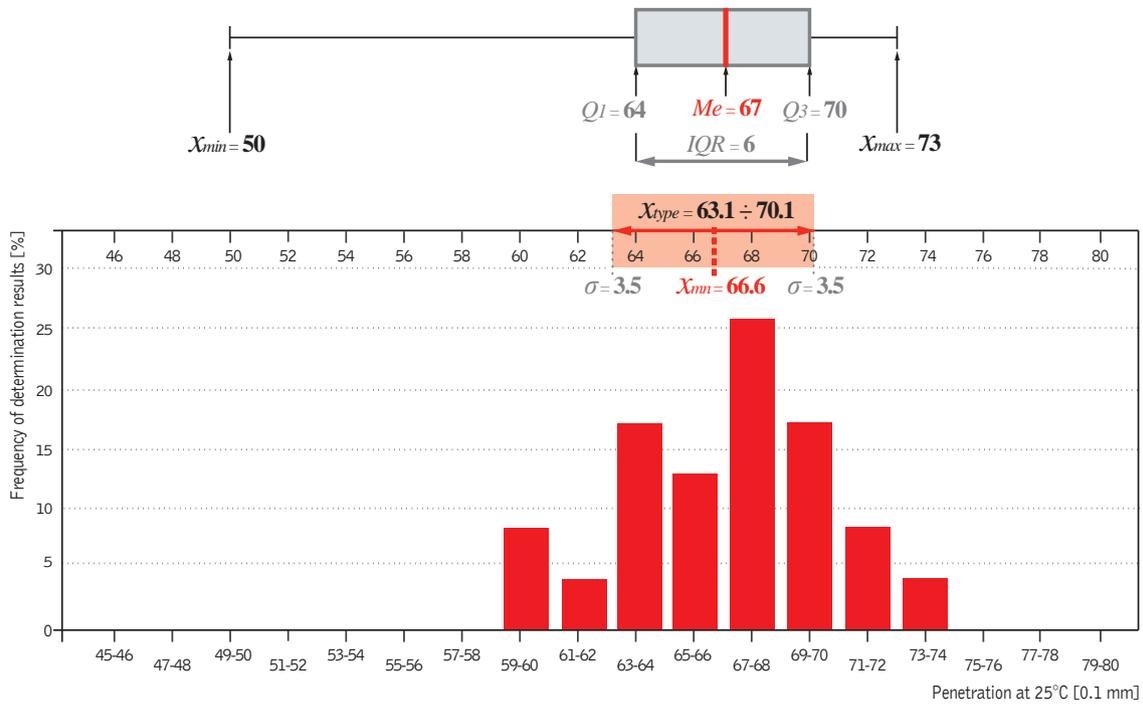


Fig. 2.12. Histogram and box plot presenting the results of penetration determinations at 25°C of polymer modified bitumen **ORBITON 45/80-55** produced in the 2018–2020 period (standard range: 45–80 [0.1 mm])

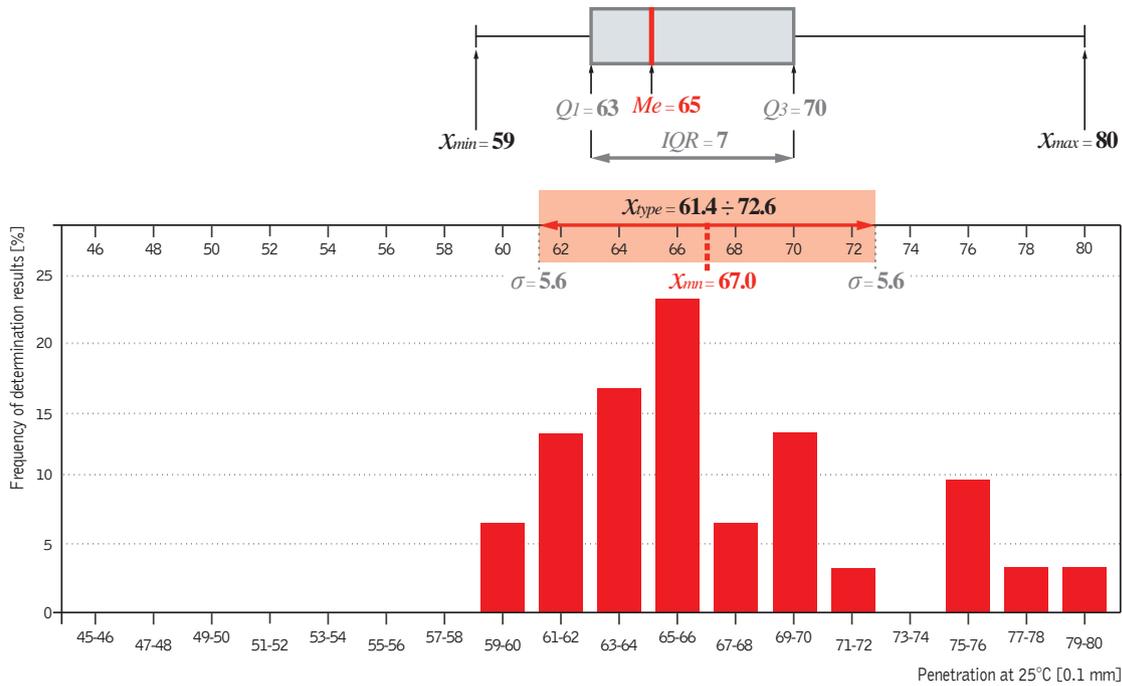


Fig. 2.13. Histogram and box plot presenting the results of penetration determinations at 25°C of highly modified bitumen **ORBITON 45/80-80 HiMA** produced in the 2018–2020 period (standard range: 45–80 [0.1 mm])

2.2.2. SOFTENING POINT

The softening point is the basic parameter determining bitumen properties at high service temperatures and represents a conventional approximate upper limit of the viscoelastic consistency.

The aim of the test is to determine the “conventional” temperature at which the bitumen reaches a given consistency. Testing the bitumen softening point is usually carried out using the “Ring and Ball” method in accordance with EN 1427.

Statistical parameters of the results of the softening point determination for bitumen produced in the 2018–2020 period are listed in Table 2.3.

Additionally, Figures 2.15. to 2.19. present histograms with box plots for softening point results of selected bitumens produced in the 2018–2020 period.

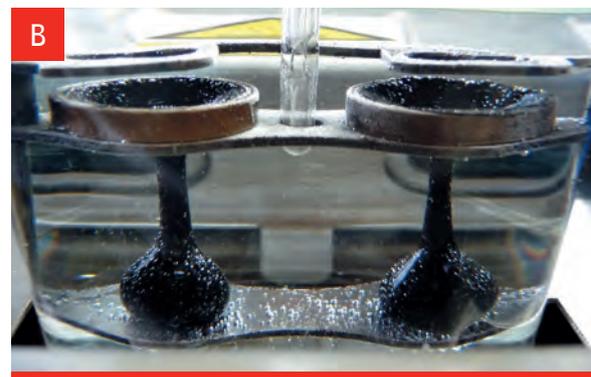
Table 2.3.

Statistical parameters of the softening point determination results for bitumens produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT** [°C] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUANTILE | THIRD QUANTILE | MIN-MAX VALUE |
|------------------------|--------------------|-----------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | \bar{x}_{min} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| 20/30 | 55 ÷ 63 | 62.3 | 0.6 | 61.7 ÷ 62.9 | 62.4 | 61.8 | 62.8 | 60.8 ÷ 63.0 |
| 35/50 | 50 ÷ 58 | 53.8 | 0.7 | 53.1 ÷ 54.5 | 53.8 | 53.4 | 54.0 | 52.4 ÷ 55.4 |
| 50/70 | 46 ÷ 54 | 48.9 | 1.3 | 47.6 ÷ 50.2 | 48.8 | 48.0 | 49.6 | 47.0 ÷ 53.8 |
| 70/100 | 43 ÷ 51 | 44.9 | 1.4 | 43.5 ÷ 46.3 | 44.6 | 44.2 | 45.0 | 43.6 ÷ 50.8 |
| 100/150 | 39 ÷ 47 | 41.7 | 0.7 | 41.0 ÷ 42.4 | 41.8 | 41.2 | 42.0 | 40.2 ÷ 43.2 |
| 160/220 | 35 ÷ 43 | 38.5 | 0.6 | 37.9 ÷ 39.1 | 38.6 | 38.2 | 38.8 | 37.0 ÷ 39.8 |
| ORBITON 25/55-60 | ≥ 60 | 64.9 | 1.4 | 63.5 ÷ 66.3 | 65.0 | 64.0 | 66.0 | 61.8 ÷ 67.0 |
| ORBITON 45/80-55 | ≥ 55 | 63.7 | 4.9 | 58.8 ÷ 68.6 | 62.4 | 61.2 | 65.6 | 56.8 ÷ 75.2 |
| ORBITON 45/80-65 | ≥ 65 | 76.2 | 5.3 | 70.9 ÷ 81.5 | 75.7 | 72.4 | 79.8 | 66.6 ÷ 88.0 |
| ORBITON 25/55-80 HiMA | ≥ 80 | 95.2 | 3.3 | 91.9 ÷ 98.5 | 94.5 | 93.0 | 99.0 | 89.0 ÷ 101.0 |
| ORBITON 45/80-80 HiMA | ≥ 80 | 92.6 | 3.1 | 89.5 ÷ 95.7 | 92.5 | 91.0 | 95.5 | 83.0 ÷ 97.5 |
| ORBITON 65/105-80 HiMA | ≥ 80 | 90.5 | 2.6 | 87.9 ÷ 93.1 | 90.0 | 88.5 | 92.5 | 86.5 ÷ 94.0 |

* test results refer to the period: January 2018–April 2020

** for paving grade bitumen according to EN 12591; for ORBITON and ORBITON HiMA according to EN 14023


Fig. 2.14.

General view of a bitumen sample before (A) and after (B) performing a softening point test with the R&B method (photo by ORLEN Asphalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

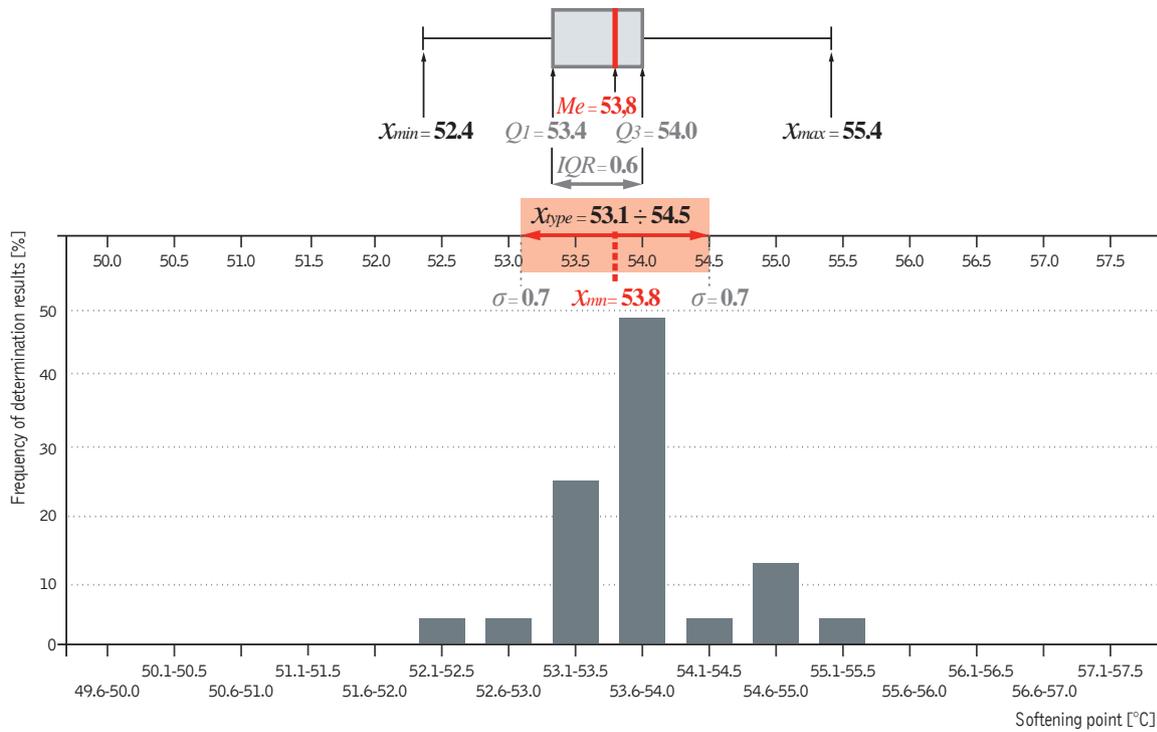


Fig. 2.15. Histogram and box plot presenting the results of softening point R&B determinations of **paving grade bitumen 35/50** produced in the 2018–2020 period (standard range: 50–58 [°C])

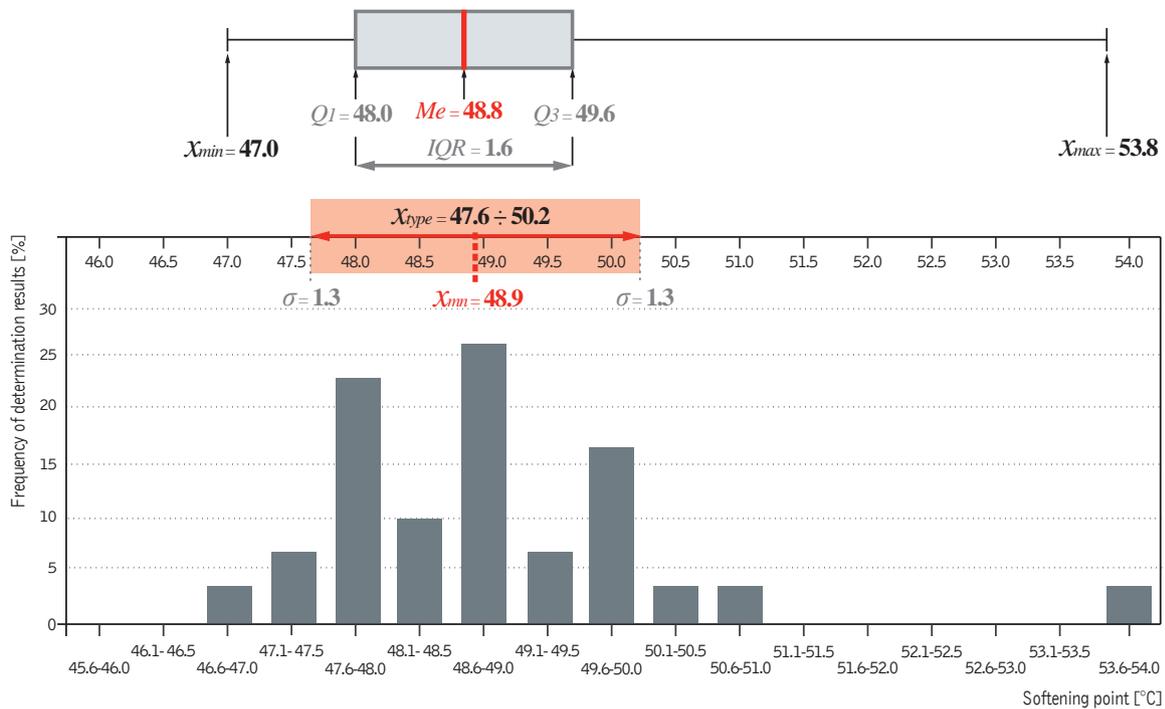


Fig. 2.16. Histogram and box plot presenting the results of softening point R&B determinations of **paving grade bitumen 50/70** produced in the 2018–2020 period (standard range: 46–54 [°C])

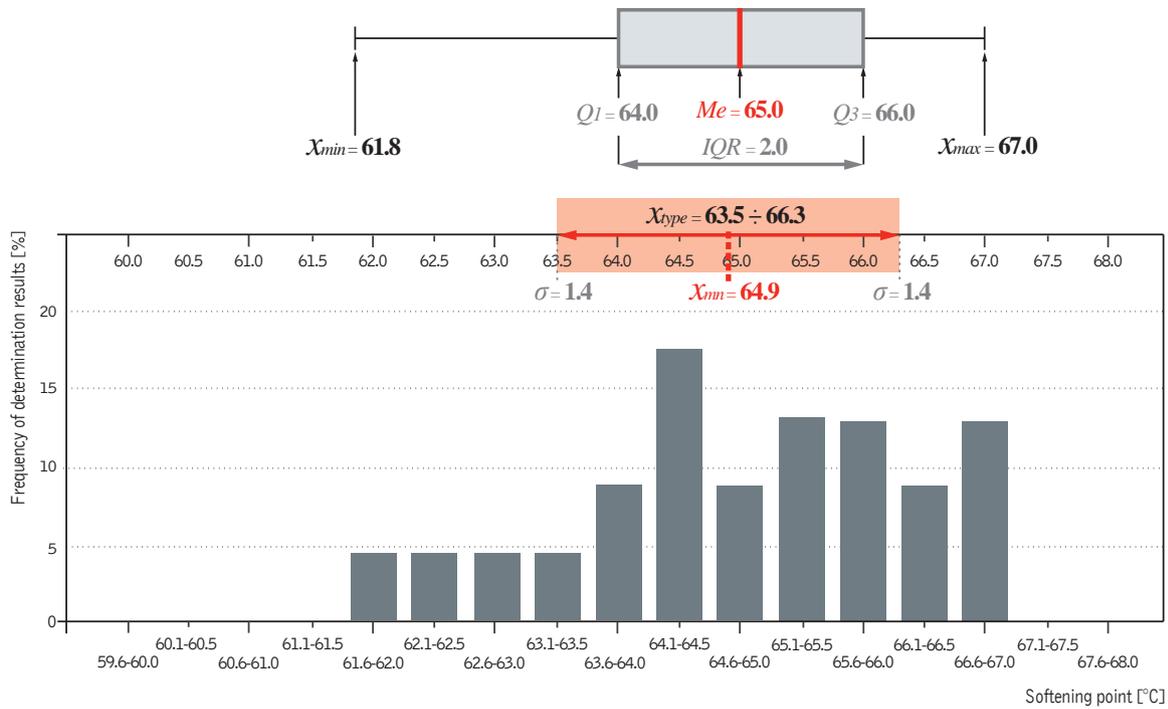


Fig. 2.17. Histogram and box plot presenting the results of softening point R&B determinations of polymer modified bitumen **ORBITON 25/55-60** produced in the 2018–2020 period (standard range: ≥ 60 [°C])

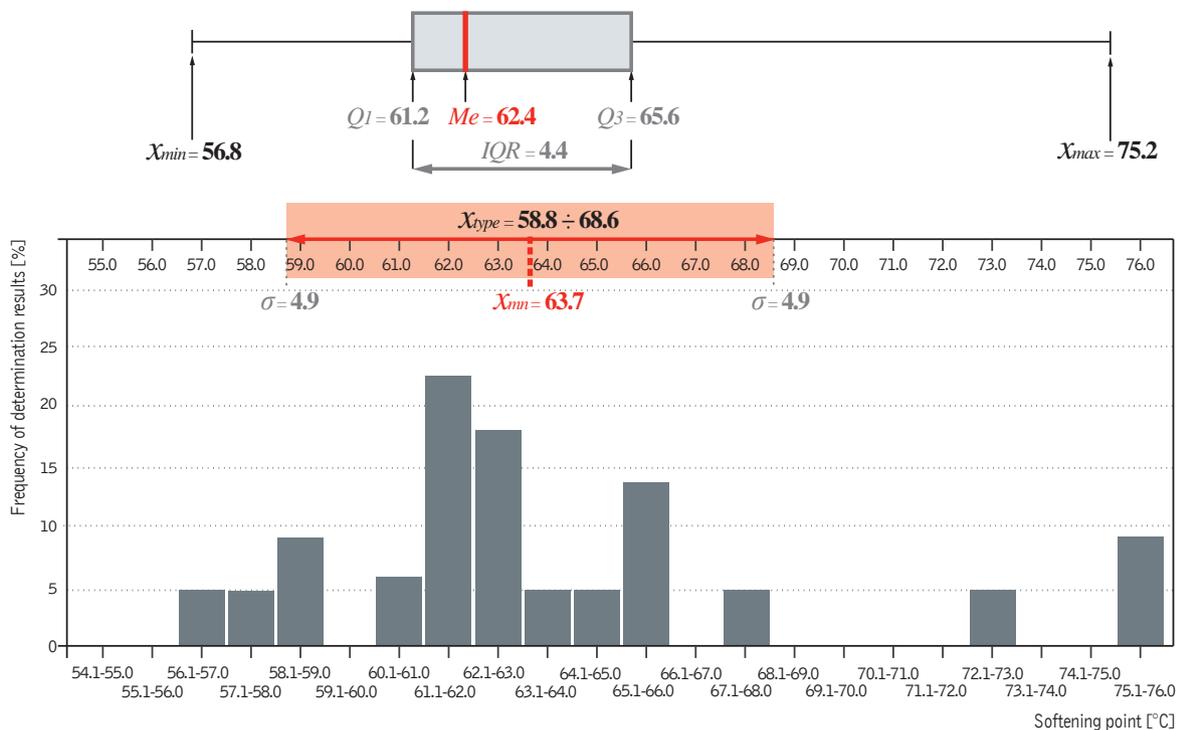


Fig. 2.18. Histogram and box plot presenting the results of softening point R&B determinations of polymer modified bitumen **ORBITON 45/80-55** produced in the 2018–2020 period (standard range: ≥ 55 [°C])

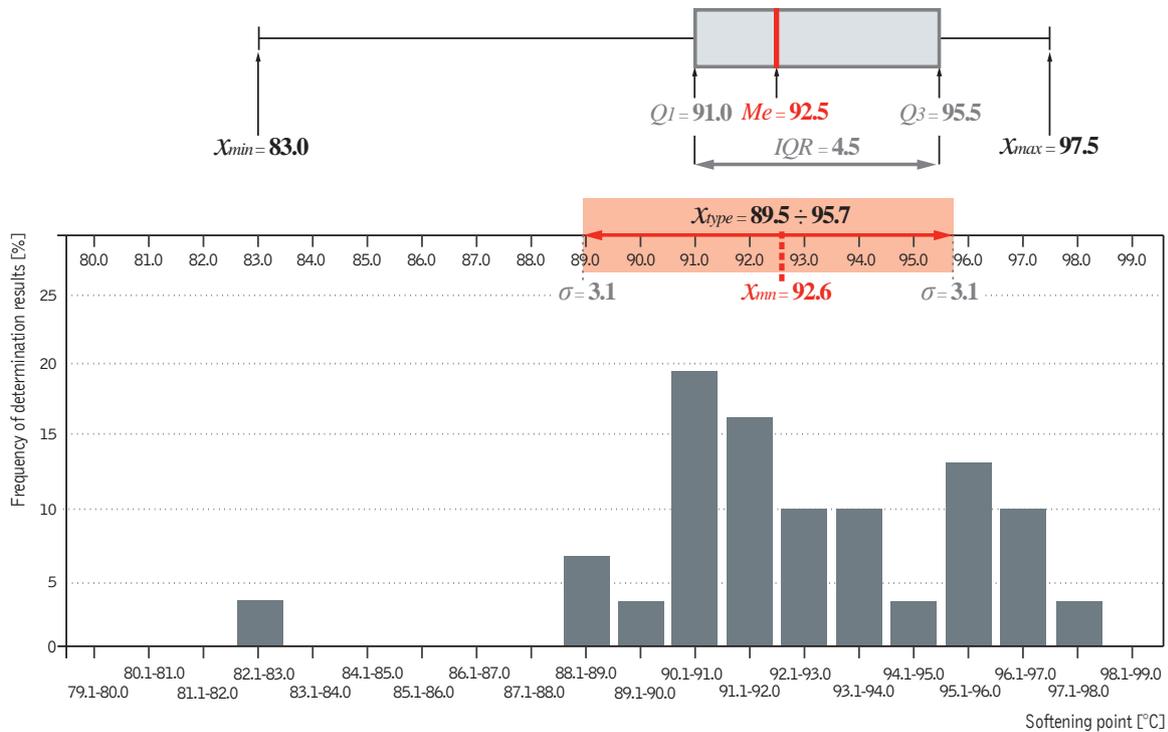


Fig. 2.19. Histogram and box plot presenting the results of softening point R&B determinations of highly modified bitumen **ORBITON 45/80-80 HiMA** produced in the 2018–2020 period (standard range: ≥ 80 °C)

2.2.3. FRAASS BREAKING POINT

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

The Fraass breaking point test is carried out in accordance with EN 12593 standard.

Fig. 2.20. shows pictures of the apparatus used to measure the Fraass breaking point.

Table 2.4. presents statistical parameters of the results of the Fraass breaking point for bitumens produced in the 2018–2020 period.

Additionally, Figures 2.21.–2.25. show histograms with box plots for Fraass breaking point results of selected bitumens produced in the 2018–2020 period.

Table 2.4.

Statistical parameters of Fraass breaking point determination results for bitumens produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT** [°C] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUARTILE | THIRD QUARTILE | MIN-MAX VALUE |
|------------------------|--------------------|----------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | \bar{x}_{mn} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| 20/30 | NR | -9.6 | 1.4 | -11.0 ÷ -8.2 | -10 | -10 | -9 | -12 ÷ -7 |
| 35/50 | ≤ -5 | -12.6 | 1.5 | -14.1 ÷ -11.1 | -13 | -14 | -12 | -15 ÷ -11 |
| 50/70 | ≤ -8 | -14.8 | 2.1 | -16.9 ÷ -12.7 | -14 | -16 | -14 | -18 ÷ -12 |
| 70/100 | ≤ -10 | -16.2 | 1.7 | -17.9 ÷ -14.5 | -16 | -16 | -16 | -21 ÷ -13 |
| 100/150 | ≤ -12 | -16.0 | 1.4 | -17.4 ÷ -14.6 | -16 | -17 | -15 | -18 ÷ -14 |
| 160/220 | ≤ -15 | -17.2 | 1.1 | -18.3 ÷ -16.1 | -17 | -18 | -16 | -19 ÷ -15 |
| ORBITON 25/55-60 | ≤ -10 | -14.8 | 2.2 | -17.0 ÷ -12.6 | -14 | -16 | -13 | -21 ÷ -12 |
| ORBITON 45/80-55 | ≤ -15 | -17.7 | 1.8 | -19.5 ÷ -15.9 | -17 | -19 | -16 | -22 ÷ -15 |
| ORBITON 45/80-65 | ≤ -15 | -17.5 | 1.2 | -18.7 ÷ -16.3 | -18 | -18 | -17 | -20 ÷ -16 |
| ORBITON 25/55-80 HiMA | ≤ -15 | -21.2 | 1.1 | -22.3 ÷ -20.1 | -22 | -22 | -20 | -22 ÷ -20 |
| ORBITON 45/80-80 HiMA | ≤ -18 | -20.9 | 1.3 | -22.2 ÷ -19.6 | -21 | -22 | -20 | -22 ÷ -19 |
| ORBITON 65/105-80 HiMA | ≤ -18 | -21.2 | 1.3 | -22.5 ÷ -19.9 | -21 | -22 | -20 | -23 ÷ -20 |

* test results refer to the period: January 2018–April 2020

** for paving grade bitumen according to EN 12591; for ORBITON and ORBITON HiMA according to EN 14023

NR – No Requirement


Fig. 2.20.

(A) General view of the Fraass breaking point apparatus and (B) view of a bitumen sample on the plate after the test (photo by ORLEN Asphalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

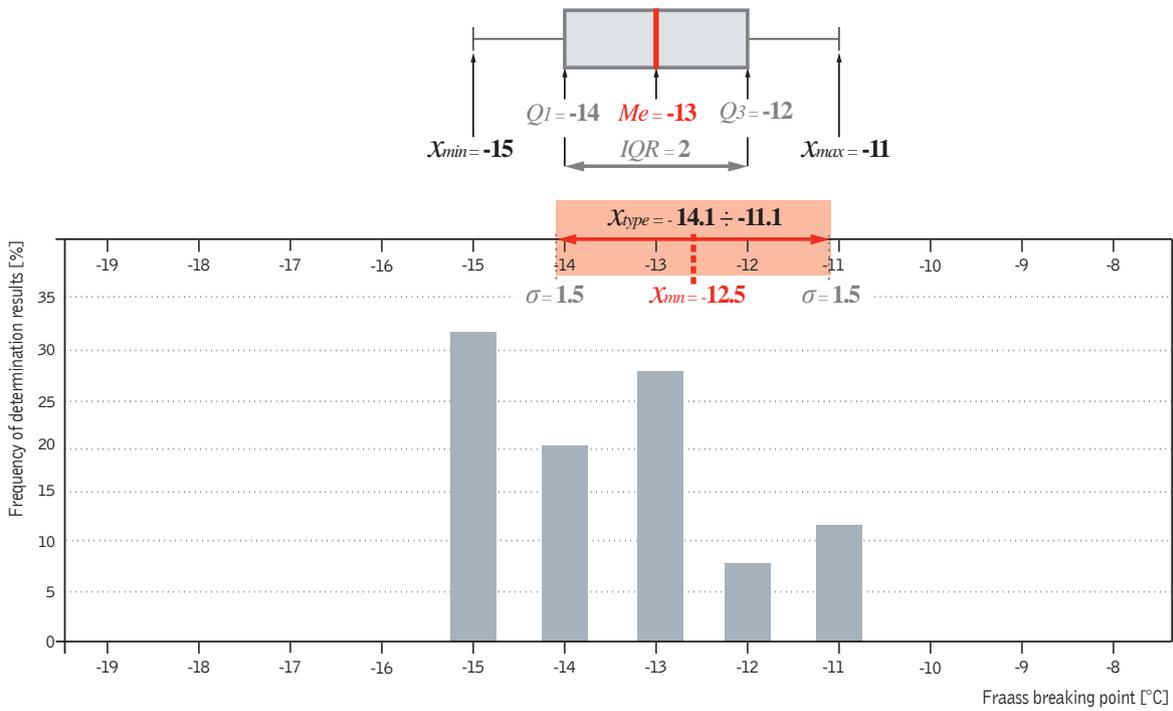


Fig. 2.21. Histogram and box plot presenting the results of Fraass breaking point determinations of **paving grade bitumen 35/50** produced in the 2018–2020 period (standard range: ≤ -5 [°C])

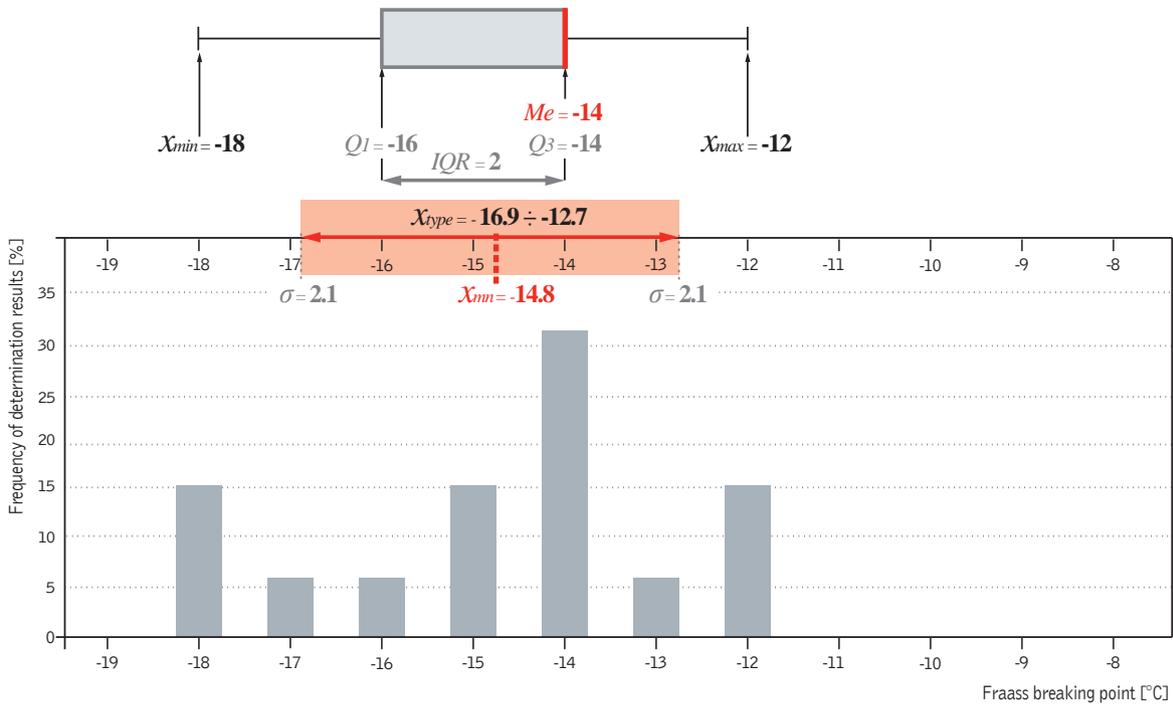


Fig. 2.22. Histogram and box plot presenting the results of Fraass breaking point determinations of **paving grade bitumen 50/70** produced in the 2018–2020 period (standard range: ≤ -8 [°C])

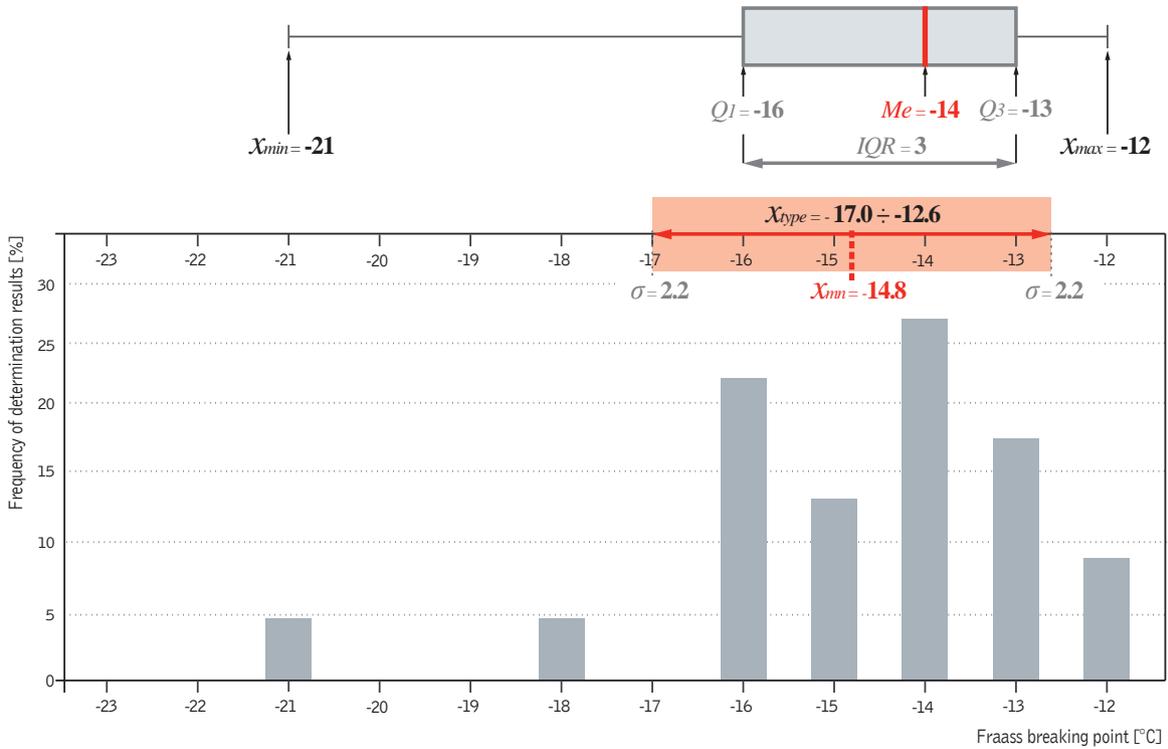


Fig. 2.23. Histogram and box plot presenting the results of Fraass breaking point determinations of polymer modified bitumen **ORBITON 25/55-60** produced in the 2018–2020 period (standard range: ≤ -10 [°C])

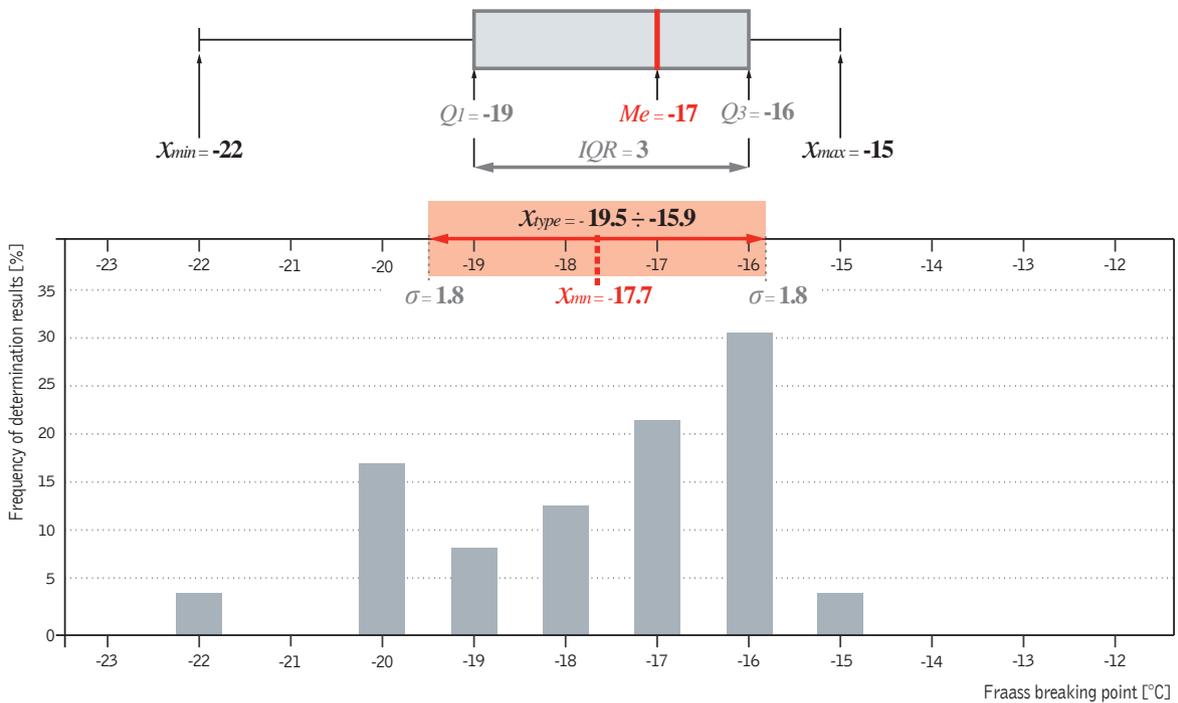


Fig. 2.24. Histogram and box plot presenting the results of Fraass breaking point determinations of polymer modified bitumen **ORBITON 45/80-55** produced in the 2018–2020 period (standard range: ≤ -15 [°C])

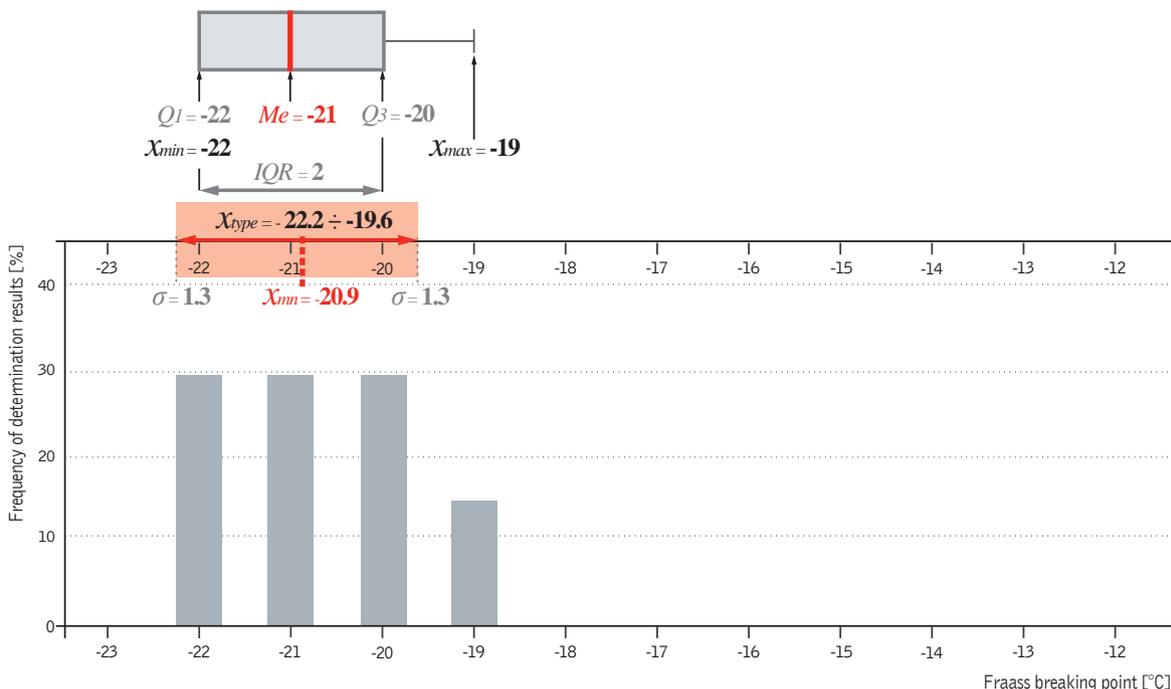


Fig. 2.25.

Histogram and box plot presenting the results of Fraass breaking point determinations of highly modified bitumen **ORBITON 45/80-80 HiMA** produced in the 2018–2020 period (standard range: ≤ -18 [°C])

2.2.4. VISCOSITY

Viscosity is one of the key technological and functional parameters of bituminous binders. There are several definitions and test methods regarding viscosity. In relation to bitumen, the concept of viscosity can be defined as the internal friction occurring between the particles when one layer of bitumen is displaced relative to another [15].

Bitumens are treated as liquids with complex rheological characteristics. Their viscosity can vary depending on: temperature, shear rate, test duration, type of method or measurement system used in a given method. In other words, **this means that the comparability of viscosity results obtained with different methods can only be maintained if strictly defined measuring conditions are met (appropriate temperature, measuring systems, shear rate, testing time, etc.).**

In other cases, comparison and substitution of viscosity results is incorrect and may lead to erroneous conclusions.

The higher the temperature of the bitumen, the lower the viscosity [15]. This relation can be used to establish viscosity–temperature characteristics and determine the temperature of pumping of the bitumen, coating the aggregate and compacting the pavement. However, in the case of polymer modified bitumen and highly modified bitumen HiMA, taking into account their atypical features resulting from the specific properties of the used polymer, adopting the viscosity–temperature relation for precise determination of process temperature does not seem appropriate. The temperatures determined in this way are largely approximate.

For more information on process temperature, see Chapter 5.

Dynamic viscosity testing can be performed using:

- Cannon-Manning vacuum capillary method, according to EN 12596;
- Rheometer, by cone and plate method according to EN 13702-1;
- Brookfield's rotational viscometer, according to EN 13302 or ASTM D 4402.

The kinematic viscosity test is performed with a BS/IP/RF type viscometer in accordance with EN 12595.

Figures 2.27. to 2.38. show the average values of viscosity obtained for bitumens produced in the 2018–2020 period, performed with a Brookfield's rotational viscometer (Fig. 2.26.) according to EN 13302. For soft paving grade bitumens used primarily for the production of bituminous emulsions (using the "cold" technology), average viscosity values were given only before RTFOT ageing. For binders used in the "hot" technology, both relations – before and after RTFOT ageing – are presented.

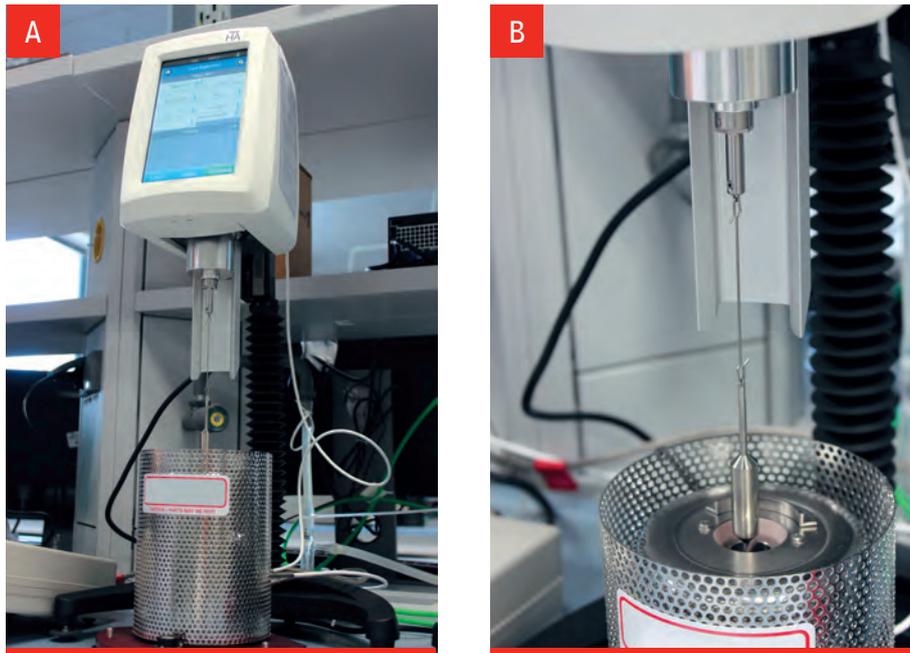


Fig. 2.26. (A) General view of Brookfield's viscometer and (B) close-up of spindle and thermostated container for a bitumen sample (photo by ORLEN Asphalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

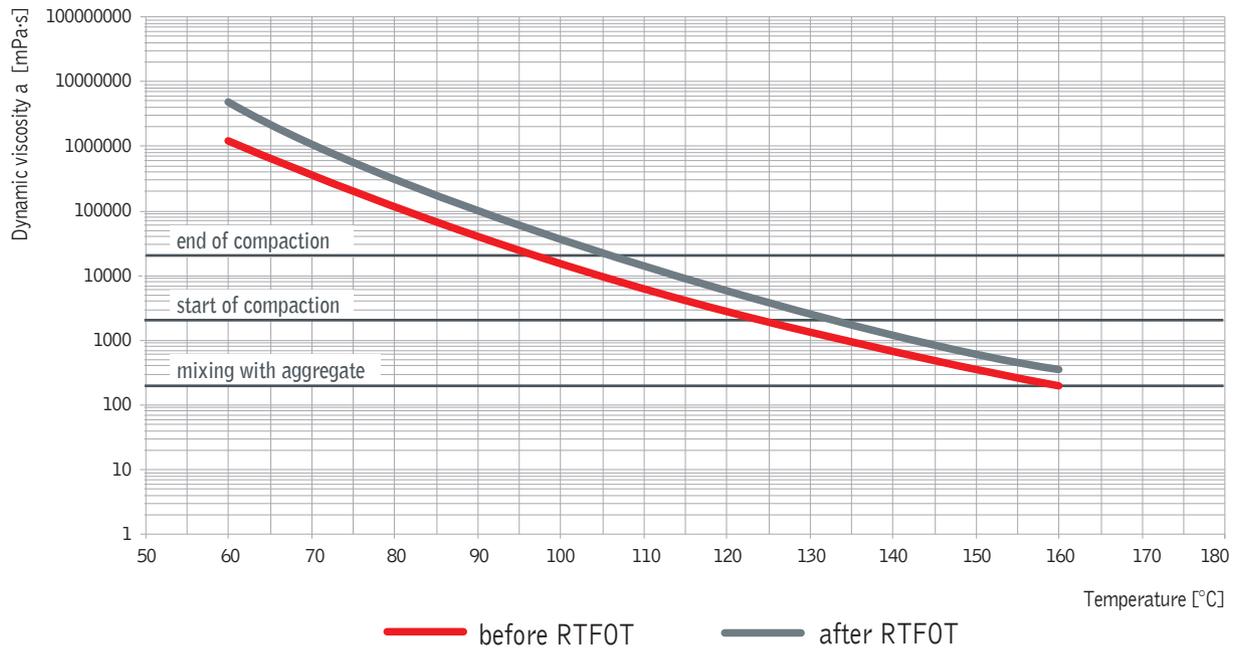


Fig. 2.27. Relation between viscosity and temperature for **paving grade bitumen 20/30** produced in the 2018–2020 period

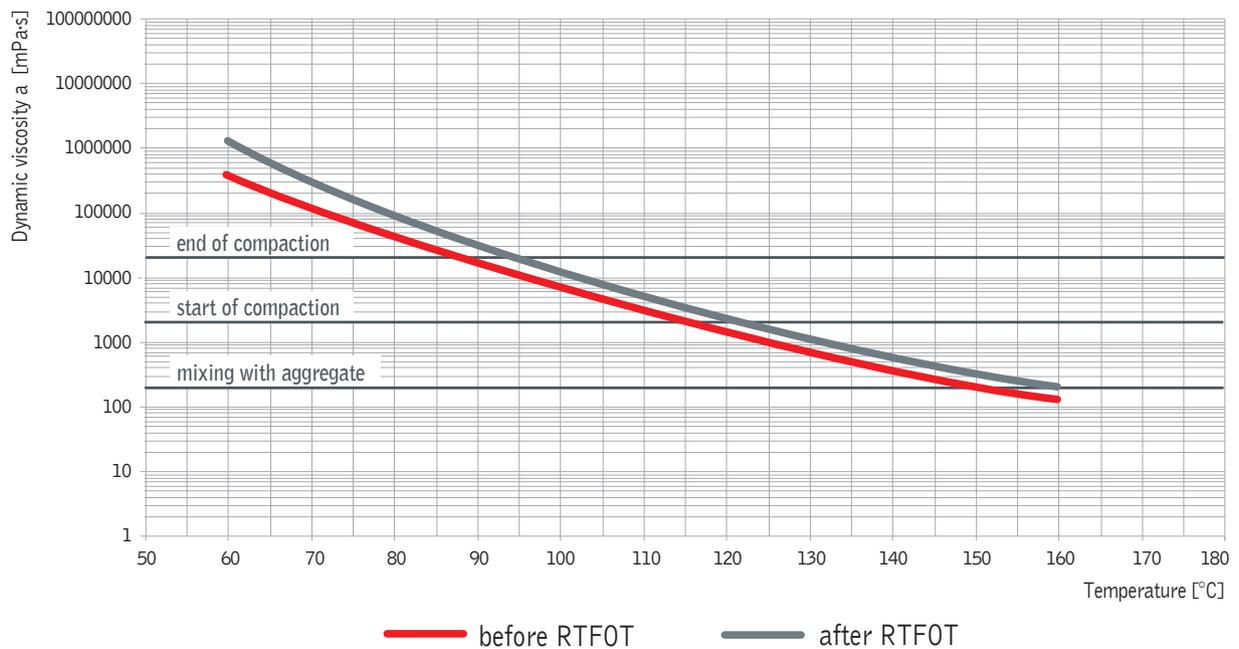


Fig. 2.28. Relation between viscosity and temperature for **paving grade bitumen 35/50** produced in the 2018–2020 period

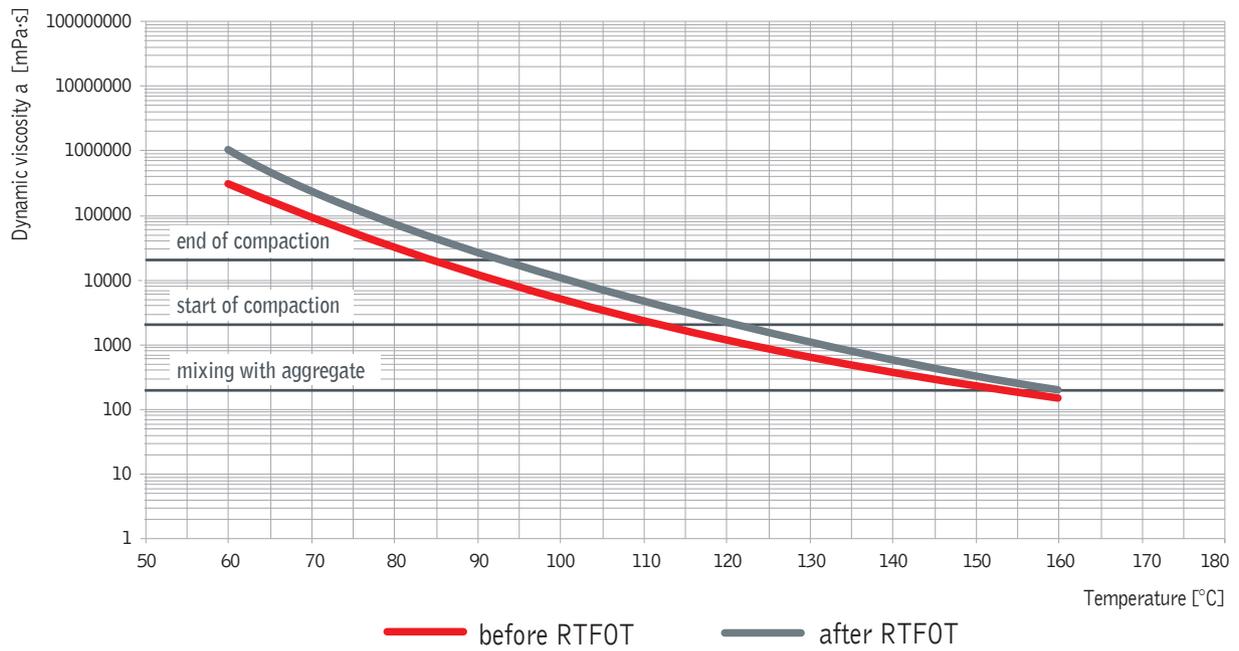


Fig. 2.29. Relation between viscosity and temperature for **paving grade bitumen 50/70** produced in the 2018–2020 period

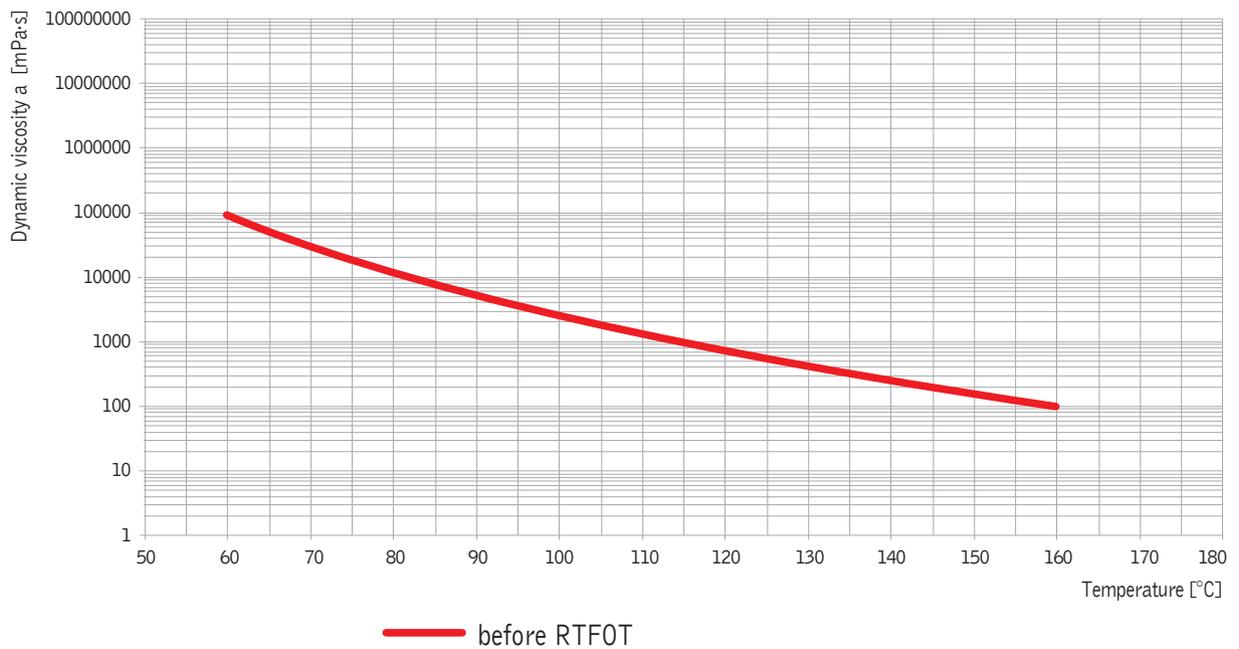


Fig. 2.30. Relation between viscosity and temperature for **paving grade bitumen 70/100** produced in the 2018–2020 period

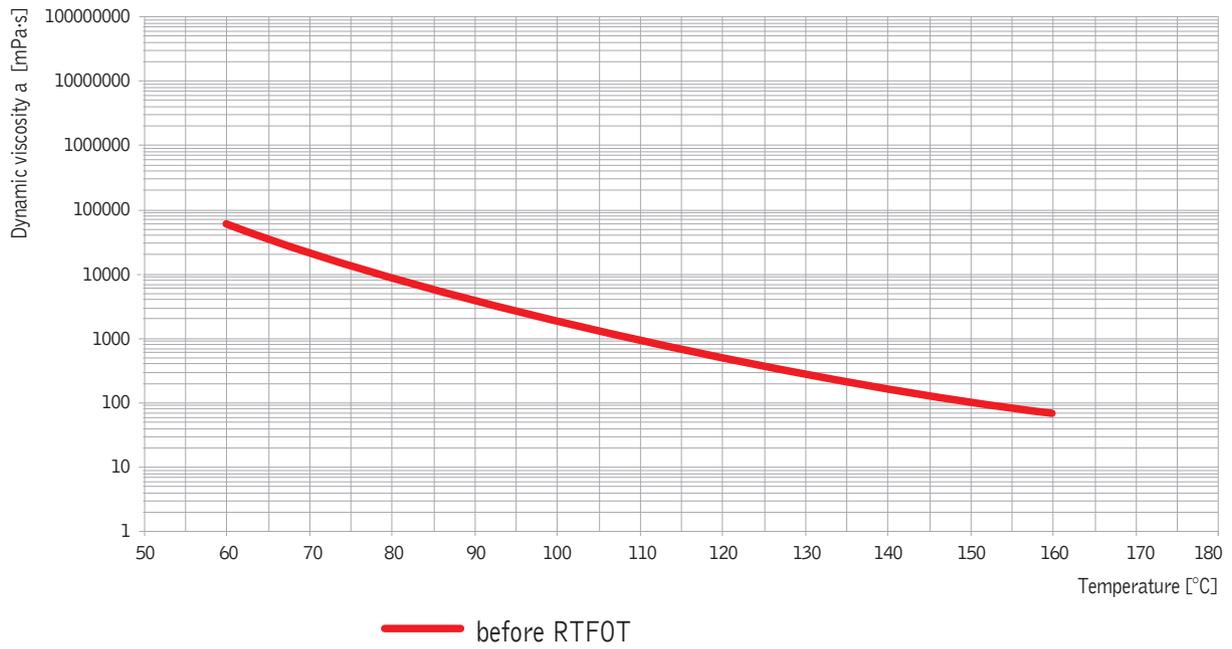


Fig. 2.31. Relation between viscosity and temperature for **paving grade bitumen 100/150** produced in the 2018–2020 period

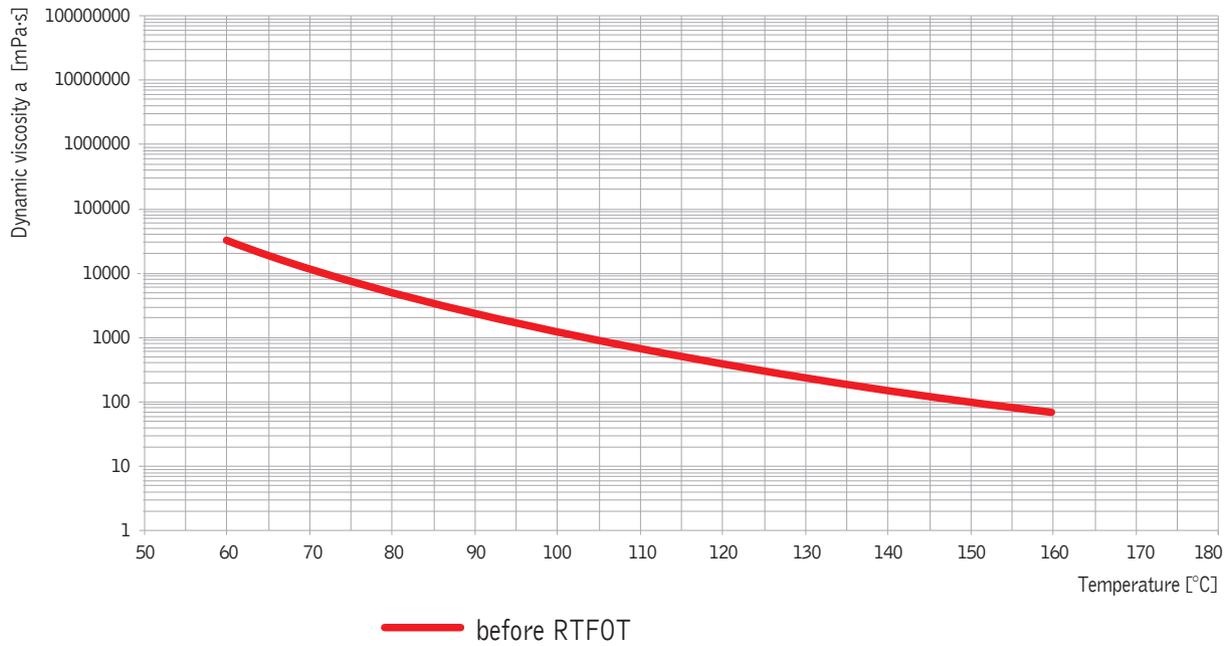


Fig. 2.32. Relation between viscosity and temperature for **paving grade bitumen 160/220** produced in the 2018–2020 period

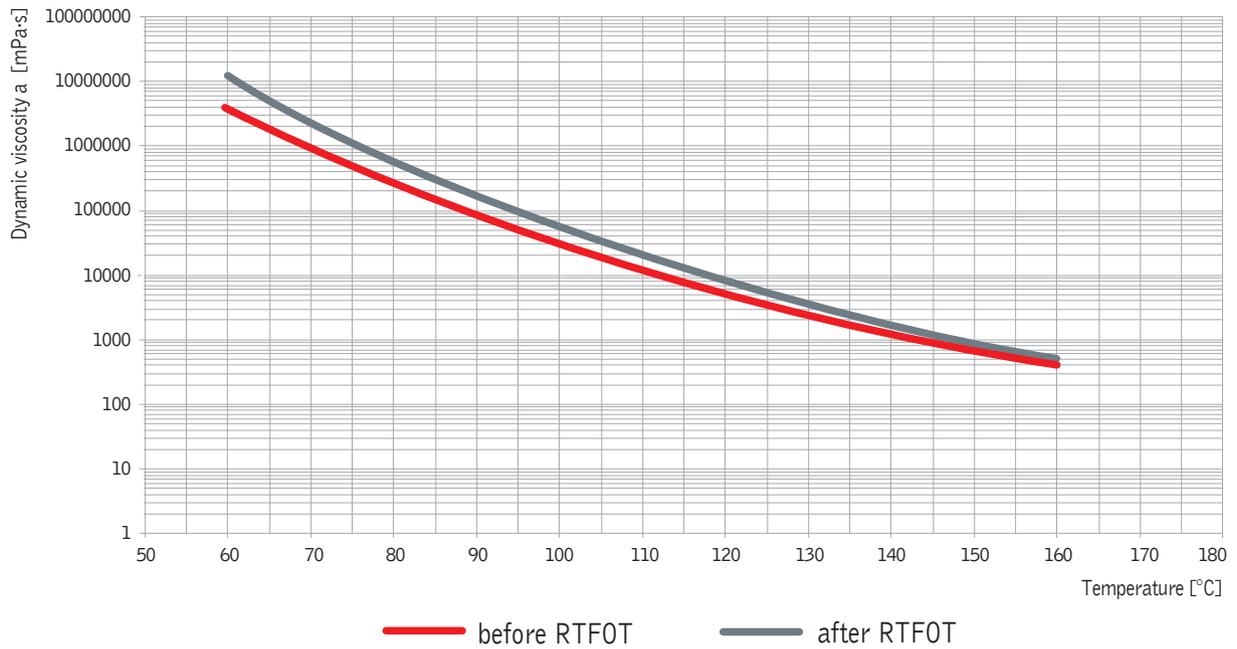


Fig. 2.33. Relation between viscosity and temperature for **polymer modified bitumen ORBITON 25/55-60** produced in the 2018–2020 period

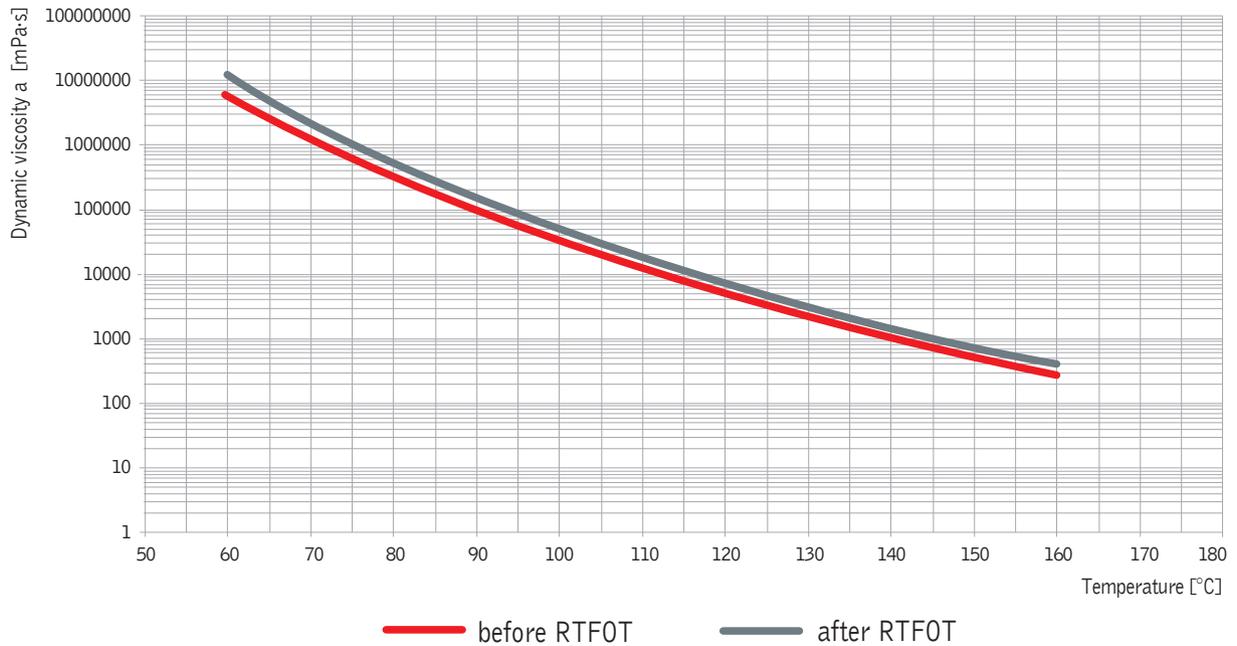


Fig. 2.34. Relation between viscosity and temperature for **polymer modified bitumen ORBITON 45/80-55** produced in the 2018–2020 period

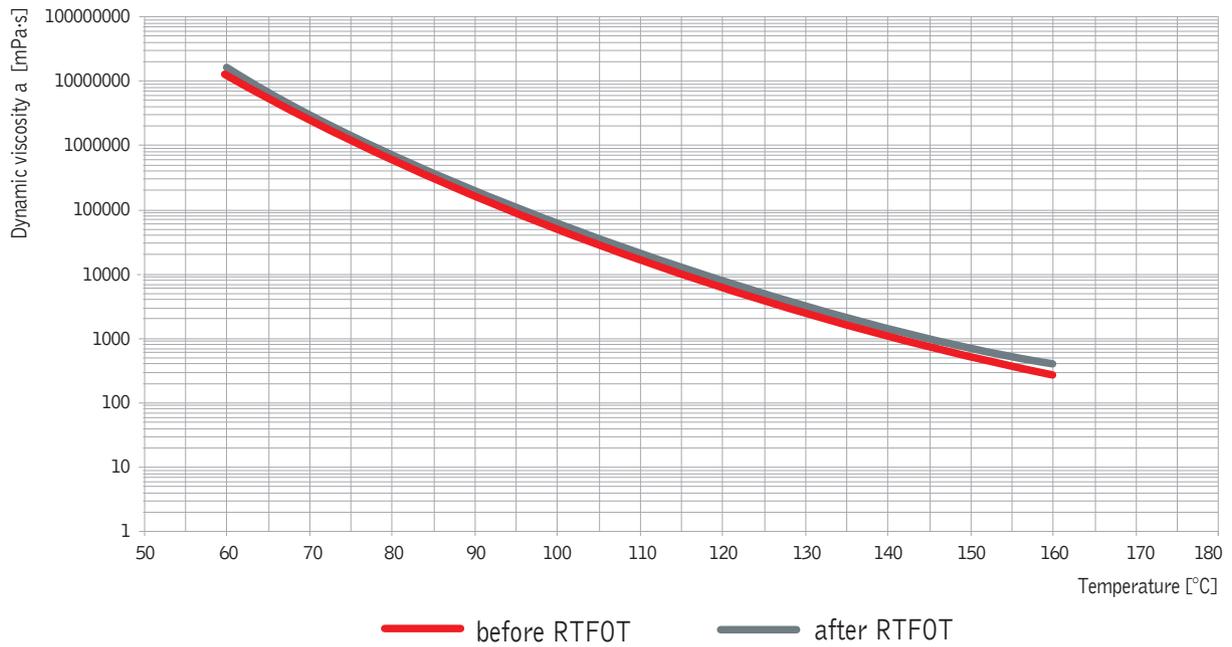


Fig. 2.35. Relation between viscosity and temperature for **polymer modified bitumen ORBITON 45/80-65** produced in the 2018–2020 period

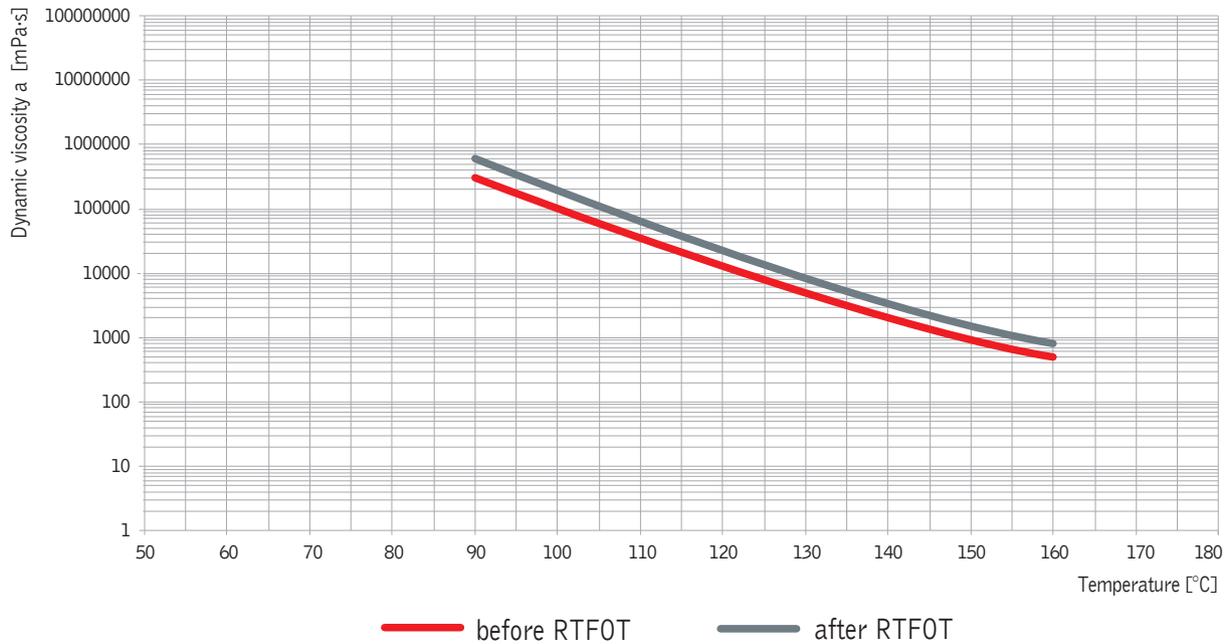


Fig. 2.36. Relation between viscosity and temperature for **highly modified bitumen ORBITON 25/55-80** produced in the 2018–2020 period

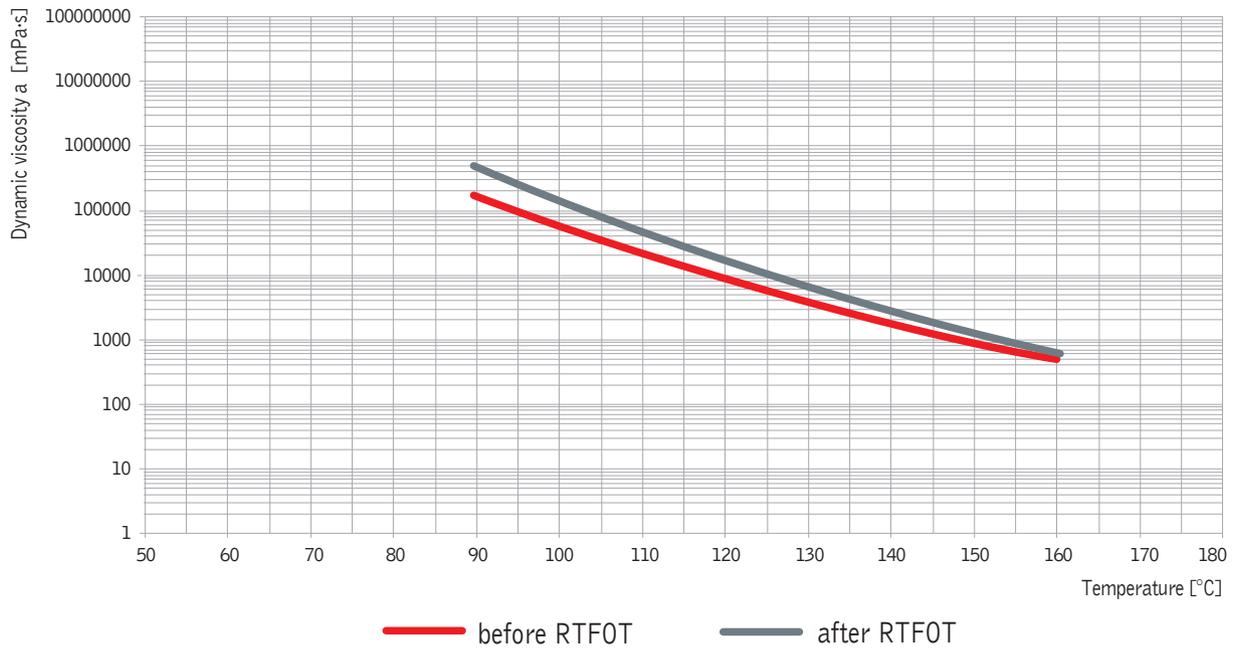


Fig. 2.37. Relation between viscosity and temperature for **highly modified bitumen ORBITON 45/80-80** produced in the 2018–2020 period

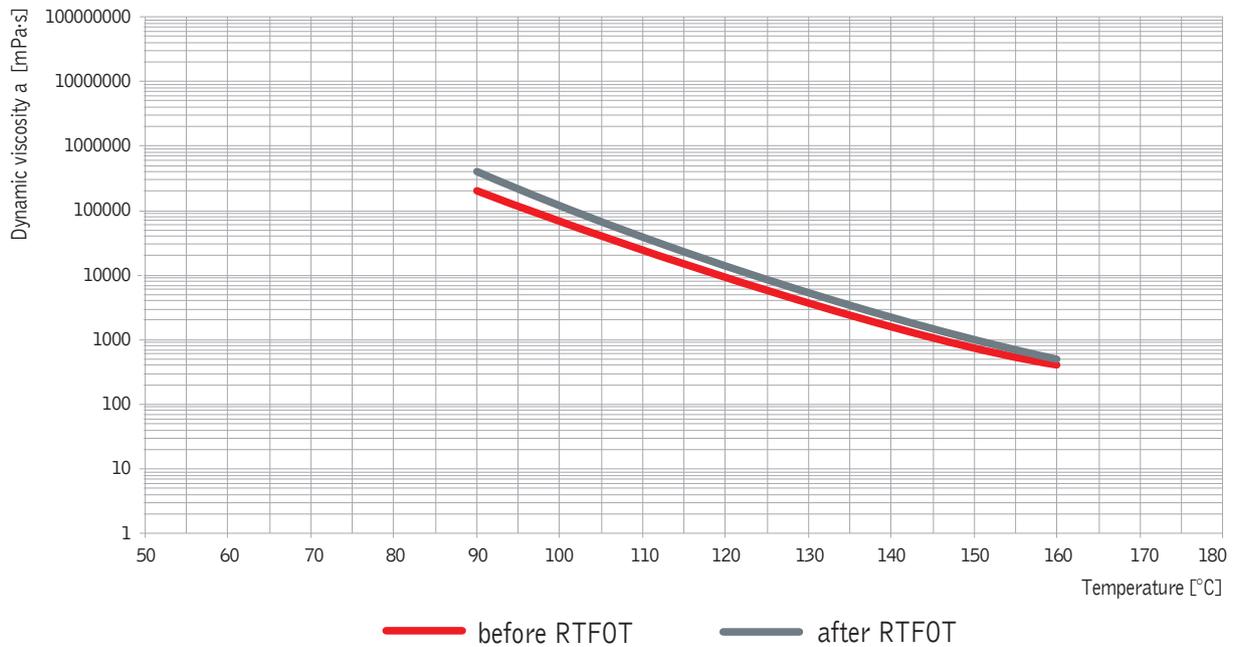


Fig. 2.38. Relation between viscosity and temperature for **highly modified bitumen ORBITON 65/105-80** produced in the 2018–2020 period

Table 2.5. shows an example of the viscosity test results for bitumens produced in the 2018–2020 period.

Table 2.5.

Example results of testing the viscosity of bitumens produced in the 2018–2020 period

| TYPE OF BINDER | DYNAMIC VISCOSITY ACC. TO EN 13302 [Pa·s] | | | | | | | | KINEMATIC VISCOSITY ACC. TO EN 12595 [mm ² /s] |
|------------------------|---|--------|-------|-------|-------------|--------|-------|-------|---|
| | BEFORE RTFOT | | | | AFTER RTFOT | | | | |
| | 60°C | 90°C | 135°C | 160°C | 60°C | 90°C | 135°C | 160°C | |
| 20/30 | 3 940.00 | 56.25 | 1.42 | 0.39 | 12 300.00 | 131.00 | 2.28 | 0.56 | 1 502 |
| 35/50 | 740.00 | 20.80 | 0.78 | 0.24 | 2 750.00 | 45.40 | 1.10 | 0.32 | 792 |
| 50/70 | 211.00 | 8.21 | 0.43 | 0.15 | 511.00 | 15.08 | 0.60 | 0.20 | 491 |
| 70/100 | 154.00 | 6.81 | 0.39 | 0.14 | – | – | – | – | 362 |
| 100/150 | 83.00 | 4.00 | 0.26 | 0.10 | – | – | – | – | 275 |
| 160/220 | 41.92 | 2.64 | 0.20 | 0.08 | – | – | – | – | 226 |
| ORBITON 25/55-60 | 6 450.00 | 89.00 | 2.17 | 0.62 | 17 400.00 | 196.00 | 3.18 | 0.79 | – |
| ORBITON 45/80-55 | 4 633.00 | 34.45 | 1.18 | 0.40 | 7 480.00 | 57.00 | 1.48 | 0.47 | – |
| ORBITON 45/80-65 | 1 656.00 | 31.80 | 1.61 | 0.58 | 34 200.00 | 79.50 | 2.28 | 0.63 | – |
| ORBITON 25/55-80 HiMA | – | 338.00 | 3.39 | 0.84 | – | 394.00 | 3.53 | 0.85 | – |
| ORBITON 45/80-80 HiMA | – | 187.00 | 2.20 | 0.53 | – | 511.00 | 3.33 | 0.79 | – |
| ORBITON 65/105-80 HiMA | – | 274.00 | 2.03 | 0.51 | – | 508.00 | 2.80 | 0.60 | – |

2.2.5. RESISTANCE TO AGEING

As a result of the ageing process, the properties of bituminous binders change in the following way:

- penetration reduction,
- softening point increase,
- increase (deterioration) of Fraass breaking point,
- increase of viscosity,
- reduction of ductility,
- several other chemical and mechanical changes.

The most intensive ageing of bitumen occurs when it is mixed with the aggregate in the plant's mixer, when the process

temperature is the highest and the layer of binder on the aggregate is the thinnest.

It must be remembered that the bitumen built into the pavement is binder after short-term (technologically) ageing. From the perspective of pavement durability, it is therefore desirable to test to what extent the properties of a given binder have changed under the influence of high temperature, i.e. penetration, softening point, viscosity or elastic recovery in the case of polymer modified bitumens. Resistance to short-term ageing by RTFOT is determined in accordance with standard 12607-1.

Fig. 2.39. shows an oven for simulation of short-term ageing using the RTFOT method.

2.2.5.1. RETAINED PENETRATION AFTER HARDENING

As a result of the ageing process, the bitumen penetration decreases, which means that the bitumen hardens. The result of the retained penetration after ageing is calculated as the percentage of penetration of the bitumen after RTFOT in relation to the original penetration value of the bitumen before ageing (taking the penetration of fresh bitumen as 100%). Data on the retained penetration is provided in Table 2.6.



Fig. 2.39. General view of the oven for short-term ageing by RTFOT method (photo by ORLEN Asphalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

Table 2.6.

Statistical parameters of the determinations results of resistance to ageing – retained penetration [%], for bitumens produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT** [%] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUANTILE | THIRD QUANTILE | MIN-MAX VALUE |
|------------------------|-------------------|------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | x_{mm} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| 20/30 | ≥ 55 | 75.3 | 3.7 | 71.6 ÷ 79.0 | 75 | 73 | 79 | 69 ÷ 82 |
| 35/50 | ≥ 53 | 69.7 | 4.6 | 65.1 ÷ 74.3 | 71 | 67 | 73 | 59 ÷ 77 |
| 50/70 | ≥ 50 | 67.6 | 9.4 | 58.2 ÷ 77.0 | 66 | 61 | 70 | 52 ÷ 87 |
| 70/100 | ≥ 46 | 63.7 | 7.0 | 56.7 ÷ 70.7 | 63 | 60 | 65 | 51 ÷ 84 |
| 100/150 | ≥ 43 | 57.8 | 3.4 | 54.4 ÷ 61.2 | 58 | 55 | 61 | 51 ÷ 63 |
| 160/220 | ≥ 37 | 55.0 | 4.9 | 50.1 ÷ 59.9 | 55 | 52 | 59 | 47 ÷ 67 |
| ORBITON 25/55-60 | ≥ 60 | 73.2 | 5.2 | 68.0 ÷ 78.4 | 74 | 70 | 77 | 62 ÷ 82 |
| ORBITON 45/80-55 | ≥ 60 | 69.5 | 6.0 | 63.5 ÷ 75.5 | 69 | 64 | 73 | 61 ÷ 87 |
| ORBITON 45/80-65 | ≥ 60 | 73.4 | 6.5 | 66.9 ÷ 79.9 | 74 | 70 | 77 | 63 ÷ 88 |
| ORBITON 25/55-80 HiMA | ≥ 60 | 78.1 | 8.0 | 70.1 ÷ 86.1 | 82 | 70 | 86 | 67 ÷ 86 |
| ORBITON 45/80-80 HiMA | ≥ 60 | 79.1 | 7.1 | 72.0 ÷ 86.2 | 80 | 76 | 86 | 66 ÷ 88 |
| ORBITON 65/105-80 HiMA | ≥ 60 | 77.9 | 7.1 | 70.8 ÷ 85.0 | 78 | 72 | 84 | 70 ÷ 88 |

* test results refer to the period: January 2018–April 2020

** for paving grade bitumen according to EN 12591; for ORBITON and ORBITON HiMA according to EN 14023

2.2.5.2. INCREASE AND DROP IN SOFTENING POINT AFTER HARDENING

The bitumens softening point usually increases after short-term ageing. The bitumen sample after the RTFOT test is subjected to the softening point test according to EN 1427. Then, the result of the increase in the softening point after ageing is calculated in [°C] from the difference in the softening temperature obtained for the RTFOT-aged bitumen sample and the unaged bitumen sample, respectively. The requirements limiting the increase in softening point after ageing apply to each type of bitumen used in hot-mix technologies: paving grade, polymer modified, and highly modified HiMA. However, sometimes softening

point RTFOT ageing decreases. This may be the case with some polymer modified and highly modified bitumens. The requirement for a drop in softening point after RTFOT ageing is specified in National Annex NA of the amendment of PN-EN 14023:2011/Ap2, published in 2020, as a TBR value (to be reported).

Table 2.7 presents statistical data on the change in softening point (increase and decrease) under RTFOT ageing for the tested binders.

Table 2.7.

Statistical parameters of the results of the resistance of hardening determination – change in softening point R&B [°C], for bitumens produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT** [°C] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUANTILE | THIRD QUANTILE | MIN-MAX VALUE |
|------------------------|--------------------|------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | x_{min} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| 20/30 | ≤ 8 | 6.8 | 1.3 | 5.5 ÷ 8.1 | 7.4 | 6.4 | 7.8 | 3.6 ÷ 8.0 |
| 35/50 | ≤ 8 | 6.5 | 1.2 | 5.3 ÷ 7.7 | 6.8 | 6.2 | 7.3 | 3.6 ÷ 8.0 |
| 50/70 | ≤ 9 | 5.1 | 1.4 | 3.7 ÷ 6.5 | 5.4 | 3.8 | 6.2 | 2.4 ÷ 7.2 |
| 70/100 | ≤ 9 | 5.6 | 1.5 | 4.1 ÷ 7.1 | 5.9 | 4.8 | 6.4 | 2.8 ÷ 8.2 |
| 100/150 | ≤ 10 | 5.3 | 1.0 | 4.3 ÷ 6.3 | 5.2 | 4.8 | 6.4 | 3.6 ÷ 7.0 |
| 160/220 | ≤ 11 | 5.2 | 0.9 | 4.3 ÷ 6.1 | 5.2 | 4.6 | 5.6 | 3.4 ÷ 7.4 |
| ORBITON 25/55-60 | ≤ 8 or TBR*** | 5.8 | 1.2 | 4.6 ÷ 7.0 | 6.0 | 5.2 | 6.4 | 3.6 ÷ 7.8 |
| ORBITON 45/80-55 | ≤ 8 or TBR*** | 2.3 | 2.6 | -0.3 ÷ 4.9 | 2.4 | 1.0 | 4.0 | -2.0 ÷ 6.8 |
| ORBITON 45/80-65 | ≤ 8 or TBR*** | 1.6 | 2.5 | -0.9 ÷ 4.1 | 2.6 | -1.0 | 3.8 | -2.0 ÷ 6.0 |
| ORBITON 25/55-80 HiMA | ≤ 8 or TBR*** | 2.4 | 2.0 | 0.4 ÷ 4.4 | 2.0 | 1.0 | 3.0 | 0.5 ÷ 6.5 |
| ORBITON 45/80-80 HiMA | ≤ 8 or TBR*** | 1.9 | 1.4 | 0.5 ÷ 3.3 | 2.5 | 1.0 | 3.0 | -1.0 ÷ 3.5 |
| ORBITON 65/105-80 HiMA | ≤ 8 or TBR*** | 2.6 | 1.2 | 1.4 ÷ 3.8 | 3.0 | 2.5 | 3.5 | 0.5 ÷ 3.5 |

* test results refer to the period: January 2018–April 2020

** increase in R&B for paving grade bitumen according to EN 12591; for ORBITON and ORBITON HiMA according to EN 14023

*** TBR – To Be Reported applies to drop in softening point R&B for ORBITON and ORBITON HiMA acc. to PN-EN 14023

2.2.5.3. CHANGE OF MASS AFTER HARDENING

As a result of the ageing process, the bitumen mass may change (increase or decrease). The change of mass after ageing is determined according to EN 12607-1. It is the absolute value of the percentage difference between the mass of a fresh bitumen

sample and the mass of the same sample after the RTFOT ageing. Data on the change in mass after ageing for the tested bitumens are shown in Table 2.8. The table takes into account that the value of mass change can be a positive or a negative value.

Table 2.8.

Statistical parameters of the results of resistance of hardening determination – change of mass [%] resulting from the ageing using RTFOT method, for bitumens produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT** [%] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUANTILE | THIRD QUANTILE | MIN-MAX VALUE |
|------------------------|-------------------|-----------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | \bar{x}_{min} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| 20/30 | ≤ 0.5 | -0.05 | 0.03 | -0.08 ÷ -0.02 | -0.04 | -0.07 | -0.02 | -0.11 ÷ -0.01 |
| 35/50 | ≤ 0.5 | -0.05 | 0.04 | -0.09 ÷ -0.01 | -0.05 | -0.08 | -0.03 | -0.16 ÷ 0.01 |
| 50/70 | ≤ 0.5 | -0.02 | 0.06 | -0.08 ÷ 0.04 | -0.03 | -0.06 | 0.06 | -0.10 ÷ 0.08 |
| 70/100 | ≤ 0.8 | -0.03 | 0.06 | -0.09 ÷ 0.03 | -0.04 | -0.08 | 0.02 | -0.12 ÷ 0.10 |
| 100/150 | ≤ 0.8 | -0.06 | 0.08 | -0.14 ÷ 0.02 | -0.06 | -0.09 | 0.02 | -0.25 ÷ 0.06 |
| 160/220 | ≤ 1.0 | -0.04 | 0.06 | -0.10 ÷ 0.02 | -0.04 | -0.08 | 0.01 | -0.16 ÷ 0.04 |
| ORBITON 25/55-60 | ≤ 0.5 | -0.05 | 0.08 | -0.13 ÷ 0.03 | -0.05 | -0.12 | -0.01 | -0.16 ÷ 0.18 |
| ORBITON 45/80-55 | ≤ 0.5 | -0.03 | 0.05 | -0.08 ÷ 0.02 | -0.04 | -0.06 | -0.01 | -0.12 ÷ 0.11 |
| ORBITON 45/80-65 | ≤ 0.5 | -0.03 | 0.06 | -0.09 ÷ 0.03 | -0.04 | -0.06 | 0.02 | -0.15 ÷ 0.08 |
| ORBITON 25/55-80 HiMA | ≤ 0.5 | 0.03 | 0.08 | -0.05 ÷ 0.11 | 0.06 | -0.02 | 0.08 | -0.12 ÷ 0.10 |
| ORBITON 45/80-80 HiMA | ≤ 0.5 | 0.03 | 0.07 | -0.04 ÷ 0.10 | 0.06 | -0.05 | 0.08 | -0.10 ÷ 0.10 |
| ORBITON 65/105-80 HiMA | ≤ 0.5 | 0.06 | 0.03 | 0.03 ÷ 0.09 | 0.06 | 0.03 | 0.06 | 0.03 ÷ 0.10 |

* test results refer to the period: January 2018–April 2020

** for paving grade bitumen according to EN 12591; for ORBITON and ORBITON HiMA according to EN 14023

2.2.6. BITUMEN DENSITY

Tests of the bitumen density is performed in accordance with EN 15326. The goal of the test is to determine the ratio of the density of the tested bituminous binders to the density of the test fluid, determined under the same temperature conditions.

The density of the bitumen is necessary for calculations of volume parameters of asphalt mixtures in accordance with EN 12697-8. The bituminous binders densities shown

in Table 2.9 can be used in the design of asphalt mixtures.

As a standard, in laboratories cooperating with ORLEN Asphalt, densities of all bituminous binders are determined at a temperature of 15°C twice a year. Table 2.9 below presents the average results of density testing of bitumens produced in the 2018–2020 period. The test was performed according to the method using a pycnometer with a capillary plug, at 15°C.

Table 2.9.

Average results of the density of bituminous binders produced in the 2018–2020 period

| TYPE OF BINDER | DENSITY AT 15°C ACC. TO > EN 15326 [kg/m ³] |
|--|---|
| Paving grade bitumen 20/30 | 1028 |
| Paving grade bitumen 35/50 | 1026 |
| Paving grade bitumen 50/70 | 1024 |
| Paving grade bitumen 70/100 | 1021 |
| Paving grade bitumen 100/150 | 1019 |
| Paving grade bitumen 160/220 | 1015 |
| Polymer modified bitumen ORBITON 25/55-60 | 1023 |
| Polymer modified bitumen ORBITON 45/80-55 | 1021 |
| Polymer modified bitumen ORBITON 45/80-65 | 1021 |
| Highly modified bitumen ORBITON 25/55-80 HiMA | 1021 |
| Highly modified bitumen ORBITON 45/80-80 HiMA | 1019 |
| Highly modified bitumen ORBITON 65/105-80 HiMA | 1015 |

The density values of bitumen given in Table 2.9. refer to measurements at 15°C. If bitumens are used at other temperatures, the given density at 15°C can be converted into the density at the application temperature by using a conversion factor in the form of a bitumen volume expansion coefficient of 0.00061°C⁻¹ [16]. To simplify the calculations, the following equation can be used:

$$\rho_x = \rho_{15} - (0.00061 \cdot \Delta t)$$

where:

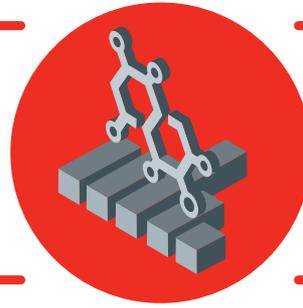
ρ_x – density at the calculated temperature X

ρ_{15} – density at 15°C in Mg/m³

Δt – temperature difference (X – 15), X ∈ <15,16...200>

Calculated density values are obviously approximations and not exact values.

2.3. ADDITIONAL PROPERTIES OF POLYMER MODIFIED BITUMENS



2.3.1. ELASTIC RECOVERY AT 25°C BEFORE AND AFTER RTFOT

The elastic recovery test both before and after the RTFOT ageing is performed in accordance with EN 13398. The goal of the test is to determine the conventional elasticity of bitumen by

measuring the distance between the ends of the stretched and cut sample under specified conditions, Fig. 2.40.



Fig. 2.40.

(A) General view of the apparatus for testing elastic recovery, and (B) view of a bitumen sample during testing (photo by ORLEN Asfalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

In the case of testing bitumens after the RTFOT ageing, the result allows for answering the question to what extent the polymer (elastomer) remains effective after ageing, and thus, how effectively the polymer network can act in the asphalt pavement.

Tables 2.10. and 2.11. contain statistical parameters of the results of elastic recovery determination at 25°C for polymer modified bitumens ORBITON and highly modified bitumens ORBITON HiMA produced in the 2018–2020 period, before and after the RTFOT ageing, respectively.

Table 2.10.

Statistical parameters of the results of elastic recovery determination at 25°C before the RTFOT ageing for ORBITON and ORBITON HiMA binders produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT EN 14023 [%] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUARTILE | THIRD QUARTILE | MIN-MAX VALUE |
|------------------------|--------------------------|------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | x_{mm} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| ORBITON 25/55-60 | ≥ 60 | 77.9 | 4.8 | 73.1 ÷ 82.7 | 80 | 76 | 81 | 65 ÷ 83 |
| ORBITON 45/80-55 | ≥ 70 | 85.9 | 4.0 | 81.9 ÷ 89.9 | 86 | 84 | 88 | 70 ÷ 90 |
| ORBITON 45/80-65 | ≥ 80 | 87.5 | 3.0 | 84.5 ÷ 90.5 | 89 | 85 | 89 | 80 ÷ 91 |
| ORBITON 25/55-80 HiMA | ≥ 80 | 93.1 | 2.6 | 90.5 ÷ 95.7 | 93 | 90 | 95 | 90 ÷ 97 |
| ORBITON 45/80-80 HiMA | ≥ 80 | 95.3 | 2.6 | 92.7 ÷ 97.9 | 96 | 95 | 96 | 90 ÷ 100 |
| ORBITON 65/105-80 HiMA | ≥ 80 | 95.2 | 3.4 | 91.8 ÷ 98.6 | 95 | 95 | 98 | 87 ÷ 99 |

* test results refer to the period: January 2018–April 2020

Table 2.11.

Statistical parameters of the results of elastic recovery determination at 25°C after ageing by the RTFOT method for ORBITON and ORBITON HiMA binders produced by ORLEN Asphalt in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT EN 14023 [%] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUARTILE | THIRD QUARTILE | MIN-MAX VALUE |
|------------------------|--------------------------|------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | x_{mm} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| ORBITON 25/55-60 | ≥ 50 | 71.2 | 6.8 | 64.4 ÷ 78.0 | 72 | 66 | 77 | 59 ÷ 80 |
| ORBITON 45/80-55 | ≥ 50 | 82.3 | 3.4 | 78.9 ÷ 85.7 | 82 | 81 | 85 | 70 ÷ 86 |
| ORBITON 45/80-65 | ≥ 60 | 83.7 | 2.5 | 81.2 ÷ 86.2 | 85 | 82 | 86 | 80 ÷ 87 |
| ORBITON 25/55-80 HiMA | ≥ 50 | 89.1 | 3.4 | 85.7 ÷ 92.5 | 90 | 86 | 92 | 84 ÷ 94 |
| ORBITON 45/80-80 HiMA | ≥ 60 | 91.8 | 3.3 | 88.5 ÷ 95.1 | 92 | 88 | 95 | 87 ÷ 96 |
| ORBITON 65/105-80 HiMA | ≥ 70 | 94.4 | 0.9 | 93.5 ÷ 95.3 | 95 | 94 | 95 | 93 ÷ 95 |

* test results refer to the period: January 2018–April 2020

Additionally, Figures 2.41. to 2.43. show histograms and box plots for the elastic recovery results of selected polymer modified and highly modified bitumens produced in the 2018–2020 period.

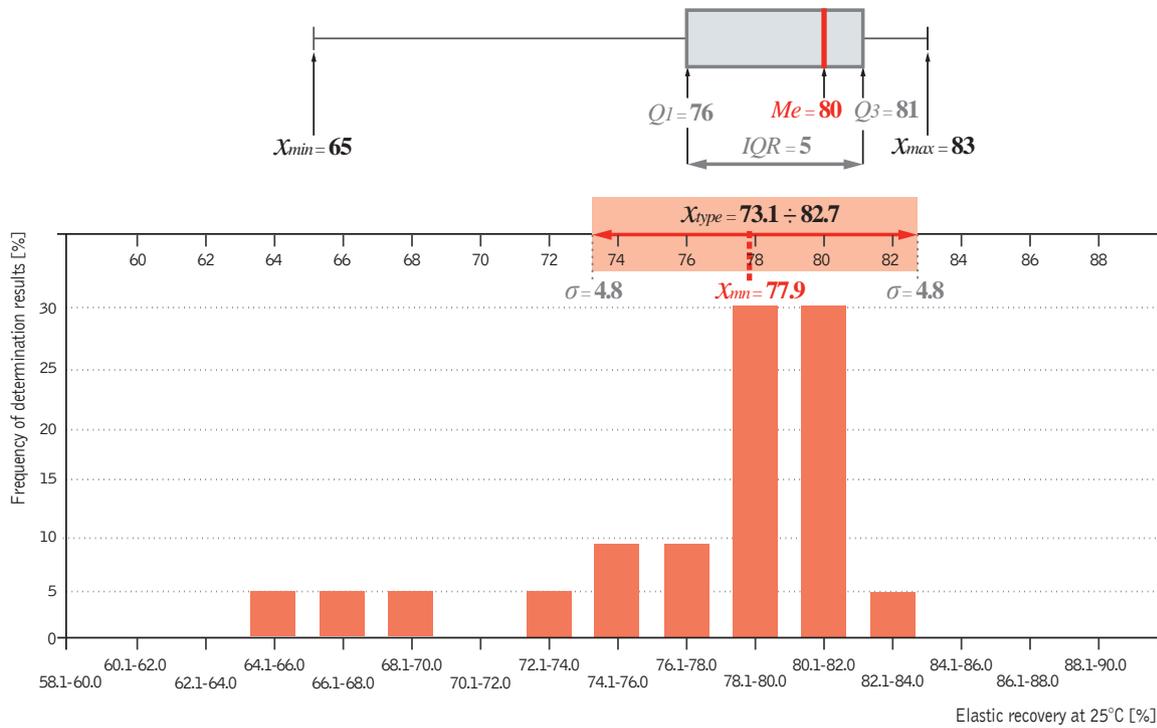


Fig. 2.41. Histogram and box plot presenting the results of elastic recovery at 25°C of polymer modified bitumen **ORBITON 25/55-60** before ageing produced in the 2018–2020 period (standard range: ≥ 60 [%])

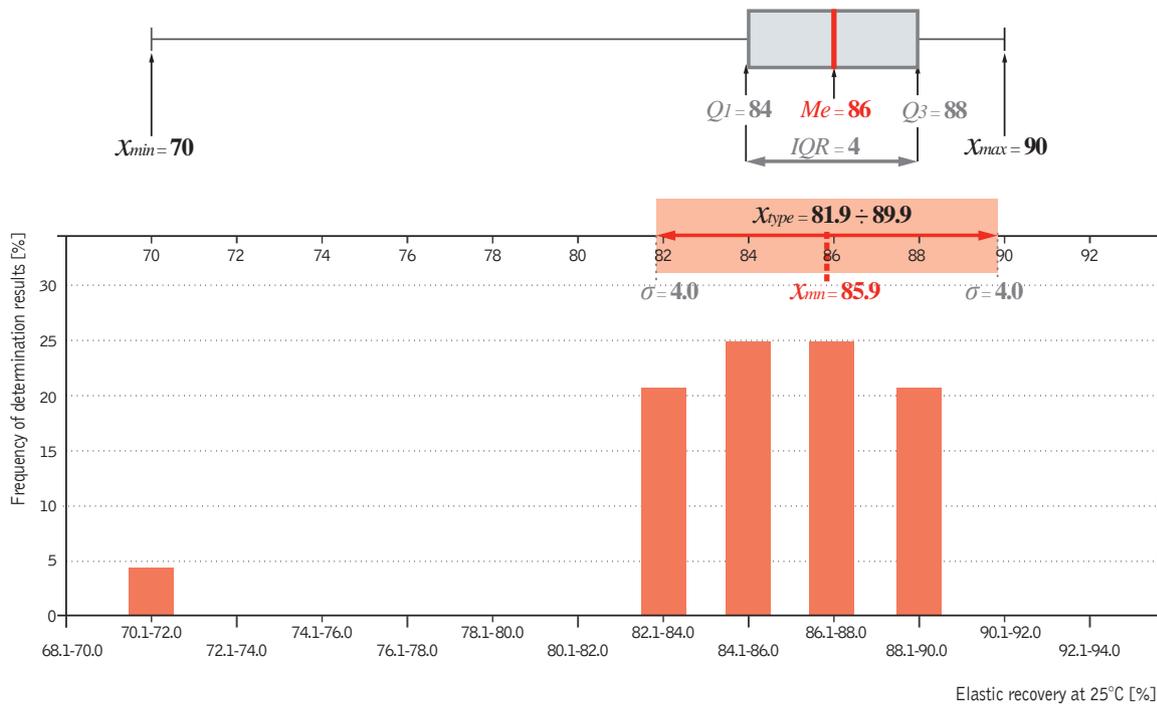


Fig. 2.42. Histogram and box plot presenting the results of elastic recovery at 25°C of polymer modified bitumen **ORBITON 45/80-55** before ageing produced in the 2018–2020 period (standard range: ≥ 70 [%])

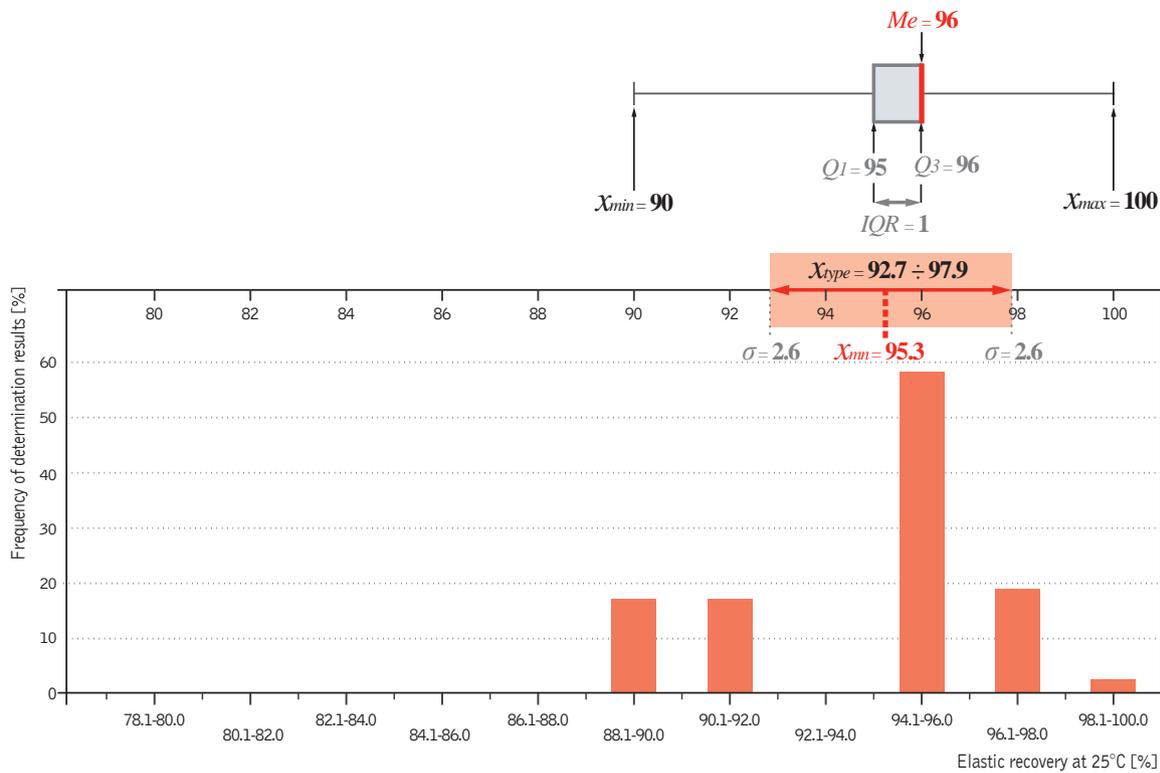


Fig. 2.43. Histogram and box plot presenting the results of elastic recovery at 25°C of highly modified bitumen **ORBITON 45/80-80 HiMA** before ageing produced in the 2018–2020 period (standard range: ≥ 80 [%])

2.3.2. PLASTICITY RANGE

Plasticity range is the difference between the softening point and the Fraass breaking point.

It is therefore the temperature range over which a bituminous binder retains its viscoelastic properties.

From the point of view of the binder user, classical bitumen theory emphasises the greatest possible value for this range, i.e. the lowest possible breaking point and the highest possible softening point. The average plasticity ranges of polymer modified bitumens produced in the 2018–2020 period are shown in Table 2.12.

Table 2.12. Average plasticity ranges of bituminous binders produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT EN 14023 | PLASTICITY RANGE [°C] |
|--|----------------------|-----------------------|
| Polymer modified bitumen ORBITON 25/55-60 | NR | 71.9 |
| Polymer modified bitumen ORBITON 45/80-55 | NR | 81.6 |
| Polymer modified bitumen ORBITON 45/80-65 | NR | 94.7 |
| Highly modified bitumen ORBITON 25/55-80 HiMA | NR | 113.1 |
| Highly modified bitumen ORBITON 45/80-80 HiMA | NR | 100.4 |
| Highly modified bitumen ORBITON 65/105-80 HiMA | NR | 110.7 |

* test results refer to the period: January 2018 – April 2020
NR – No Requirement

2.3.3. MICROSTRUCTURE

One of the methods enabling observation of the polymer microstructure of bitumens is fluorescence microscopy. The test is performed in accordance with EN 13632 standard.



Fig. 2.44.
General view of fluorescence microscope with UV lamp (photo by ORLEN Asfalt sp. z o.o. with permission of ORLEN Laboratorium S.A.)

The test is performed on a fresh fracture of a frozen modified bitumen sample, using a fluorescence microscope with a UV lamp (Fig. 2.44.) and analysing the image in reflected light.

On the basis of Annex A.3 of EN 13632 the structure of polymer modified bitumens can be described according to the letter markings characterising the PMB dispersion system:

| | |
|-----------------------------|---|
| 1. PHASE CONTINUITY: | P: CONTINUOUS POYMER PHASE B: CONTINUOUS BITUMEN PHASE |
| 2. PHASE DESCRIPTION | X: CONTINUITY OF BOTH PHASES H: HOMOGENOUS I: INHOMOGENOUS |
| 3. SIZE DESCRIPTION | S: SMALL (< 10 μm) M: MEDIUM (10 – 100 μm) L: LARGE (> 100 μm) |
| 4. SHAPE DESCRIPTION | r: ROUNDISH, OVAL s: STRIPES o: OTHER |

Example microstructure of polymer modified bitumens according to EN 13632, produced by ORLEN Group is presented in Figure 2.45.

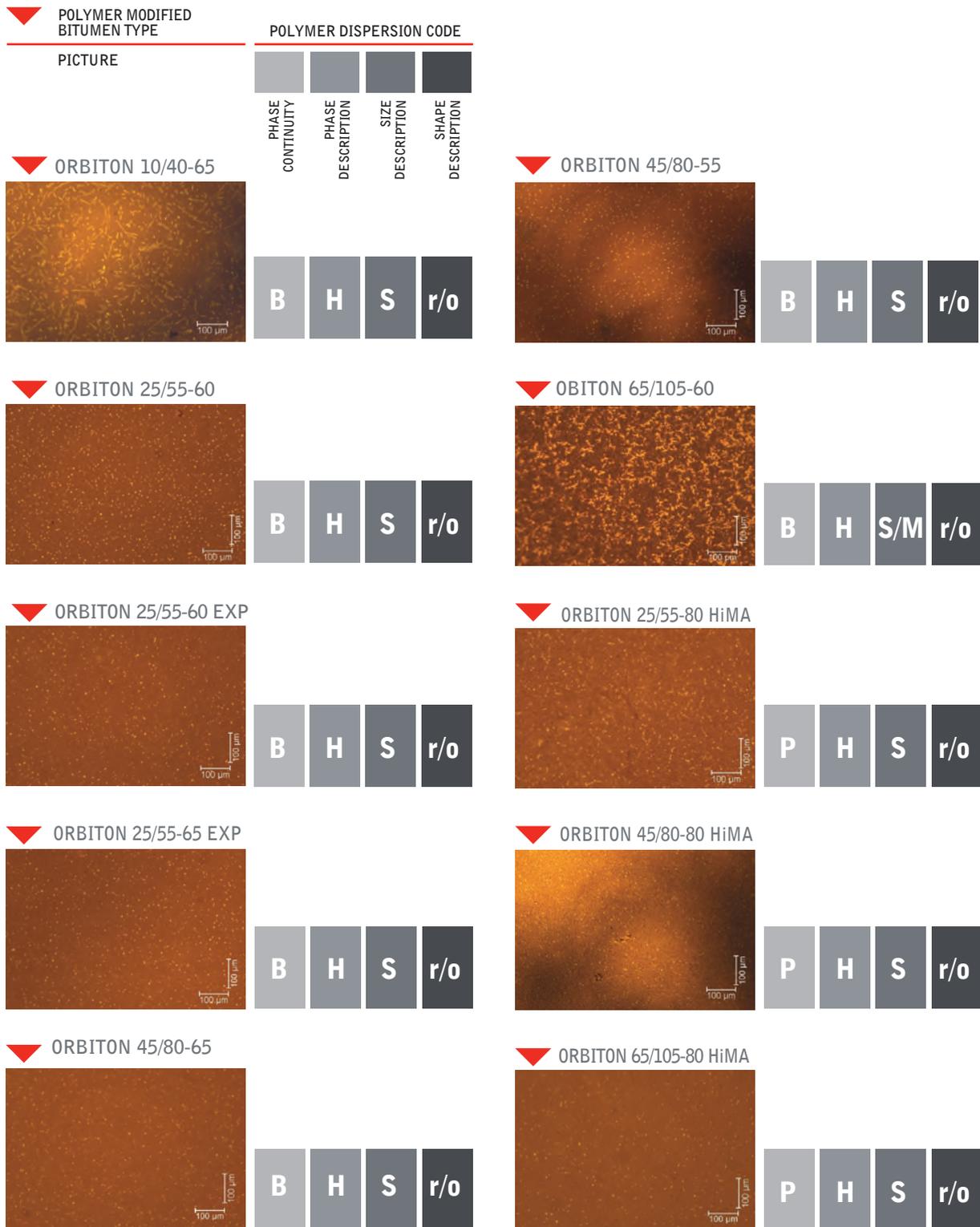


Fig. 2.45. Microstructure of polymer modified bitumens according to EN 13632

2.3.4. FORCE DUCTILITY (COHESION)

Cohesion describes the mutual attraction of particles of the same material due to intermolecular forces. For bitumens, a sufficiently high value of cohesion enables the bituminous binder to transmit tensile stress in the road pavement. It is assumed that it makes it more resistant to cracking and fatigue.

The force ductility test (at low tensile rate) is performed in accordance with EN 13589 revision published in 2019. The essence of performing the test is to determine the force required to stretch the sample at a specified temperature.

A properly formed sample is placed in a ductilometer, in a water bath at an appropriate (specified for each type of modified bitumen) temperature. The sample is then continuously stretched at the rate of 50 mm/min until it breaks or reaches a minimum elongation of 1333%, i.e. 400 mm. The force is recorded throughout the tensile process using sensors. According to the revised standard, the final result of cohesion is calculated

as the difference between the cohesion energy at breaking or at the elongation of 1333% (400 mm) and the cohesion energy at the elongation of 667% (200 mm). To determine the compliance with the specification, the cohesion energy at 400 mm elongation is adopted.

Figure 2.46 shows a view of the bitumen before and after the force ductility test, respectively.

Table 2.13. contains statistical parameters of the results of cohesion determinations for polymer modified bitumen ORBITON and highly modified bitumen ORBITON HiMA produced in the 2018–2020 period. It should be noted that following the publication of the amendment to the Polish standard PN-EN 14023:2011/Ap2:2020-02, the requirement classes of certain bituminous binders have changed. These changes are included in Table 2.13. which shows the current requirements.

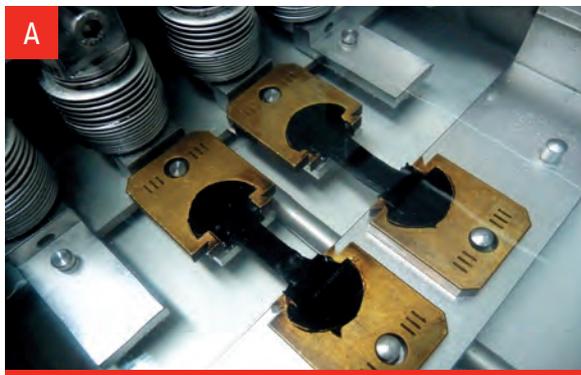


Fig. 2.46.

(A) View of a bitumen sample before testing (B) and after testing (elongated to 400 mm) in a ductilometer (photo by ORLEN Asfalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

Table 2.13.

Statistical parameters of the results of cohesion determinations for ORBITON and ORBITON HiMA binders produced in the 2018–2020 period*

| TYPE OF BINDER | REQUIREMENT EN 14023 [J/cm ²] | MEAN VALUE | STANDARD DEVIATION | TYPICAL RANGE | MEDIAN | FIRST QUANTILE | THIRD QUANTILE | MIN-MAX VALUE |
|--------------------------|---|-----------------|--------------------|---------------|-----------|----------------|----------------|---------------------|
| | | \bar{x}_{min} | σ | x_{type} | Me, Q_2 | Q_1 | Q_3 | $x_{min} - x_{max}$ |
| ORBITON 25/55-60 | ≥ 2.0 (at 10°C) | 5.0 | 1.0 | 4.0 ÷ 6.0 | 5.2 | 4.8 | 5.9 | 2.3 ÷ 6.2 |
| ORBITON 45/80-55 | ≥ 3.0 (at 5°C) | 7.7 | 0.9 | 6.8 ÷ 8.6 | 7.9 | 7.0 | 8.2 | 6.0 ÷ 9.3 |
| ORBITON 45/80-65 | ≥ 2.0 (at 10°C) | 4.3 | 0.8 | 3.5 ÷ 5.1 | 4.6 | 4.2 | 4.9 | 2.0 ÷ 5.2 |
| ORBITON 25/55-80 HiMA | ≥ 0.5*** (at 15°C) | 3.2 | 0.6 | 2.6 ÷ 3.8 | 3.1 | 2.6 | 4.0 | 2.5 ÷ 4.1 |
| ORBITON 45/80-80 HiMA | ≥ 2.0*** (at 10°C) | 3.6 | 0.7 | 2.9 ÷ 4.3 | 3.6 | 3.1 | 4.2 | 2.3 ÷ 4.9 |
| ORBITON 65/105-80 HiMA** | ≥ 1.0*** (at 5°C) | 8.4 | — | — | — | — | — | 7.4 ÷ 9.4 |

* test results refer to the period: January 2018–April 2020

** test results refer to the period: November 2019–April 2020; due to the insufficient number of results caused by the change in the test temperature, only selected statistical parameters were determined for the binder

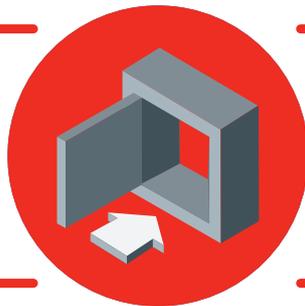
*** requirement specified in PN-EN 14023:2011/Ap2:2020-02

3. FUNCTIONAL PROPERTIES OF BITUMEN

The Research, Development and Innovation Department of ORLEN Asphalt periodically conducts research programs on functional properties of bituminous binders produced by ORLEN Group. The research is carried out using cutting-edge research methods used in Europe and the United States.

This chapter presents the results of functional tests of selected paving grade, polymer modified and highly modified bitumen. The tests were carried out in the research laboratory ORLEN UniCRE in Czech Republic, belonging to ORLEN Group.

3.1. INTRODUCTION



Modern test methods for bituminous binders are based mainly on the tests of rheological properties. They enable us to carry out a comprehensive assessment of the functional properties of bitumens, taking into account the requirements arising from the real working conditions of these materials [3, 4]. Bituminous binders are viscoelastic materials which properties depend on temperature and load time and vary from conventionally elastic properties – at low temperature and/or short load time, to viscous properties – at high temperature and/or long load time.

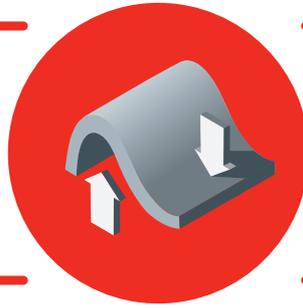
Rheology is a branch of science that deals with aspects of deformation of bodies subjected to stress. It describes phenomena occurring between the solid and the fluid states of materials, as most materials exhibit intermediate properties – they are to varying degrees both elastic and viscous and therefore they are called viscoelastic materials. In other words, we can treat rheology as a science dealing with the behaviour of real substances

which, when subjected to deformation, exhibit more than one basic rheological property, namely elasticity or viscosity [1].

In this chapter, the results of testing the properties at low-, intermediate- and high-temperatures for bituminous binders are presented. They were carried out in accordance with the American methodology (conventionally “Superpave”) and the EN 14023 standard and its draft amendment from 2021.

At the same time, it is worth pointing out that the published test results should be analysed bearing in mind that they are the results of randomly selected bitumen samples and do not represent typical values achieved during the entire (and each) production season. Obviously, the values are not guaranteed by ORLEN Asphalt sp. z o.o. Also, the results should not be treated as average values based on statistical principles.

3.2. FUNDAMENTALS OF RHEOLOGY



RHEOLOGY

Rheology (from the Greek rheo – to flow and logos – science) is the science that deals with the flow and deformation of materials under the influence of external forces (in other words – under stress).

To better understand the described phenomena, basic rheological terms are defined below [2].

STRESS

Stress is the ratio of the applied force F to the surface area A . The unit of stress is Pascal [Pa]. If the force acts perpendicular to the surface, then we speak of normal stress, when the force acts parallel to the surface then we speak of shear stress.

STRAIN

Strain occurs in any material subject to force (stress). There are three basic types of strain: elastic strain, plastic strain and flow. Elastic strain disappears once the stress causing the deformation is removed (it is reversible). Plastic strain, as opposed to the elastic strain, is irreversible and does not disappear once the stress causing the deformation is removed. Flow, on the other hand, is an irreversible deformation that increases continuously over time.

Bituminous binders are subject to viscoelastic deformation, which means that they cannot completely return to their original form once the applied stress is removed. In the case of multiple stress cycles, an accumulation of permanent strains is observed. In practice, they take form of deepening ruts in the pavement.

PHASE ANGLE

In the study of viscoelastic materials, we observe a delay in the occurrence of a strain resulting from a load. The measure of this delay is phase angle δ . Phase angle, therefore, defines the delay of the resulting strain with respect to the applied stress.

If the phase angle is:

- 0° (zero), then there is no phase shift between stress and strain and the material is perfectly elastic (Hooke's body),
- greater than 0° (zero) but less than 90° , then there is a phase shift between stress and strain and the material is viscoelastic,
- equal to 90° , then the material is perfectly viscous (Newtonian fluid).

Figure 3.1 shows the phase shift that occurs between the applied stress and the response of the material, i.e. the strain in a sinusoidal load test.

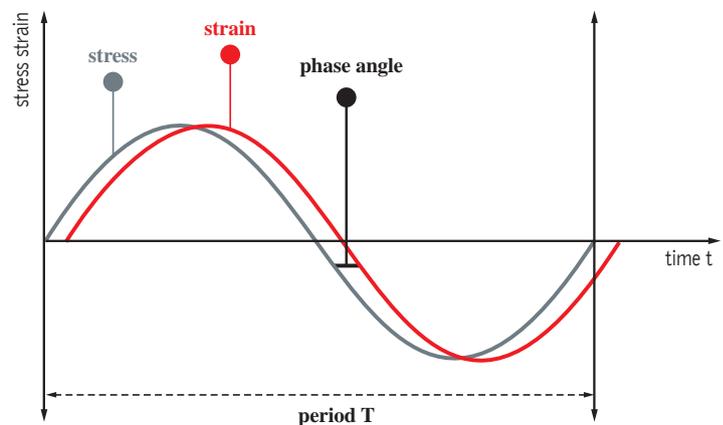


Fig. 3.1.

Graphical interpretation of phase angle δ [own fig.]

CREEP

It is a phenomenon defined as the process of deformation of a material under prolonged load. A good road example of creep is the deformation of an asphalt layer under a load caused by a stationary heavy vehicle.

Important assumptions of the creep process for viscoelastic materials are that the acting load does not exceed the strength of the material, the process accelerates with increasing temperature and load magnitude. Destruction of a material by creep does not occur suddenly, this process takes time.

RELAXATION

Stress relaxation is a phenomenon related to the viscoelastic behaviour of materials. It consists in an initially rapid and then very slow decrease of stress in a deformed element of a material (with no change in the value of this deformation). The relaxation phenomenon is caused by flow.

Stress relaxation processes are of great importance in asphalt pavements, e.g. as one of the factors reducing the problem of low temperature cracking. If we consider asphalt mixtures as viscoelastic materials, we can speak of stress relaxation, e.g. in winter periods when the wearing course is subjected to shrinkage deformations. The relaxation phenomenon can compensate for thermal stresses.

The magnitude of the relaxation in a given material depends on the temperature, time and level of the resulting stress.

COMPLEX STIFFNESS MODULUS $|G^*|$

This is a measure of the overall resistance of a binder to deformation. It consists of an elastic part and a viscous part, (Fig. 3.2.).

It is desirable that the elastic part of the binder, G' , is the dominant component of the complex stiffness modulus $|G^*|$.

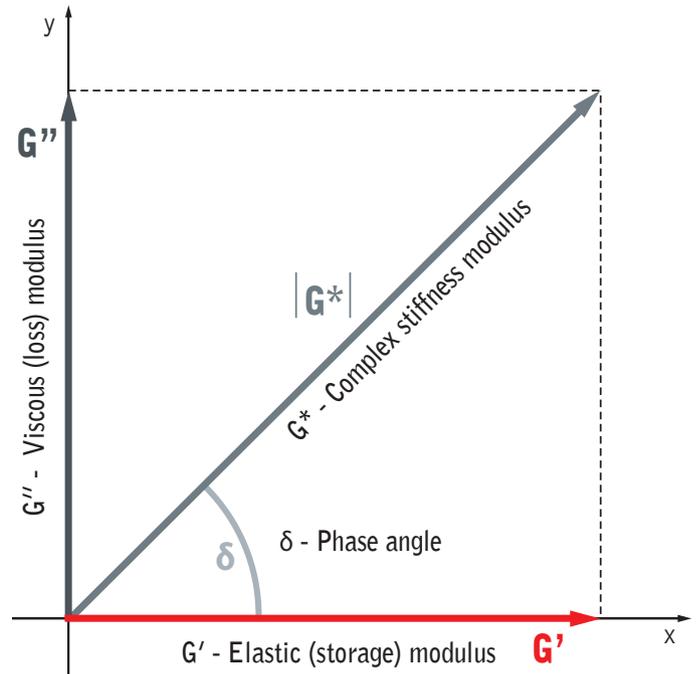


Fig. 3.2. Graphical interpretation of the complex stiffness modulus $|G^*|$ [Own figure]

A full characteristic of a given bituminous binder can be obtained by knowing both $|G^*|$ and δ because binders can show different proportions of elastic and viscous parts for the same value of $|G^*|$. Both values depend on the temperature and load time of the sample.

SHEAR RATE

Also known as strain rate or deformation rate, it is defined as the rate of deformation changes in time.

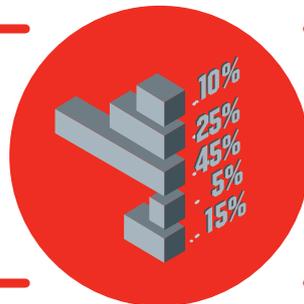
3.3. TEST PROGRAMME



In a research programme carried out by the Research, Development and Innovation Department of ORLEN Asfalt, the following types of bitumens were used to test functional properties:

- Paving grade bitumen:
20/30, 35/50, 50/70, 70/100,
- Polymer modified bitumen:
ORBITON 25/55-60, ORBITON 45/80-55, ORBITON 45/80-65, ORBITON 65/105-60,
- Highly modified bitumen:
ORBITON 25/55-80 HiMA, ORBITON 45/80-80 HiMA, ORBITON 65/105-80 HiMA.

3.4. TEST RESULTS



3.4.1. TESTING OF LOW-TEMPERATURE PROPERTIES



The low-temperature behaviour of the bitumen was tested using a Bending Beam Rheometer (BBR) – Fig. 3.3. The tests were performed in accordance with EN 14771:2012 and AASHTO T 313.



Fig.3.3.

A) Bending Beam Rheometer BBR, B) Mould for sample preparation (photo by: ORLEN Asfalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

The data obtained during the BBR test are used to determine the behaviour of bitumen at low temperature. The following parameters are obtained from the BBR test:

- **creep stiffness $S(t)$ at bending**, depending on a load time and measurement temperature,
- **parameter m -value**, as a function of the load time and measurement temperature.

During the test, a beam sample of dimensions $127 \pm 5 \times 12.7 \pm 0.1 \times 6.4 \pm 0.1^1$ is loaded with a constant force in the middle of the span, at a constant temperature. The deflection of

the sample due to creep of the bituminous binder is measured at specified time intervals (after 8 s; 15 s; 30 s; 60 s; 120 s and 240 s). The resulting force and displacement values are then used to calculate the stiffness of the sample at each time point. This yields a curve describing the dependence of sample stiffness as a function of load time – $S(t)$. In turn, performing the test at several temperatures allows to determine a series of curves $S(t)_{T_1, T_2, \dots}$ – i.e. the temperature sensitivity of the stiffness of a given bituminous binder. An example of a diagram presenting the test at different temperatures is shown in Figure 3.4.

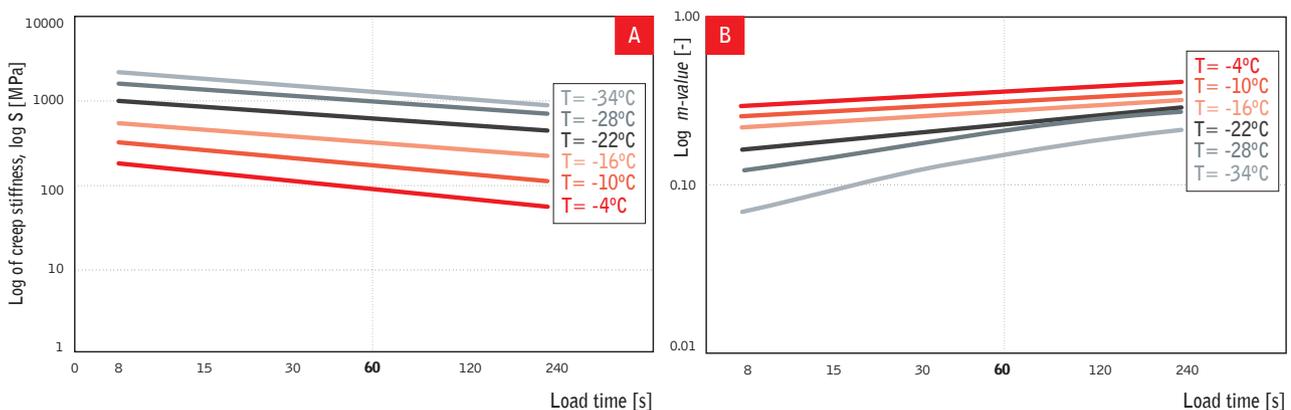


Fig.3.4.

Curves defining the relation of: a) sample stiffness as a function of a load time, b) parameter m as a function of a load time, PMB 25/55-60 results, a sample after RTFOT + PAV [research by ORLEN Asphalt]

It can be said that BBR tests can be used to assess:

- the degree of bitumen stiffening at low temperature (stiffness parameter **S**) – too high stiffness of the binder is unfavourable as it leads to the formation of cracks,
- the stress-relaxation capacity of the bitumen (parameter **m -value**), with $m \geq 0.300$ being the optimum, as thermal stress relaxation occurs more efficiently in bitumens with a high parameter m .

Both parameters (S and m) should be analysed in conjunction – they should not be treated separately as each describes a different behaviour of the bituminous binder and each is important for the assessment of low temperature properties.

In summary, it can be said that in terms of the desired low-temperature properties of bitumens, a lower value of S and a higher value of the m parameter are advantageous.

In previous editions of Bitumen Handbook, low-temperature property results were given based on the *Performance Grade* methodology used in the USA. The lower critical temperature (LCT) in this method is the higher of the two temperature values determined at the 60th second of loading, at which:

- the creep stiffness is $300 \text{ MPa} > T(S_{(60)} = 300 \text{ MPa})$,
- the parameter m is $0.300 > T(m_{(60)} = 0.300)$.

Additionally, the so-called lower PG is determined by reducing the LCT by 10°C , and rounding up to the appropriate functional series, according to a gradation of 6°C , e.g. $\text{LCT} = -24^\circ\text{C} > \text{lower PG} = -34^\circ\text{C}$ or $\text{LCT} = -17^\circ\text{C} > \text{lower PG} = -22^\circ\text{C}$ (lower PG type series: -10, -16, -22, -28, -34, -40, -46).

1) Sample dimensions are given in millimetres, in the order: length x height x width

European standardisation takes advantage of the US experience and in the 2020 draft standard prEN 14023 the BBR test was introduced as a test to check the consistency durability at low service temperature of modified bitumens. This durability is determined by the following parameters:

- temperature T at which the creep stiffness is 300 MPa $\rightarrow T(S_{(60)} = 300 \text{ MPa})$;
- parameter m at this temperature $T(S_{(60)} = 300 \text{ MPa})$.

As can be seen, in both cases, only the first part of the test is the same, i.e. the determination of the temperature at which the bitumen has a stiffness of $S_{(60)} = 300 \text{ MPa}$. In the European method of classification of polymer modified bitumen, the value of the parameter m at the same temperature where $S_{(60)} = 300 \text{ MPa}$ was introduced instead of the temperature where $m_{(60)} = 0.300$. In both the American and the European methods, the choice of how to evaluate the low-temperature properties of modified bitumen is similar; however, the adoption of a different rule for calculating the parameters can lead to significantly different

interpretations of the results, to which special attention should be paid.

The temperature $T(S_{(60)} = 300 \text{ MPa})$, according to prEN 14023, is determined by interpolation of the creep stiffness–temperature relation curve read at the 60th second of the measurement. In practice, this means that the creep stiffness value at 60th second of the measurement at each applied test temperature should be read from the graph presented in Fig. 3.4 and the values obtained should be plotted on the creep stiffness-temperature relation diagram, Fig. 3.5a.

The scheme for reading the values of temperature $T(S_{(60)} = 300 \text{ MPa})$ and parameter m at temperature T is shown in Fig. 3.5 a-b. The evaluation of the result of the parameter m as proposed in prEN 14023 comes down to finding out what its value is. For the initiated, it remains to interpret whether the value of the parameter m is greater or lower than the limit value, i.e. 0.300, and what this means.

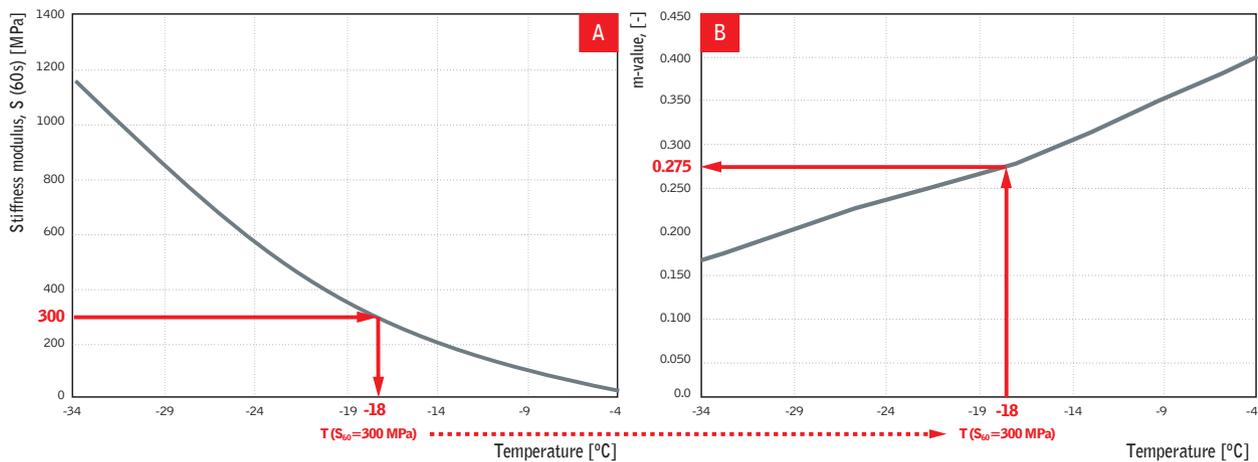


Fig.3.5. Example of the determination of a) the temperature $T(S_{(60)} = 300 \text{ MPa})$, b) the parameter m at $T(S_{(60)} = 300 \text{ MPa})$ [fig. based on prEN 14023]

Figures 3.6. to 3.8. show a comparison of the change in stiffness and parameter m , depending on the measurement temperature, for all tested bitumens. The test was performed in the temperature range from -4°C to -34°C . The samples were aged using RTFOT and PAV methods before testing.

Interpretation of graphs: lower value of $S =$ better, higher value of $m =$ better. The orange line indicates the stiffness at $S_{(60)} = 300 \text{ MPa}$ and the parameter $m_{(60)} = 0.300$.

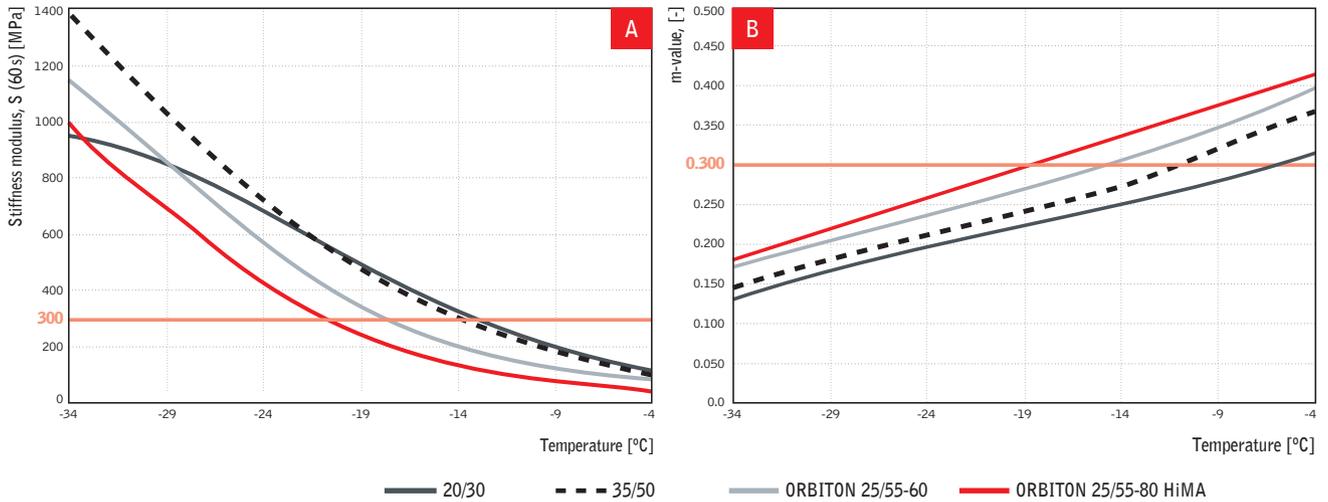


Fig. 3.6.

Change of a) stiffness S , b) parameter m , depending on temperature for bitumens: 20/30, 35/50, PMB 25/55-60 and PMB 25/55-80 HiMA [source: ORLEN Asphalt internal research]

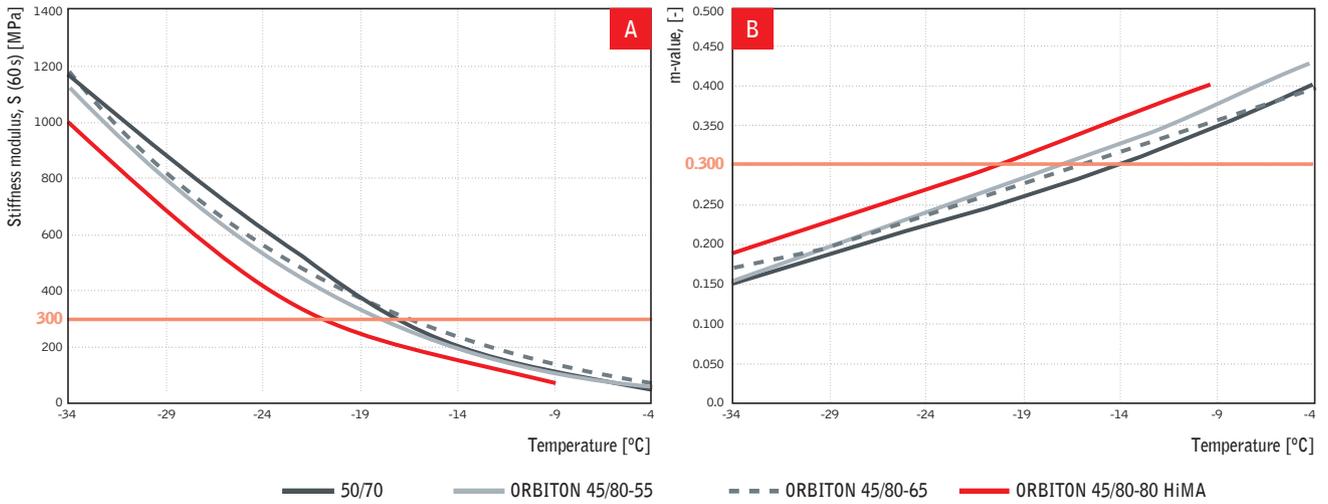


Fig. 3.7.

Change of a) stiffness S , b) parameter m , depending on temperature for bitumens: 50/70, PMB 45/80-55, PMB 45/80-65 and PMB 45/80-80 HiMA [source: ORLEN Asphalt internal research]

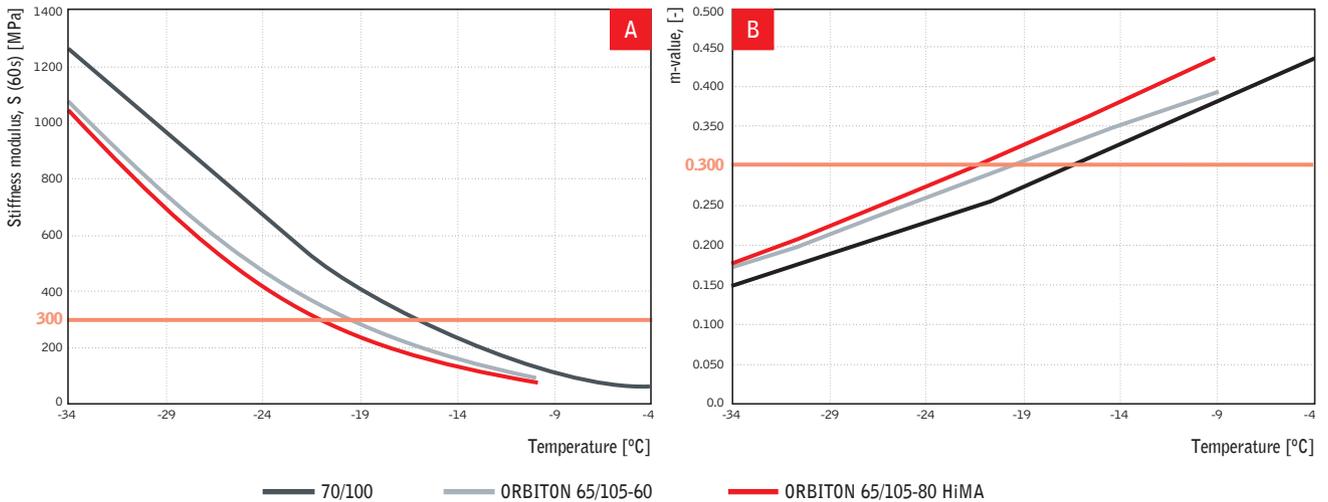


Fig. 3.8.

Change of a) stiffness S , b) parameter m , depending on temperature for bitumens: 70/100, PMB 65/105-60 and PMB 65/105-80 HiMA [source: ORLEN Asphalt internal research]

Table 3.1 shows the results of low-temperature testing of bituminous binders according to the American system

AASHTO M320/M332, and based on the 2020 draft standard prEN 14023.

Table 3.1.

Results of tests of low-temperature properties of the tested bitumens in accordance with the Superpave system and prEN 14023 of 2020 [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | Superpave System | | | prEN 14023 | |
|------------------------|---|---|--------------------------|---|--|
| | Low critical temperature LCT [°C] | | Difference LCT(S)-LCT(m) | Temperature T at which creep stiffness is 300 MPa | Parameter <i>m</i> -value at temperature T |
| | Temperature T at which creep stiffness is 300 MPa | Temperature T at which the <i>m</i> -parameter is 0.300 | | | |
| | $T(S_{(60)}=300 \text{ MPa}), [^{\circ}\text{C}]$ | $T(m_{(60)}=0.300), [^{\circ}\text{C}]$ | ΔT_c | $T(S_{(60)}=300 \text{ MPa}), [^{\circ}\text{C}]$ | $m T(S_{(60)}=300 \text{ MPa}), [-]$ |
| INTERPRETATION | <i>less = better</i> | | | | <i>informative</i> |
| 20/30 | -13.8 | -7.1 | -6.8 | -13.8 | 0.256 |
| 35/50 | -15.2 | -11.0 | -4.3 | -15.2 | 0.276 |
| 50/70 | -16.2 | -14.4 | -1.7 | -16.2 | 0.291 |
| 70/100 | -16.3 | -16.9 | 0.6 | -16.3 | 0.314 |
| ORBITON 25/55-60 | -18.1 | -15.1 | -3.0 | -18.1 | 0.283 |
| ORBITON 45/80-55 | -18.6 | -16.9 | -1.7 | -18.6 | 0.290 |
| ORBITON 45/80-65 | -18.5 | -15.9 | -2.6 | -18.5 | 0.284 |
| ORBITON 65/105-60 | -20.7 | -20.0 | -0.7 | -20.7 | 0.295 |
| ORBITON 25/55-80 HiMA | -21.1 | -18.4 | -2.7 | -21.1 | 0.280 |
| ORBITON 45/80-80 HiMA | -21.8 | -20.1 | -1.7 | -21.8 | 0.288 |
| ORBITON 65/105-80 HiMA | -21.6 | -21.8 | 0.2 | -21.6 | 0.304 |

$T(S_{(60)}=300 \text{ MPa})$ is determined in the same way

To make the above data easier to analyse, Figure 3.9 graphically shows the results obtained for the temperature T at which the creep stiffness is 300 MPa ($T(S_{(60)} = 300 \text{ MPa})$), according to prEN 14023 of 2020. Bitumens were grouped according to penetration ranges corresponding to binder groups for specific road applications.

In turn, Fig. 3.10 shows the results of the temperature at which the creep stiffness is 300 MPa ($T(S_{(60)} = 300 \text{ MPa})$) and the corresponding value of the parameter *m*.

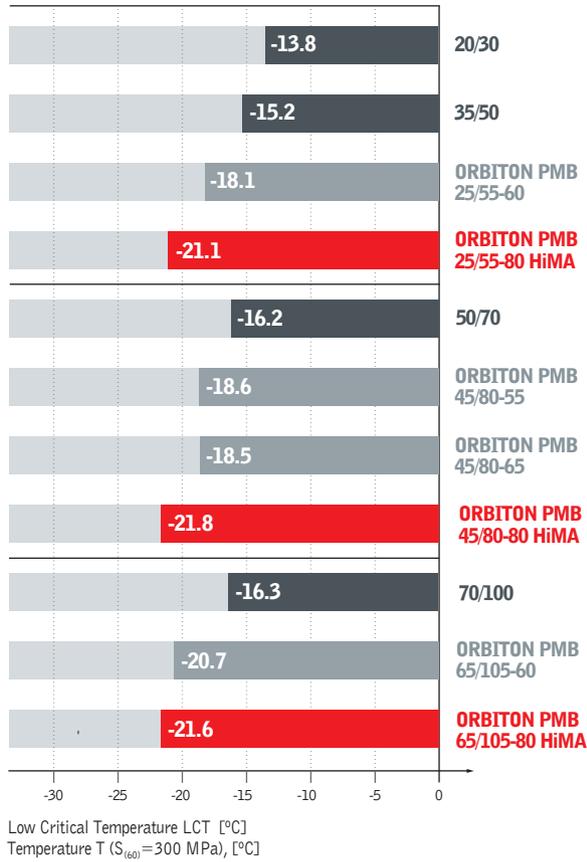


Fig. 3.9. Presentation of temperature T results at which the creep stiffness is 300 MPa (T (S₆₀ = 300 MPa)). Bitumens after RTFOT + PAV [source: ORLEN Asfalt internal research]. Interpretation of the graph: less = better

In the technical literature on bitumens and their low-temperature properties, a new **parameter ΔTc** has been recently introduced. The parameter ΔTc is defined, according to the American methodology as the difference between the low critical temperature determined at stiffness S₍₆₀₎ = 300 MPa, and the low critical temperature where the value of the parameter m is m₍₆₀₎ = 0.300. It is calculated according to the formula [8]:

$$\Delta Tc = Tc_{(S=300MPa)} - Tc_{(m=0,300)}$$

The parameter ΔTc is used to predict the susceptibility of bituminous binders to low-temperature cracking. It has been estimated that the value of the parameter ΔTc should not be lower than -5°C as the binder may show an increased susceptibility to cracking and ageing [8, 10].

Fig. 3.11. shows the value of ΔTc parameter for the tested bitumens. The orange colour indicates the zone with increased susceptibility to low-temperature cracking.

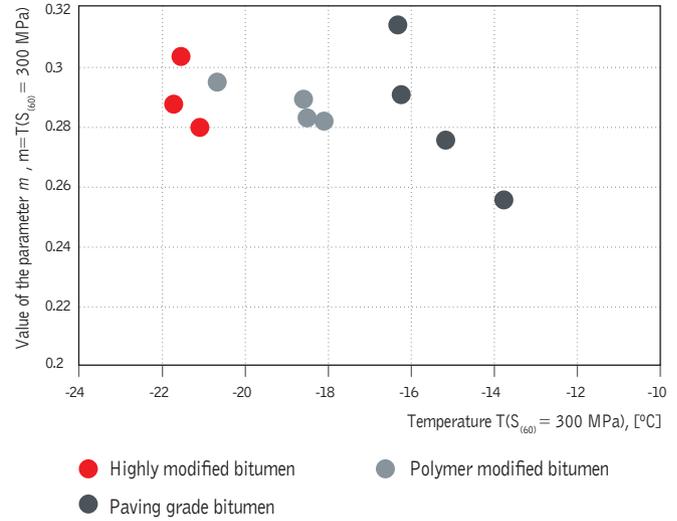


Fig. 3.10. Presentation of results of temperature T at which creep stiffness is 300 MPa (T (S₆₀ = 300 MPa)) and the corresponding value of parameter m – according to prEN 14023 of 2020. Bitumen after RTFOT + PAV ageing [source: ORLEN Asfalt internal research]

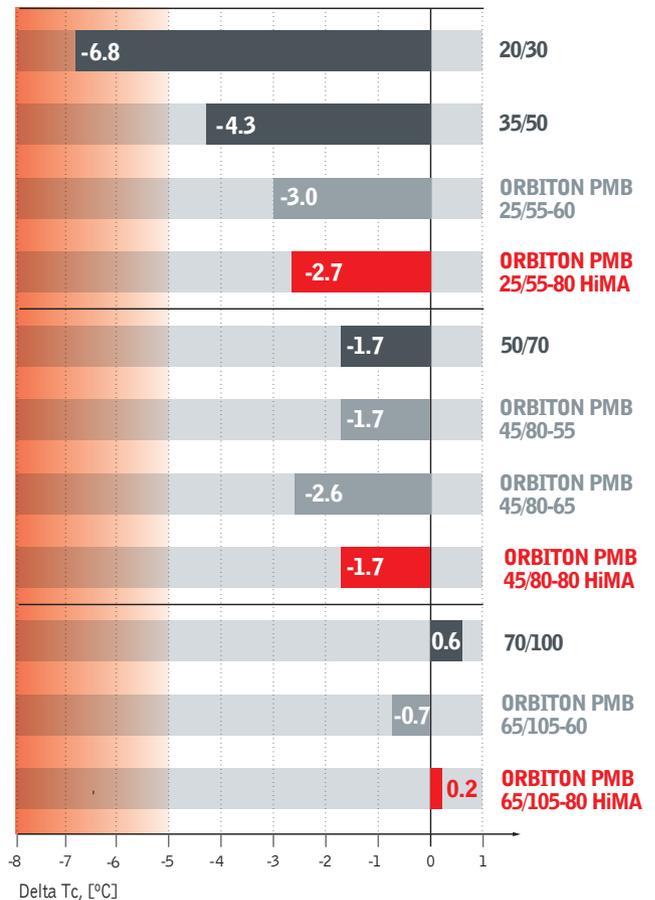


Fig. 3.11. Comparison of ΔTc parameter values, orange area indicates increased risk of low-temperature cracking [source: ORLEN Asfalt internal research]. Interpretation of the graph: less = better

Negative values of ΔT_c for the majority of the tested bitumens indicate that this parameter is controlled by the value of the $m_{(60)}$ parameter. Therefore, the key property of the tested binders

in terms of the resistance to shrinkage cracking is the ability to relax thermal stress rather than the presence of the excessive stiffening.

3.4.2. DSR TESTING OF PROPERTIES

3.4.2.1. DEPENDENCE OF THE COMPLEX STIFFNESS MODULUS $|G^*|$ AND PHASE ANGLE ON TEMPERATURE AND LOAD TIME

As described in previous chapters, the properties of bitumen as a viscoelastic material depend on temperature and load time. A tool that is used to investigate changes in the behaviour of bituminous binders is the dynamic shear rheometer (DSR), Figure 3.12. The test results presented below were performed in the DSR, based on the EN 14770 standard.

When testing the basic properties of bituminous binders using a dynamic shear rheometer, the complex stiffness modulus $|G^*|$ and the phase angle δ are determined for each sample. Both of these values depend on the bitumen type, temperature and load time.

Figures 3.13 to 3.15 show the temperature dependence of the complex stiffness modulus and the phase angle of bituminous binders. Bitumens were grouped according to penetration ranges corresponding to binder groups for specific road applications.

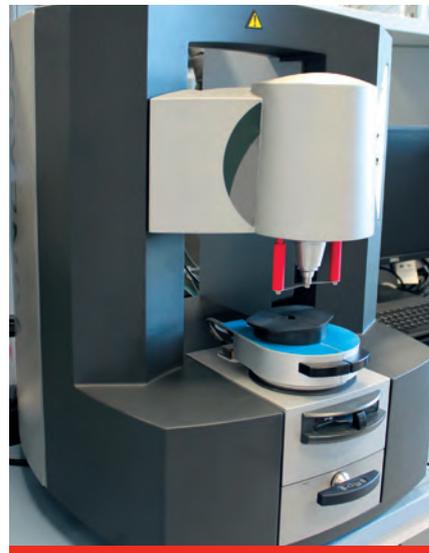


Fig.3.12. Dynamic shear rheometer DSR (photo by: ORLEN Asphalt sp. z o.o., with permission of ORLEN Laboratorium S.A.)

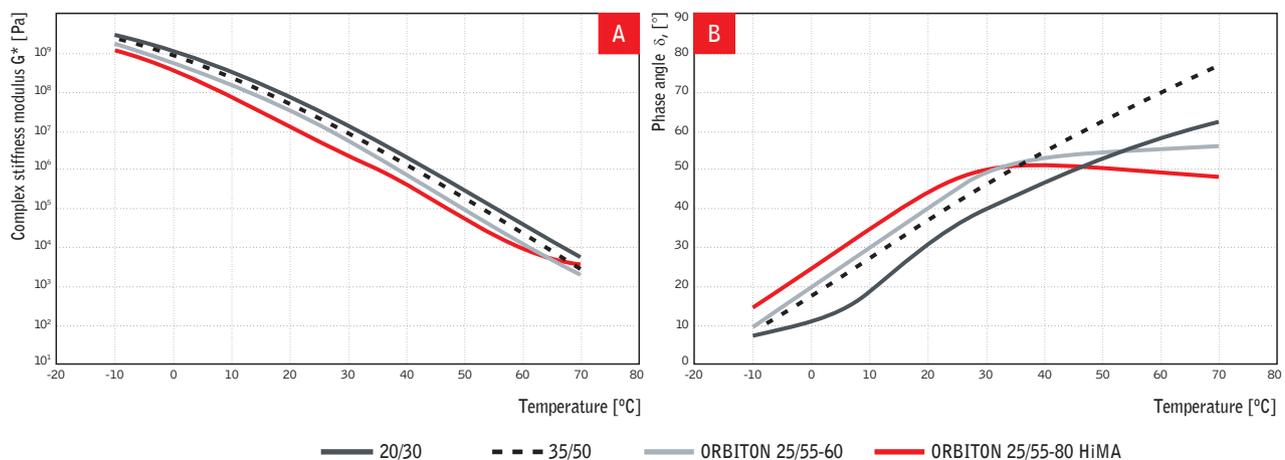


Fig. 3.13. Temperature dependence of a) complex stiffness modulus, b) phase angle for following binders: 20/30, 35/50, PMB 25/55-60 and PMB 25/55-80 HiMA. Samples after ageing using the RTFOT method [source: ORLEN Asphalt internal research]

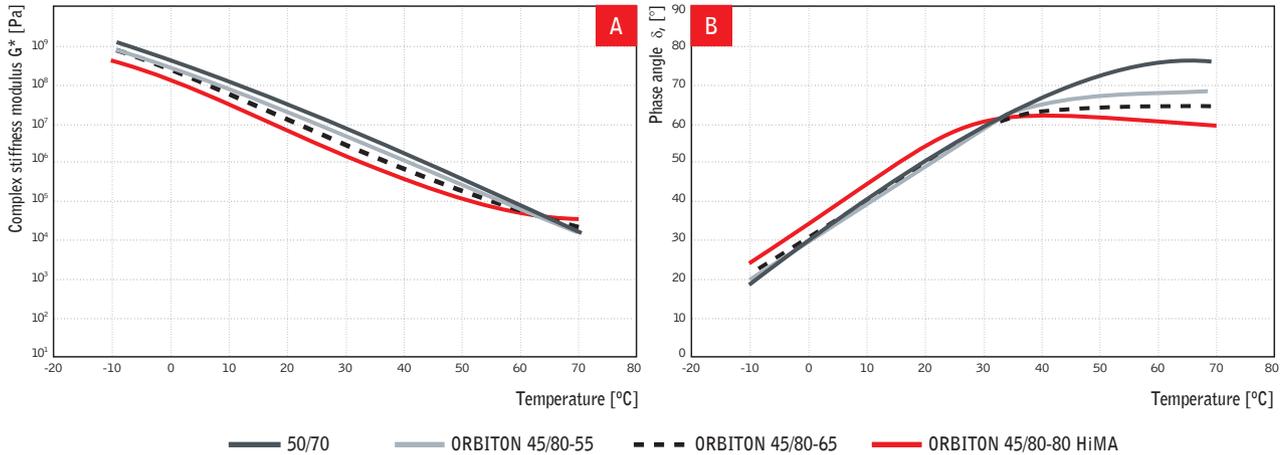


Fig. 3.14.

Temperature dependence of a) complex stiffness modulus, b) phase angle for following binders: 50/70, PMB 45/80-55, PMB 45/80-65 and PMB 45/80-80 HiMA. Samples after ageing using the RTFOT method [source: ORLEN Asphalt internal research]

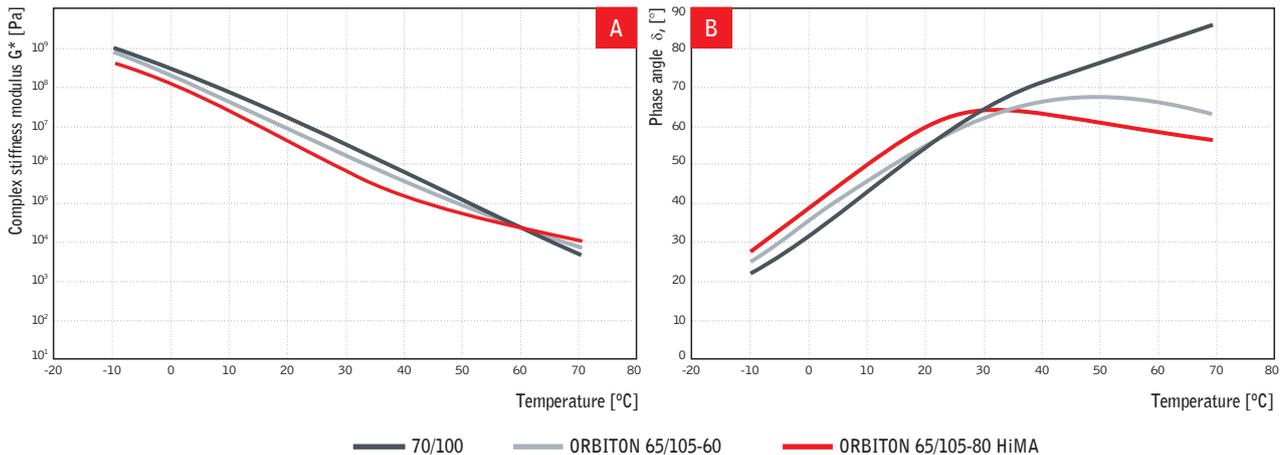


Fig. 3.15.

Temperature dependence of a) complex stiffness modulus, b) phase angle for following binders: 70/100, PMB 65/105-60 and PMB 65/105-80 HiMA. Samples after ageing using the RTFOT method [source: ORLEN Asphalt internal research]

We can see that as the temperature increases, the stiffness of the bitumen decreases, while the phase angle increases.

Considering this in terms of the desired properties of bitumen and asphalt mixtures, we can say that:

a) at low temperatures:

- **the lower the stiffness and the higher the phase angle, the better – preventing pavement cracking in winter,**

b) at high temperatures:

- **the higher the stiffness and the lower the phase angle, the better – preventing rutting.**

Let's look at the graphs showing the characteristics of ORBITON 45/80-80 HiMA (Fig. 3.14.) – we can observe that at -10°C it has the lowest stiffness and the highest phase angle. The low stiffness and the relatively high phase angle mean that the binder has the ability to relax the low-temperature stress in the material, thus protecting it from low-temperature cracking. On the other hand, when analysing the behaviour of this bitumen at $+60^{\circ}\text{C}$, we can observe that it has the highest stiffness and the lowest phase angle. This means that when the pavement is heated to, for example, $+60^{\circ}\text{C}$, the bituminous binder can prevent the formation of ruts.

Among studied bitumens, not surprisingly, different dependencies of stiffness $|G^*|$ and phase angle δ as a function of temperature are apparent. Paving grade bitumens show the highest stiffening with decreasing temperature, which in turn leads to a decrease in the δ . In PMB and PMB HiMA binders, the $|G^*|$ and δ characteristics are altered, obviously influenced by the presence of the polymer network within the binder. The SBS polymers used to modify bitumens belong to the elastomeric group, so they remain elastic even at low temperatures. The desired properties of HiMA bitumens at low temperatures are relatively lower stiffness and a higher phase angle than other binders.

On the other hand, at high temperatures, their stiffness is higher and the phase angle is lower compared to standard PMBs and paving grade bitumens.

Figures 3.16. to 3.18. show the dependence of the complex stiffness modulus $|G^*|$ and the phase angle δ as a function of frequency (in other words – as a function of load time) for all the studied bituminous binders. The measurements were conducted in the frequency range of 0.1–10 Hz at -10, 0, 10, 25, 40, 60, 70°C and then, using the temperature and frequency superposition, master curves for 25°C were obtained.

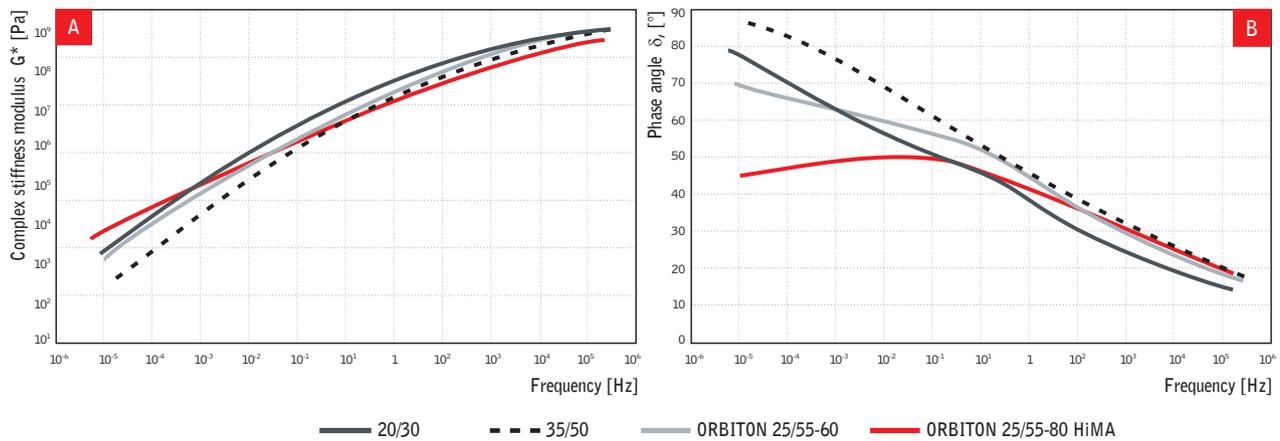


Fig. 3.16. Frequency dependence of a) complex stiffness modulus, b) phase angle for following binders: 20/30, 35/50, PMB 25/55-60 and PMB 25/55-80 HiMA. Samples after ageing using the RTFOT method. Sweep in the frequency range from 0.1 to 10 Hz, reference temperature $T = 25^\circ\text{C}$ [source: ORLEN Asphalt internal research]

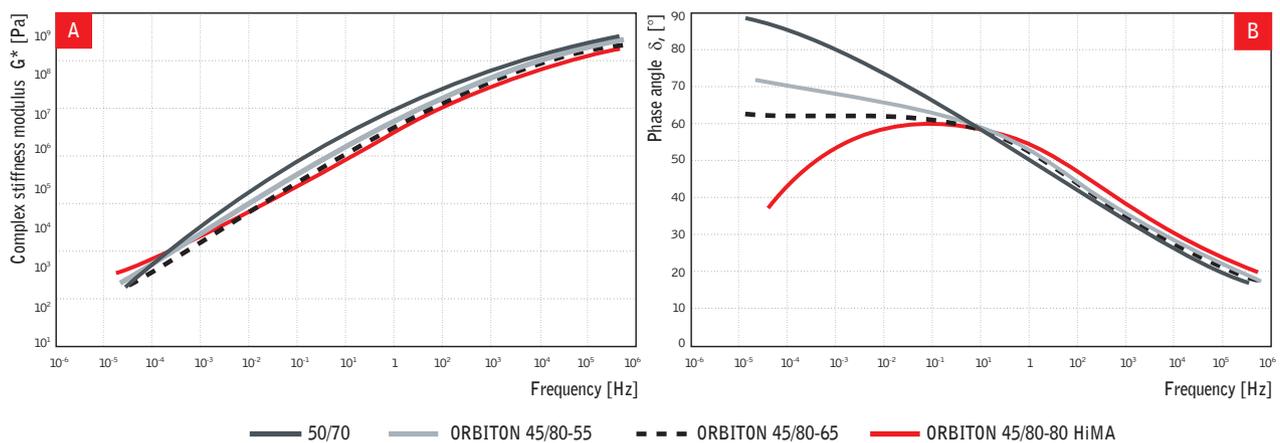


Fig. 3.17. Frequency dependence of a) complex stiffness modulus, b) phase angle for following binders: 50/70, PMB 45/80-55, PMB 45/80-65 and PMB 45/80-80 HiMA. Samples after ageing using the RTFOT method. Sweep in the frequency range from 0.1 to 10 Hz, reference temperature $T = 25^\circ\text{C}$ [source: ORLEN Asphalt internal research]

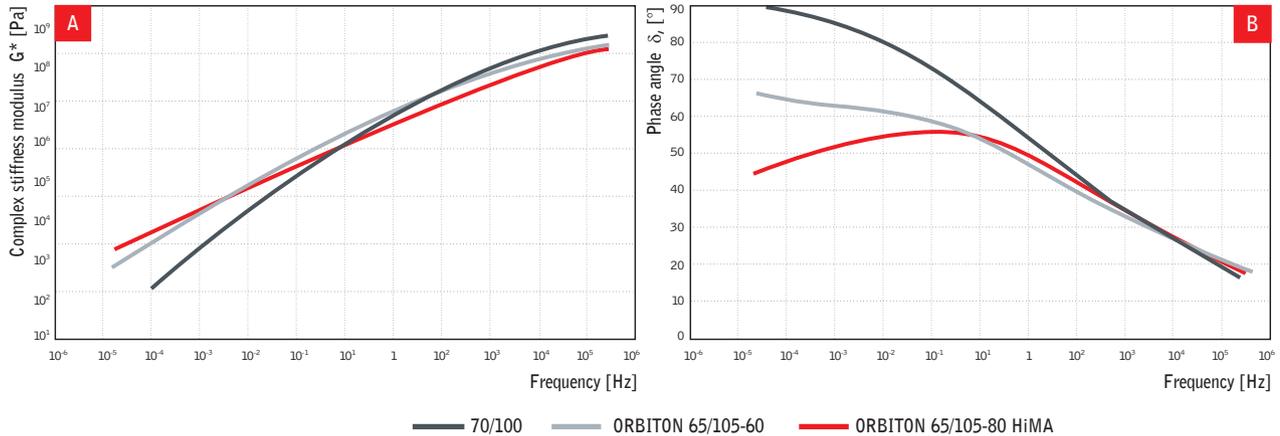


Fig. 3.18. Frequency dependence of a) complex stiffness modulus, b) phase angle for following binders: 70/100, PMB 65/105-60 and PMB 65/105-80 HiMA. Samples after ageing using the RTFOT method. Sweep in the frequency range from 0.1 to 10 Hz, reference temperature $T = 25^{\circ}\text{C}$ [source: ORLEN Asphalt internal research]

It can be seen that as the frequency increases (i.e. vehicle speed increases \rightarrow load time decreases), the complex stiffness modulus increases and the phase angle decreases. **Taking into account the desired properties of bitumens and, consequently, of asphalt mixtures, it is favourable that, especially at low frequency values (at long load times), the bitumen should have a higher stiffness and a lower phase angle.** This is how the elastic part of the bituminous binder works and then is able to prevent the deformation of the pavement in sections particularly exposed to lower speeds of passing vehicles, i.e. slow traffic lanes, parking lots, junction access areas. At high

frequencies (with short load times), the bitumen stiffness is no longer of such great importance as the load time of the pavement by a passing vehicle is much shorter (e.g. at 80 km/h).

The best properties of bituminous binders for slow traffic sections exhibit the PMB HiMA.

Table 3.2 shows the values of the complex stiffness modulus and the phase angle of bituminous binders tested at $+10^{\circ}\text{C}$ at 1 Hz (1 Hz corresponds to approx. 7 km/h) and 10 Hz (10 Hz corresponds to approx. 70 km/h).

Table 3.2. Results of complex stiffness modulus $|G^*|$ and phase angle δ of bituminous binders, samples after RTFOT, temperature 10°C , $f = 1 \text{ Hz}$, 10 Hz , [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | Complex stiffness modulus $ G^* $; 10°C , 1 Hz \cong 7 km/h | Phase angle δ ; 10°C , 1 Hz \cong 7 km/h | Complex stiffness modulus $ G^* $; 10°C , 10 Hz \cong 70 km/h | Phase angle δ ; 10°C , 10 Hz \cong 70 km/h |
|------------------------|---|--|---|--|
| | [MPa] | [$^{\circ}$] | [MPa] | [$^{\circ}$] |
| 20/30 | 22.50 | 32.00 | 48.60 | 27.83 |
| 35/50 | 12.90 | 38.08 | 32.20 | 32.41 |
| 50/70 | 10.80 | 42.80 | 30.20 | 36.01 |
| 70/100 | 9.44 | 46.24 | 27.80 | 38.50 |
| ORBITON 25/55-60 | 15.70 | 37.20 | 38.20 | 31.55 |
| ORBITON 45/80-55 | 8.39 | 43.08 | 23.40 | 36.40 |
| ORBITON 45/80-65 | 9.46 | 42.52 | 25.90 | 36.30 |
| ORBITON 65/105-60 | 5.40 | 48.00 | 17.20 | 40.97 |
| ORBITON 25/55-80 HiMA | 5.56 | 44.71 | 16.40 | 39.61 |
| ORBITON 45/80-80 HiMA | 4.72 | 47.21 | 15.10 | 40.97 |
| ORBITON 65/105-80 HiMA | 3.32 | 52.24 | 11.50 | 45.31 |

Figures 3.19. and 3.20. show graphical comparison of these properties.

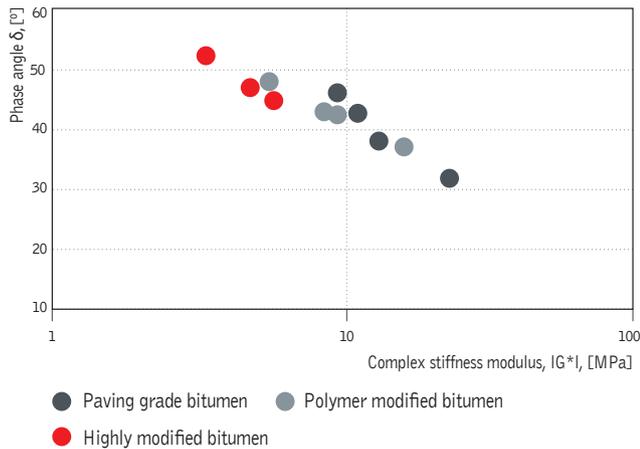


Fig. 3.19. Complex stiffness modulus $|G^*|$ and phase angle δ of the tested bitumens, temperature 10°C, **frequency 1 Hz**. Samples after ageing using the RTFOT method [source: ORLEN Asphalt internal research]

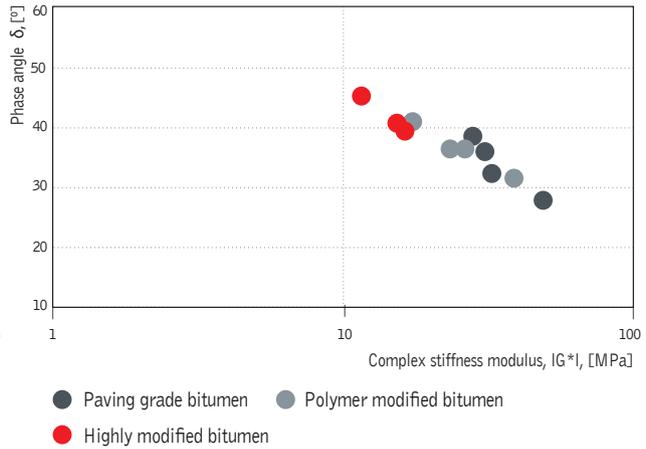


Fig. 3.20. Complex stiffness modulus $|G^*|$ and phase angle δ of the tested bitumens, temperature 10°C, **frequency 10Hz**. Samples after ageing using the RTFOT method [source: ORLEN Asphalt internal research]

3.4.2.2. TESTING OF HIGH-TEMPERATURE PROPERTIES

Tests for high-temperature properties of bituminous binders were performed acc. to AASHTO T315 and ASTM D7175. The results presented hereafter are consistent with the requirements of the US *Performance Grade* specification acc. to AASHTO M320.

According to AASHTO M 320 standard, it is required that binder demonstrates specific parameters tested in the DSR at its expected maximum pavement service temperature ("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 3.3 shows the results of the Upper Critical Temperature (UCT) determined at $G^*/\sin\delta = 1.00$ kPa (samples before RTFOT) and $G^*/\sin\delta = 2.20$ kPa (samples after RTFOT).

Table 3.3.

Test results of high-temperature properties of the bitumens: Upper critical temperature – UCT, determined at $G^*/\sin\delta = 1.00$ kPa and $G^*/\sin\delta = 2.20$ kPa [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | UPPER CRITICAL TEMPERATURE [°C] – UCT | |
|------------------------|---|--|
| | $G^*/\sin\delta = 1.00$ kPa bitumen before RTFOT | $G^*/\sin\delta = 2.20$ kPa bitumen after RTFOT |
| INTERPRETATION | <i>more = better</i> | |
| 20/30 | 83.1 | 84.4 |
| 35/50 | 72.1 | 72.3 |
| 50/70 | 66.9 | 67.1 |
| 70/100 | 62.2 | 62.5 |
| ORBITON 25/55-60 | 79.4 | 77.5 |
| ORBITON 45/80-55 | 75.2 | 71.5 |
| ORBITON 45/80-65 | 80.5 | 76.3 |
| ORBITON 65/105-60 | 80.1 | 71.4 |
| ORBITON 25/55-80 HiMA | 100.5 | 90.9 |
| ORBITON 45/80-80 HiMA | 96.1 | 78.6 |
| ORBITON 65/105-80 HiMA | 93.5 | 79.0 |

In the newer version of the specification, acc. to AASHTO M 332, the requirement $G^*/\sin\delta \geq 2.20$ kPa after RTFOT has been removed and replaced by the requirements of the MSCR method (see 3.4.2.3.). Fig. 3.21. graphically presents the results of the upper critical temperature – UCT, determined at $G^* \sin\delta = 1.00$ kPa (upper bar) and $G^*/\sin\delta = 2.20$ kPa (lower bar). Bitumens were grouped according to penetration ranges corresponding to binder groups for specific paving applications.

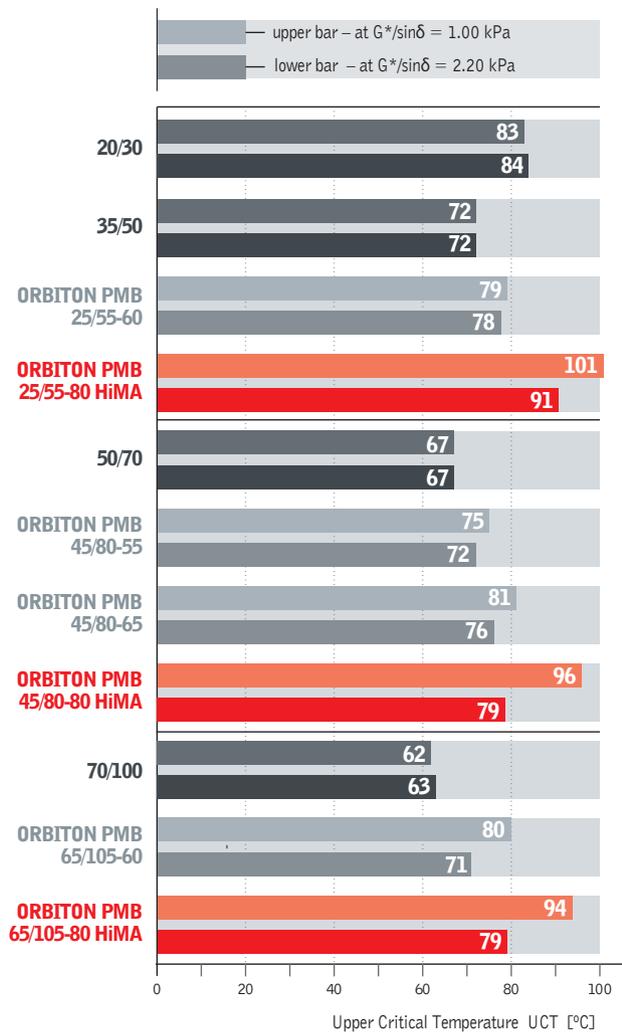


Fig. 3.21. Presentation of upper critical temperature results with $G^*/\sin\delta = 1.00$ kPa – upper bar (bitumens before RTFOT) and with $G^*/\sin\delta = 2.20$ kPa – lower bar (bitumens after RTFOT) [source: ORLEN Asfalt internal research]. Interpretation of the graph: more = better

3.4.2.3. MSCR test

The classification of bituminous binders based on the results of the upper critical temperature UCT used for the indication of the resistance of a given bitumen to permanent deformation turned out to be not fully effective, especially in relation to the polymer modified bitumens [14]. As a result of several studies carried out in the USA, the system of high-temperature classification of bituminous binders was supplemented by the Multiple Stress Creep Recovery (MSCR) test, which resulted in the AASHTO M 332 standard.

According to the 2020 draft standard prEN 14023, the MSCR test has been included in the classification testing of bituminous binders.

The test is performed:

- in Europe, acc. to EN 16659,
- in the USA, acc. to AASHTO T 350 or ASTM D 7405.

The essence of the MSCR test is to measure the binder properties at the highest expected pavement operating temperature (acc. to the American standardisation) or at the specified temperature values e.g. 50°C, 60°C and 70°C, acc. to the European guidelines.

The test is performed in a dynamic shear rheometer on samples after RTFOT ageing. **The results of the test are used to determine the effect of a given bitumen on the rutting resistance of the asphalt mixture and, in the case of PMB, assess the level of elasticity (modification efficiency) of the material.**

The test is conducted for two stress values: 0.1 kPa and 3.2 kPa. The bitumen sample is subjected to constant stress application during 1 second, after which it is relaxed for another 9 seconds. During one test, 10 cycles of creep and recovery are performed at the lower stress value and then the procedure is repeated at the higher stress value (Fig. 3.22.). Thus a total of 20 cycles are performed.

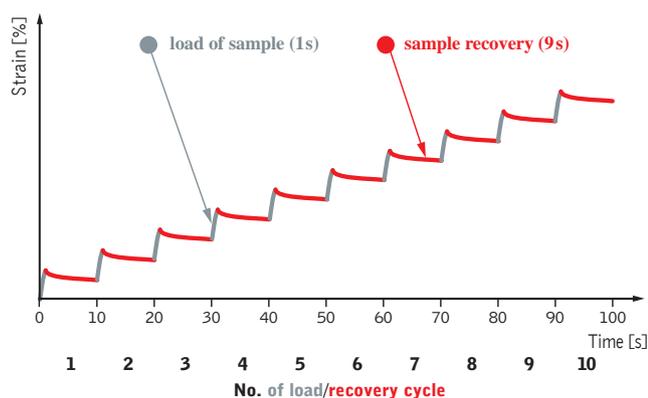


Fig. 3.22. Principle of execution of the MSCR test (10 cycles of creep loads and recovery) for one value of creep stress [own figure]

As a result of the test, the following parameters are obtained:

- **J_{nr} in $[kPa^{-1}]$ – non-recoverable creep compliance** (under cyclic shear conditions in DSR) – a direct indicator of the resistance to rutting of the bituminous binder, determined for two stress levels 0.1 kPa and 3.2 kPa;
- **percent recovery R [%]** – an indicator of the elasticity of the binder at a given temperature, also determined for two stress levels of 0.1 kPa and 3.2 kPa.

Both parameters provide valuable information on a given bituminous binder but **the key to classification is the result $J_{nr,3.2} [kPa^{-1}]$ which determines the resistance of a given bitumen to permanent deformation – the smaller the value of $J_{nr,3.2} [kPa^{-1}]$, the potentially greater resistance to rutting of the asphalt mixture with a given binder.** The percent recovery R provides additional information about the behaviour of the binder at a given temperature and load.

In addition, from the results of J_{nr} and R , two additional parameters, $J_{nr,diff}$ and R_{diff} can be calculated as indicators of the susceptibility of the bituminous binder to increasing load and the change in elasticity of the binder under increasing load, respectively.

The results of the MSCR tests performed at 50°C, 60°C and 70°C are shown in Table 3.4 and Figures 3.23 to 3.24. All bitumen samples were aged by RTFOT prior to MSCR testing. For the highly modified bitumen ORBITON HiMA, the obtained results J_{nr} are so small (i.e. below 0.1) that they do not fall within the precision range of the MSCR method; therefore, $J_{nr,diff}$ values were not calculated for them.

Table 3.4.

MSCR test results – parameters: J_{nr} [kPa^{-1}] and R [%], obtained at a stress of 0.1 and 3.2 kPa, at 50°C, 60°C and 70°C, samples after RTFOT. Interpretation: the lower the J_{nr} value, the higher the resistance to rutting and the higher the recovery R , the more elastic the binder [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | TEMPERATURE 50°C | | | | | | TEMPERATURE 60°C | | | | | | TEMPERATURE 70°C | | | | | |
|----------------|------------------|-------------|------------|--------------|----------------|---------------|------------------|-------------|------------|--------------|----------------|---------------|------------------|-------------|------------|--------------|----------------|---------------|
| | R 0.1 | R 3.2 | R_{diff} | $J_{nr,0.1}$ | $J_{nr,3.2}$ | $J_{nr,diff}$ | R 0.1 | R 3.2 | R_{diff} | $J_{nr,0.1}$ | $J_{nr,3.2}$ | $J_{nr,diff}$ | R 0.1 | R 3.2 | R_{diff} | $J_{nr,0.1}$ | $J_{nr,3.2}$ | $J_{nr,diff}$ |
| | [%] | [%] | [%] | kPa | kPa | [%] | [%] | [%] | [%] | kPa | kPa | [%] | [%] | [%] | [%] | kPa | kPa | [%] |
| 20/30 | 63.3 | 63.6 | -0.3 | <0.1 | <0.1 | – | 47.2 | 45.9 | 2.9 | 0.1 | 0.1 | 2.2 | 31.1 | 23.9 | 23.4 | 0.3 | 0.3 | 12.3 |
| 35/50 | 48.3 | 47.8 | 1.0 | <0.1 | <0.1 | – | 29.0 | 25.4 | 12.5 | 0.2 | 0.2 | 5.5 | 16.5 | 8.7 | 47.6 | 0.8 | 0.9 | 14.8 |
| 50/70 | 19.3 | 17.9 | 7.4 | 0.2 | 0.2 | 1.8 | 9.8 | 3.7 | 62.9 | 1.2 | 1.3 | 11.7 | 3.2 | -0.5 | 115.8 | 4.4 | 5.0 | 13.4 |
| 70/100 | 11.1 | 8.2 | 26.3 | 0.5 | 0.6 | 4.6 | 3.1 | 0.5 | 83.9 | 2.9 | 3.1 | 9.2 | – | – | – | – | – | – |
| 25/55-60 | 75.5 | 75.4 | 0.1 | <0.1 | <0.1 | – | 69.0 | 67.9 | 1.5 | 0.1 | 0.1 | 4.0 | 58.8 | 52.4 | 10.9 | 0.3 | 0.4 | 20.1 |
| 45/80-55 | 77.0 | 75.9 | 1.5 | 0.1 | 0.1 | 6.0 | 73.8 | 72.6 | 1.7 | 0.2 | 0.2 | 7.2 | 67.9 | 66.3 | 5.0 | 0.4 | 0.5 | 15.8 |
| 45/80-65 | 82.5 | 82.1 | 0.5 | <0.1 | <0.1 | – | 83.1 | 82.3 | 1.0 | 0.1 | 0.1 | 9.5 | 80.0 | 76.0 | 5.0 | 0.4 | 0.5 | 28.7 |
| 25/55-80 HiMA | 93.4 | 91.6 | 1.9 | <0.1 | <0.1 | – | 92.8 | 92.1 | 0.8 | <0.1 | <0.1 | – | 92.9 | 92.4 | 0.6 | <0.1 | <0.1 | – |
| 45/80-80 HiMA | 92.8 | 92.8 | 0.0 | <0.1 | <0.1 | – | 96.0 | 96.0 | 0.0 | <0.1 | <0.1 | – | 96.1 | 96.1 | 0.0 | <0.1 | <0.1 | – |
| 65/105-80 HiMA | 96.2 | 96.8 | -0.6 | <0.1 | <0.1 | – | 98.0 | 98.5 | -0.6 | <0.1 | <0.1 | – | 98.3 | 98.4 | -0.2 | <0.1 | <0.1 | – |

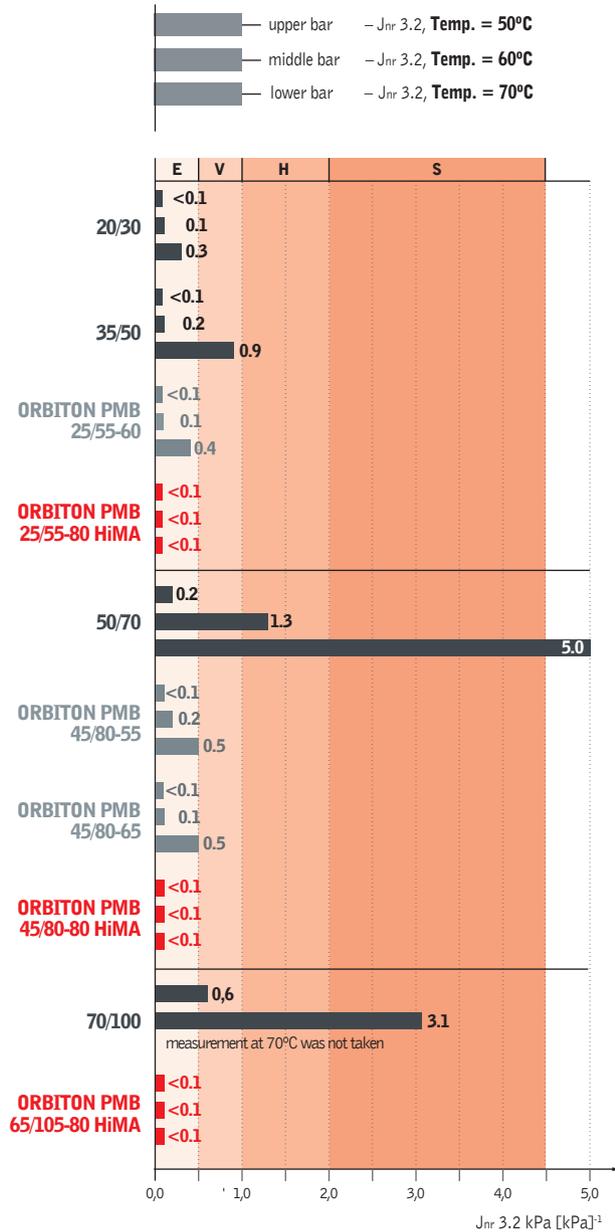


Fig. 3.23. Presentation of the results of the J_{nr} parameter at a stress of 3.2 kPa at 50°C, 60°C, 70°C for the tested bitumens. Determined traffic categories, acc. to Table 3.5. [source: ORLEN Asphalt internal research]. Interpretation of graph: lower value of $J_{nr, 3.2}$ = better

Based on the J_{nr} results, an additional classification was introduced in AASHTO M 332, in which the maximum traffic load for a given bituminous binder is specified using letter symbols.

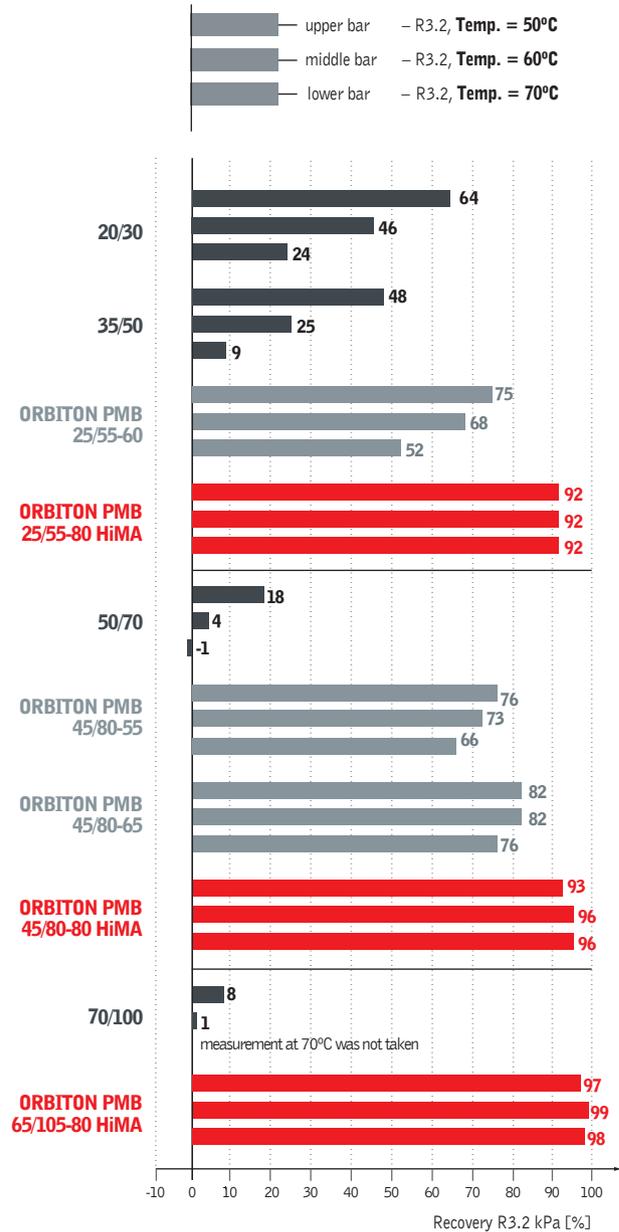


Fig. 3.24. Presentation of results of recovery R at the stress of 3.2 kPa at 50°C, 60°C, 70°C, for the tested bitumens [source: ORLEN Asphalt internal research]. Interpretation of the graph: higher value of R3.2 = better

The suitability of the bitumen for a given traffic category is assessed on the basis of the parameter $J_{nr, 3.2}$ [kPa⁻¹] and $J_{nr, diff}$. The classification of requirements for the tested bitumens is shown in Table 3.5.

Table 3.5.

Classification of bituminous binders and requirements for traffic volume and characteristics acc. to AASHTO M 332 and AASHTO T 350

| TRAFFIC CATEGORY (letter code) | LOAD (the number of equivalent, standard axes and traffic conditions) | REQUIRED FOR THE BINDER AT THE UPPER PG TEMPERATURE | |
|-----------------------------------|---|--|---|
| | | Requirement for $J_{nr, 3.2}$ | Additional requirements for $J_{nr, diff}$ |
| S – Standard | <10 million axles (ESAL) and standard traffic (>70 km/h) | ≤ 4.5 | |
| H – Heavy | 10–30 million axles (ESAL) or slow traffic (20–70 km/h) | ≤ 2.0 | ≤ 75% |
| V – Very heavy | >30 million axles (ESAL) or parking of the vehicles (<20 km/h) | ≤ 1.0 | |
| E – Extreme | >30 million axles (ESAL) and parking of the vehicles (<20 km/h) | ≤ 0.5 | |

*) binder sensitivity to stress change

ESAL – Equivalent Single Axle Load – US comparison axle with 80 kN (18,000 lbs) load and a twin wheel

Table 3.6 shows the classification of bituminous binders after MSCR testing for suitability for a given traffic category – based on data from Tables 3.4 and 3.5.

Table 3.6.

Binder classification after the MSCR test by traffic load [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | FINAL CLASSIFICATION OF SUITABILITY FOR ROAD TRAFFIC (AT TESTING TEMPERATURE) ACC. TO PG CLASSIFICATION | | |
|------------------------|--|------------------|------------------|
| | TEMPERATURE 50°C | TEMPERATURE 60°C | TEMPERATURE 70°C |
| Paving grade 20/30 | E | E | E |
| Paving grade 35/50 | E | E | V |
| Paving grade 50/70 | E | H | S |
| Paving grade 70/100 | V | S | –* |
| ORBITON 25/55-60 | E | E | E |
| ORBITON 45/80-55 | E | E | V |
| ORBITON 45/80-65 | E | E | V |
| ORBITON 25/55-80 HiMA | E | E | E |
| ORBITON 45/80-80 HiMA | E | E | E |
| ORBITON 65/105-80 HiMA | E | E | E |

E – Extreme traffic
V – Very heavy traffic

H – Heavy traffic

S – Standard traffic

* result for $J_{nr, 3.2}$ kPa outside classification (>4.5)

3.4.2.4. TESTING OF PROPERTIES AT INTERMEDIATE TEMPERATURE

Dynamic shear rheometer DSR is used to test the intermediate temperature properties of the so-called fatigue properties. The tests are performed according to AASHTO T315.

The resistance of bituminous binders to fatigue cracking is tested at what is known as the intermediate pavement service temperature, depending on the PG type of the bitumen. The bitumen samples are previously aged using RTFOT and PAV. Fatigue Cracking Critical Temperature (FCCT) can be interpreted as the temperature from which the pavement becomes so stiff that there is a risk of

fatigue cracking. This means that the lower the FCCT, the longer the binder retains the desired anti-fatigue properties.

The requirements of the Performance Grade system, acc. to AASHTO M 332, limit the stiffness of a bituminous binder, defined as the product of the parameters: $G^* \cdot \sin\delta$ to a maximum of 5000 kPa (for S traffic) and 6000 kPa (for H, V, E traffic) at the frequency of $f = 10$ rad/s.

Table 3.7 shows the FCCT results for the tested bitumens.

Table 3.7.

Results of critical temperature tests for fatigue cracking of the bituminous binders, determined at $G^* \cdot \sin\delta = 5000$ kPa and $G^* \cdot \sin\delta = 6000$ kPa [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | FATIGUE CRACKING CRITICAL TEMPERATURE – FCCT [°C] | |
|------------------------|--|--|
| | $G^* \cdot \sin\delta = 5000$ kPa bitumen after RTFOT + PAV | $G^* \cdot \sin\delta = 6000$ kPa bitumen after RTFOT + PAV |
| INTERPRETATION | <i>less = better</i> | |
| 20/30 | 25.6 | 23.4 |
| 35/50 | 21.9 | 19.8 |
| 50/70 | 20.1 | 18.1 |
| 70/100 | 18.0 | 16.7 |
| ORBITON 25/55-60 | 22.2 | 20.2 |
| ORBITON 45/80-55 | 16.1 | 14.3 |
| ORBITON 45/80-65 | 17.1 | 15.5 |
| ORBITON 65/105-60 | 14.2 | 12.6 |
| ORBITON 25/55-80 HiMA | 13.2 | 11.1 |
| ORBITON 45/80-80 HiMA | 12.9 | 11.5 |
| ORBITON 65/105-80 HiMA | 11.1 | 9.6 |

Fig. 3.25. shows a comparison of the intermediate temperature properties of all the tested bituminous binders. For easier analysis, the bitumens were grouped acc. to penetration ranges.

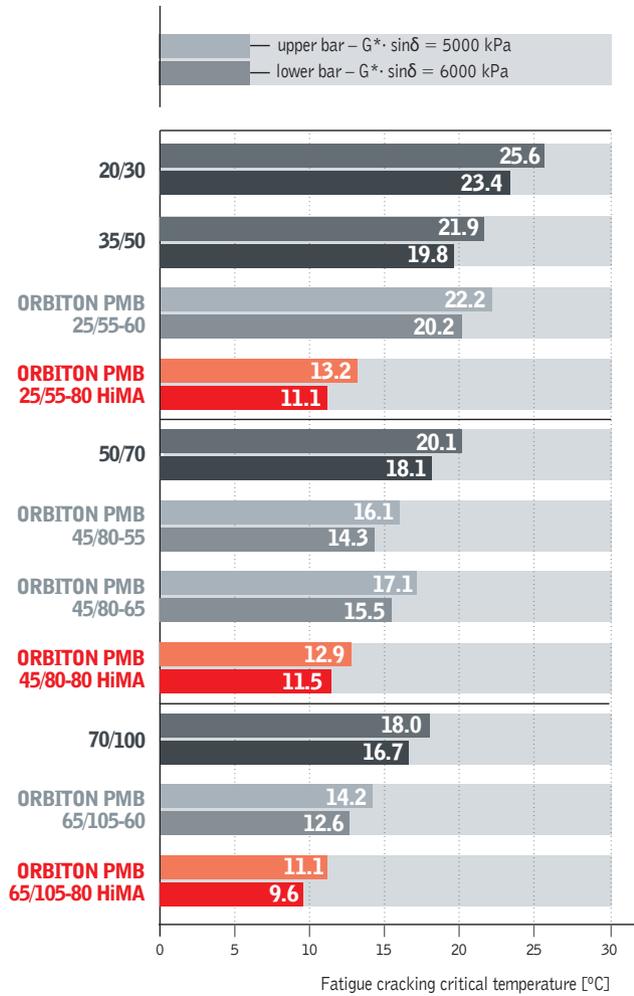


Fig. 3.25. Presentation of FCCT results, with $G^* \cdot \sin\delta = 5000$ kPa (upper bar) and $G^* \cdot \sin\delta = 6000$ kPa (lower bar). Samples after RTFOT + PAV [source: ORLEN Asphalt internal research]. Interpretation of the graph: less = better

The method of checking the fatigue critical temperature FCCT is only comparative. It has been proven that it does not describe the real fatigue phenomena occurring in bituminous binders, such as: changes of strain or different frequency of the applied load [16, 17]. Therefore, there is an ongoing worldwide search for a suitable method of predicting the fatigue properties of bituminous binders. One such method is the Linear Amplitude Sweep Test described in Chapter 4.

3.4.3. CLASSIFICATION OF BITUMINOUS BINDERS ACC. TO PERFORMANCE GRADE SYSTEM

Table 3.8 shows the grades of tested bituminous binders acc. to the Performance Grade system based on AASHTO M 320 (without MSCR), while Table 3.9 shows the grades acc. to AASHTO M 332 (including MSCR).

Table 3.8.

Classification of tested bitumens according to Performance Grade system AASHTO M 320 [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | REAL PERFORMANCE GRADE (real, continuous PG) | PERFORMANCE GRADE (PG) |
|------------------------|--|----------------------------------|
| 20/30 | 84-23 | 82-22 |
| 35/50 | 72-25 | 70-22 |
| 50/70 | 67-26 | 64-22 |
| 70/100 | 62-26 | 58-22 |
| ORBITON 25/55-60 | 77-25 | 76-22 |
| ORBITON 45/80-55 | 71-29 | 70-28 |
| ORBITON 45/80-65 | 76-29 | 76-28 |
| ORBITON 65/105-60 | 71-30 | 70-28 |
| ORBITON 25/55-80 HiMA | 90-28 | 88-28 |
| ORBITON 45/80-80 HiMA | 79-31 | 76-28 |
| ORBITON 65/105-80 HiMA | 79-31 | 76-28 |

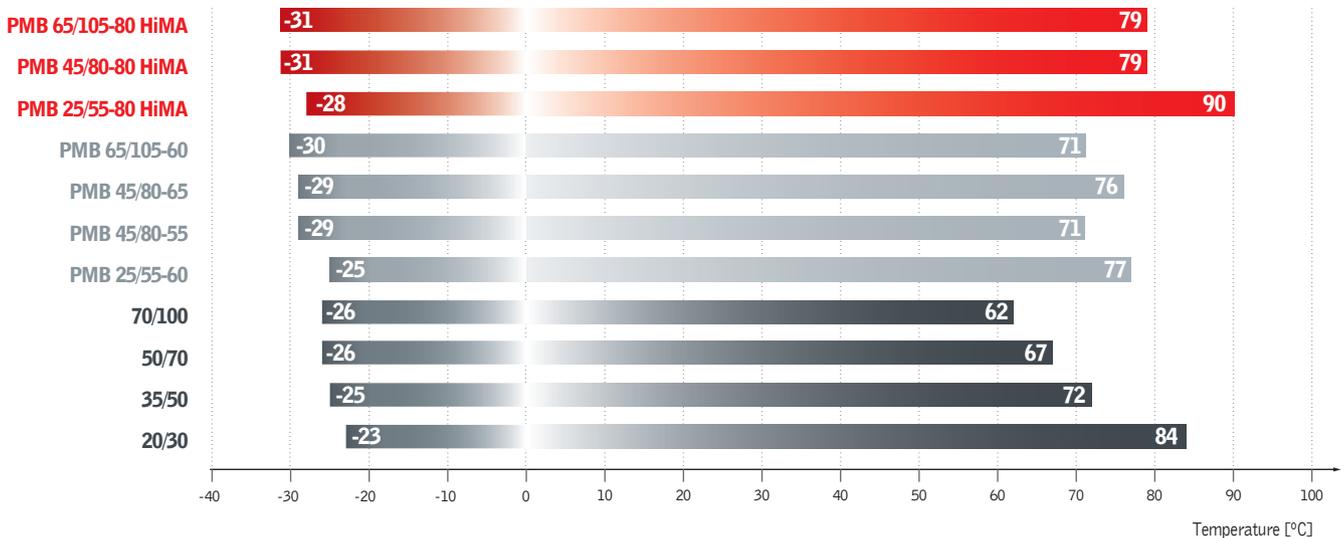


Fig. 3.26.

Graphical representation of the classification of bituminous binders – real PG acc. to AASHTO M 320 [source: ORLEN Asphalt internal research]

Table 3.9.

Classification of the tested bitumens according to Performance Grade system AASHTO M 332 [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | PERFORMANCE GRADE | BITUMENS MEETING THE REQUIREMENTS OF AASHTO M 332 FOR UCT CORRESPONDING TO POLISH CLIMATIC CONDITIONS |
|------------------------|-------------------|---|
| 20/30 | 82-22 | 50E-22, 60E-22, 70E-22 |
| 35/50 | 70-22 | 50E-22, 60E-22, 70V-22 |
| 50/70 | 64-22 | 50E-22, 60H-22, 70S-22 |
| 70/100 | 58-22 | 50V-22, 60S-22 |
| ORBITON 25/55-60 | 76-22 | 50E-22, 60E-22, 70E-22 |
| ORBITON 45/80-55 | 70-28 | 50E-28, 60E-28, 70V-28 |
| ORBITON 45/80-65 | 76-28 | 50E-28, 60E-28, 70V-28 |
| ORBITON 25/55-80 HiMA | 88-28 | 50E-28, 60E-28, 70E-28 |
| ORBITON 45/80-80 HiMA | 76-28 | 50E-28, 60E-28, 70E-28 |
| ORBITON 65/105-80 HiMA | 76-28 | 50E-28, 60E-28, 70E-28 |

The range of available bituminous binders from ORLEN Group shown in Table 3.9. was determined based on the requirements of AASHTO M 332, on the basis of a set of requirements supplemented with the results of the MSCR test (Table 3.6.). The Performance Grade column represents the nominal temperature range of application for a given binder, determined by the lower (LCT) and upper (UCT) critical temperature. The next column lists the available binders in relation to the climatic conditions of Poland, with the maximum pavement temperature range between 50°C and 70°C. For this range, binders were matched by giving their PGs supplemented with traffic classifications (S, H, V, E – based on the data from Table 3.6.).

This means that, for example, the paving grade bitumen 50/70 at a pavement temperature up to the conventional +60°C is suitable for H (heavy) traffic, but if the pavement temperature rises above +60°C, it is only suitable for S (standard) traffic.

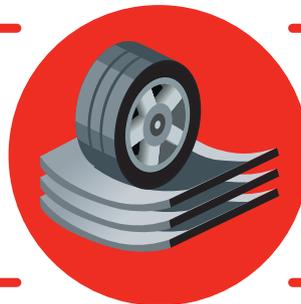
For the pavement temperature range of +50°C to +60°C, the majority of modified bitumens can be used for E (extreme) traffic. Of all the tested binders, only highly modified bitumen ORBITON HiMA meets the requirements for traffic E (extreme) in the entire temperature range of +50°C to +70°C.

4. FATIGUE PROPERTIES OF BITUMINOUS BINDERS

Fatigue cracking is one of the main factors affecting the durability of asphalt pavements. It is subject of a large number of factors, such as the composition and properties of the asphalt mixture and the properties of the applied bituminous binder [1].

Fatigue testing of asphalt mixtures is standardised based on EN 12697-24, while the fatigue resistance of bituminous binders can be tested using a dynamic shear rheometer (DSR). This chapter discusses the latest results of fatigue tests on bituminous binders carried out by ORLEN Asphalt.

4.1. EFFECT OF FATIGUE IN ASPHALT PAVEMENTS



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.

The conventional structure of the asphalt pavement consists of a set of asphalt layers (usually two to three, sometimes one) – wearing course, binder course and base course, which lie on

an aggregate base and compacted 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. 4.1.) occur in the bottom of the lowest asphalt layer. Tensile strain present in the bottom of the lowest asphalt layer (usually the base course) is regarded as the critical strain ϵ_t which defines the fatigue life of the structure. Therefore, the type of the bituminous binder applied in this bottom course is very important.

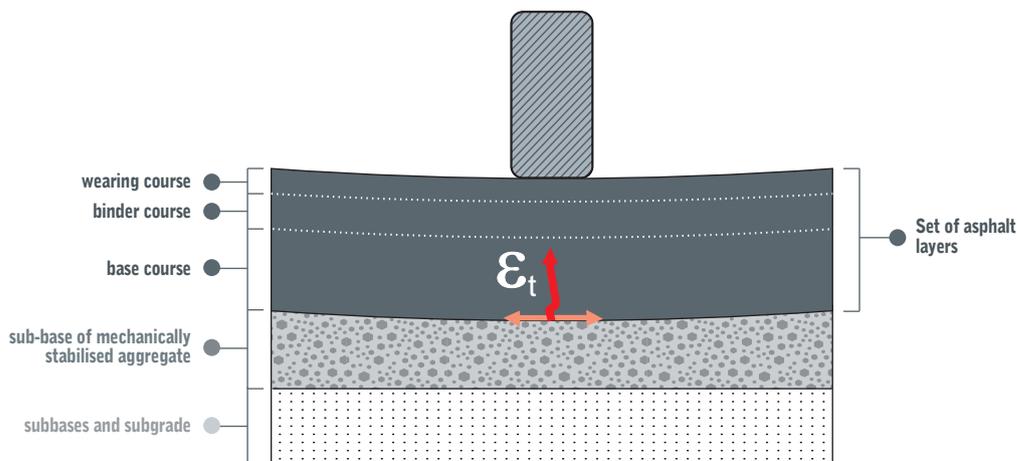


Fig. 4.1. Arrangement of layers of flexible pavement and location of occurrence of critical tensile strain [own figure]

Fatigue cracking in the asphalt pavement is caused by the accumulation of single deformations induced by loads from the wheels of passing vehicles. A single deformation does not initiate a crack in the asphalt pavement, but the accumulation of numerous load cycles results in the increase of the so-called fatigue damage in the asphalt mixture, eventually initiating a crack and starting the pavement degradation process [3]. The fatigue cracks can occur within the binder or at the binder-aggregate interface [2].

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 axles (number of deflections) that the pavement can carry in a defined period of time. This is how the pavement structure is dimensioned to guarantee the required service life specified in the technical regulations (usually 20 or 30 years).

4.2. FATIGUE TESTING OF BITUMINOUS BINDERS



In the literature, a number of dynamic shear rheometer DSR tests have been described, that can be used for testing the fatigue

life of bituminous binders. A description of some of the most popular ones is given below.

4.2.1. FATIGUE CRACKING CRITICAL TEMPERATURE – FCCT

Currently, the best known method for fatigue life testing of bituminous binders is the Fatigue Cracking Critical Temperature (FCCT) method.

The FCCT test was developed in the United States as part of the Superpave system. It uses the $|G^*| \cdot \sin \delta$ parameter to quantify the fatigue resistance of bituminous binders. The test is performed in accordance with AASHTO T315, on samples after RTFOT and PAV, in the intermediate temperature which depends on the PG grade of a particular binder. The test is executed in DSR at a very low strain amplitude (1%), at a frequency of $f = 10$ [rad/sec]. The fatigue cracking critical temperature FCCT can be interpreted as the temperature in which the pavement becomes so stiff that there is a risk of fatigue cracking. This means that the lower

the FCCT, the longer the binder retains the desired anti-fatigue properties.

The requirements of the Performance Grade system, acc. to AASHTO M 332, limit the stiffness of the bituminous binder, defined as the product of the parameters $|G^*| \cdot \sin \delta$ to a maximum of 5000 kPa (for Standard grade) and 6000 kPa (for traffic: Heavy, Very heavy, Extreme grades).

The main disadvantage of the FCCT test is that it does not describe the real fatigue phenomena occurring in the bituminous binders, such as: changing strain or different level of the frequency of applied load [4, 5]. More detailed information on the FCCT method as well as test results can be found in Chapter 3 (see Section 3.4.2.4.).

4.2.2. TIME SWEEP TEST

Another commonly used method to determine the fatigue properties of bituminous binders is the time sweep test, which is also carried out using a dynamic shear rheometer (DSR).

The time sweep test method is based on the cyclic loading of the bituminous binder sample at a constant frequency, typically 10 Hz, and at a variable strain amplitude. The time sweep test

was developed by the *National Cooperative Highway Research Program* (NCHRP) project [4] to improve the fatigue life testing methodology described in AASHTO T315.

The advantage of the time sweep test is that the amplitude of strain is freely selectable, which allows, among other things, the pavement structure and traffic load to be taken into account.

4.2.3. LINEAR AMPLITUDE SWEEP TEST

Due to the continuing debate in the scientific community on the adequacy of the methods described above, work has been ongoing in the USA to develop a new method for testing the fatigue properties of bituminous binders.

The result of this work is the method described in AASHTO TP 101-14 – *Estimating Damage Tolerance of Asphalt Binders Using the Linear Amplitude Sweep* (abbreviation LAS).

The LAS test is performed using a dynamic shear rheometer. It involves cyclic loading a test sample at a constant frequency with progressively increasing amplitude to induce fatigue damage in the sample at an accelerated rate.

The LAS test starts with 100 initial cycles of sinusoidal loading at the amplitude of 0.1% and a frequency of 10 Hz. The frequency of 10 Hz remains constant throughout the test. The amplitude increases in 1% steps until a level of 30% is reached. Each level of amplitude increase contains 100 load cycles, so that a total of 3100 load cycles are applied to the test material.

The complex stiffness modulus $|G^*|$, phase angle δ , shear stress in the sample and associated shear strains are recorded as the test results. **The fatigue life, N_f , is calculated at the strain level of the sample, which corresponds to the maximum value of the stress occurring** [6, 8].

The AASHTO TP101-14 standard assumes that the theoretically arbitrary strain value γ_{max} can be chosen and its effect on the N_f value can be monitored. However, since it has been shown that the deformation occurring in the bituminous binder is about 50 times higher than the level of deformation acting in the asphalt mixture, it is recommended to calculate the fatigue parameter N_f for two strain levels: $\gamma=2.5\%$ and $\gamma=5.0\%$ [9].

The LAS test should be performed at an intermediate temperature determined according to the PG grade of the bitumen. The test sample can be aged by RTFOT or RTFOT + PAV.

4.3. TEST PROGRAMME



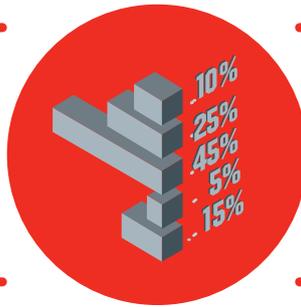
In the research programme conducted by the Research, Development and Innovation Department of ORLEN Asphalt in 2019, the following types of bitumen were used for fatigue properties test:

- Paving grade bitumen:
35/50, 50/70;
- Polymer modified bitumen:
ORBITON 25/55-60, ORBITON 45/80-55;

- Highly modified bitumen:
ORBITON 25/55-80 HiMA, ORBITON 45/80-80 HiMA, ORBITON 65/105-80 HiMA.

Testing according to the LAS methodology at 10°C on samples aged in RTFOT was performed at the ORLEN UniCRE Research and Development Centre in Czech Republic.

4.4. TEST RESULTS



As a result of the study, fatigue characteristics of the tested bituminous binders were obtained, Fig. 4.2. The value of the N_f parameter was calculated at two recommended strain levels: $\gamma = 2.5\%$ and $\gamma = 5.0\%$

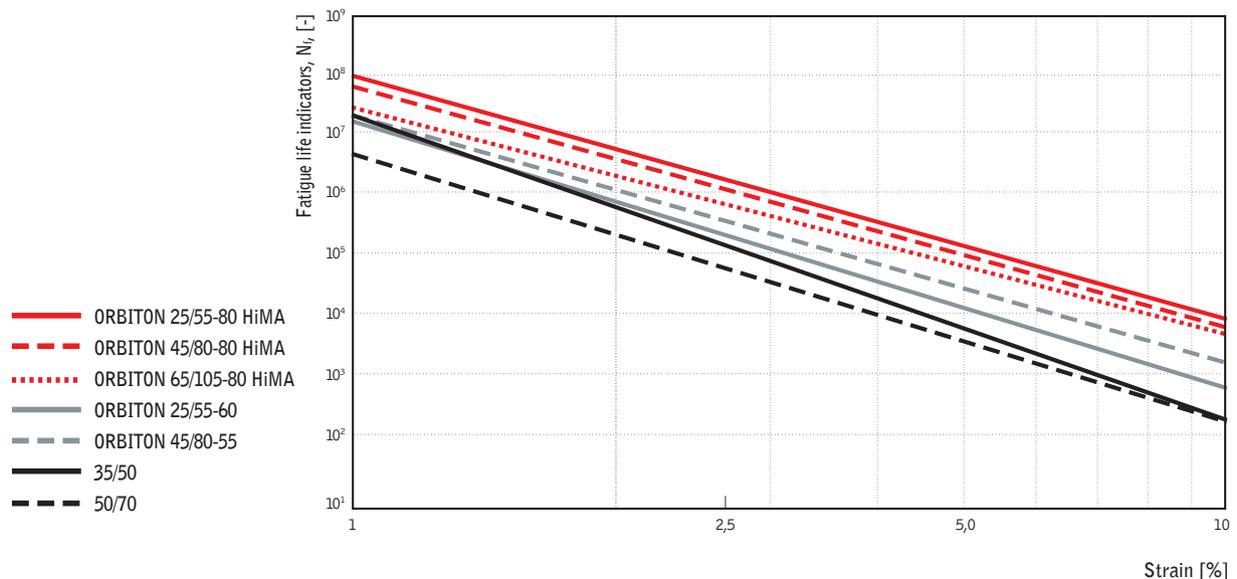


Fig. 4.2.

Comparison of fatigue characteristics of the tested bituminous binders, samples after RTFOT, test temperature 10°C, [source: ORLEN Asphalt internal research]

Comparing the fatigue curves of the tested bituminous binders, it can be observed that the highly modified bitumen ORBITON HiMA are characterized by the best fatigue properties at both applied strain: $\gamma = 2.5\%$ and $\gamma = 5.0\%$. A major role in this behaviour of HiMA binders is played by the dominance of the polymer phase over the bitumen phase and the continuity of the elastomeric network, which essentially enhances the tensile strength of these binders and consequently increases the fatigue life.

As expected, paving grade bitumens show the weakest fatigue properties. Bitumens modified with a standard amount of SBS polymer possess intermediate properties.

Researchers from the USA [11] proposed to include the fatigue life results obtained in the LAS test as an additional classification property of bituminous binders in the Performance Grade system.

They proposed to divide the results obtained in relation to the thickness of the asphalt layer in which a given binder is to be used [11]. Based on the conducted tests, it was found that:

- the strain $\gamma = 2.5\%$ corresponds to asphalt pavements >10 cm thick, so-called strong pavements,
- the strain $\gamma = 5.0\%$ corresponds to asphalt pavements <10 cm thick, so called weak pavements.

Colloquially speaking, these are thin (→ weak) and normal (→ strong) asphalt structures.

A summary of the fatigue life requirements acc. to the LAS test in relation to traffic volume is provided in Table 4.1.

Table 4.1.

Additional classification of bituminous binders and requirements for traffic volume and its characteristics, based on the LAS test [32]

| TRAFFIC CATEGORY (letter code) | REQUIREMENT FOR BINDER AT TEST TEMPERATURE, ACC. TO LAS TEST, AASHTO TP 101-14 | |
|---|---|--|
| | N_f at 2.5% strain for asphalt layers > 10 cm | N_f at 5.0% strain for asphalt layers < 10 cm |
| S – standard | > 15 000 | > 15 000 |
| H – heavy | > 19 000 | > 19 000 |
| V – very heavy E – extreme | > 31 000 | > 31 000 |

Table 4.2 shows the classification of the tested bitumen with respect to their suitability for a given traffic category, based on the data from Table 4.1.

Table 4.2.

Bitumens classification after the LAS testing according to traffic volume [source: ORLEN Asphalt internal research]

| BITUMEN TYPE | FINAL CLASSIFICATION OF ROAD TRAFFIC SUITABILITY ACC. TO PG CLASSIFICATION, TEMPERATURE 10°C, SAMPLES AFTER RTFOT | |
|----------------------------|--|--|
| | N_f at 2.5% strain for asphalt layers > 10 cm | N_f at 5.0% strain for asphalt layers < 10 cm |
| Paving grade bitumen 35/50 | V/E | • |
| Paving grade bitumen 50/70 | V/E | • |
| ORBITON 25/55-60 | V/E | • |
| ORBITON 45/80-55 | V/E | H |
| ORBITON 25/55-80 HiMA | V/E | V/E |
| ORBITON 45/80-80 HiMA | V/E | V/E |
| ORBITON 65/105-80 HiMA | V/E | V/E |

E – Extreme; V – Very heavy; H – Heavy; S – Standard
• result outside classification

Analysing the data from Table 4.2, it can be stated that for the pavements of the thickness of the asphalt layers exceeding 10 cm (N_f criterion for strain $\gamma = 2.5\%$) – the tested bitumens meet the adopted criteria for very heavy and extreme traffic.

This is rather obvious, as thick pavements characterise with good load-bearing capacity and fatigue life. Also the paving grade bitumens, which are the most susceptible to fatigue, show good results under the formulated load conditions.

For pavements which thickness of the asphalt layers does not exceed 10 cm (N_f criterion for strain $\gamma = 5.0\%$), i.e. for large deflections and large critical deformation, only bitumens with polymer additives are able to withstand such load. These are

either the conventional polymer modified bitumens – ORBITON 45/80-55 or all highly modified bitumens ORBITON HiMA, which meet the criteria for extreme traffic. It is worth noting that the polymer modified bitumen ORBITON 25/55-60 (hard) did not meet the criteria, as in such a thin asphalt layer requires flexibility and elasticity and this is a property of softer binders. This dependence did not occur with highly modified bitumens HiMA which, due to their reversed phase, retain elasticity and compliance even at lower penetration.

This confirms the results obtained at the NCAT *Pavement Test Track* in the USA, where a reduced thickness pavement designed with highly modified bitumen HiMA has been shown to resist fatigue cracking [12, 13].

4.5. CONCLUSIONS



- The linear amplitude sweep (LAS) test is a practical method for evaluating the fatigue life of bituminous binders. This method allows to estimate the complex behaviour of binders over a wide range of applied loads (amplitude).
- From the presented results, it can be clearly concluded that the best fatigue properties is achieved by the highly modified bitumens ORBITON HiMA. As expected, paving grade bitumens show the weakest fatigue properties. Conventional modified bitumens are characterised by intermediate properties.
- The results of the LAS test for bituminous binders match the results obtained in a comparative study by ORLEN Asphalt for asphalt mixtures. The order of binder groups remained the same, from the best HiMA through conventional PMB to paving grade (non-modified) bitumens [3, 24].

5. BITUMEN APPLICATION TECHNOLOGY

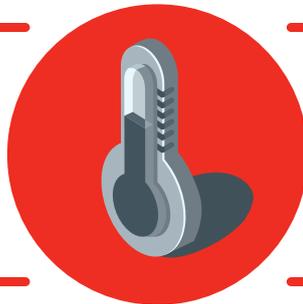
Bituminous binders are thermoplastic materials, i.e. they change their consistency as the temperature changes. The correct use of bitumens requires, above all, knowledge about the optimum temperature during the whole technological process, starting from the delivery of the bitumen from the refinery to its application in the road pavement.

The following sections of this chapter present a range of important information related to the process temperature

of bitumens, correct handling of samples and the methods of storage of binder in tanks. It should be noted that there are big differences between the procedure for paving grade bitumen (non-modified), polymer modified bitumen and highly modified bitumen.

All presented guidelines concern bitumens produced by ORLEN Group and supplied by ORLEN Asphalt.

5.1. PROCESS TEMPERATURES



Process temperatures are a set of three basic values:

- **pumping temperature** – it determines the lower limit of binder pumpability from the tanker truck to the tank at the mixing plant; pumping problems can arise if the delivered bitumen is too viscous;
- **aggregate coating temperature** – this is key information for the mixing plant operation; to effectively coat the aggregate with the binder, the temperature of the aggregate coating (i.e. production of the asphalt mixture) must not be neither too high nor too low;
 - excessive temperature can result in an insufficient binder viscosity and a high risk of the binder flowing off the aggregate grains and a risk of its “overheating” and excessive ageing;
 - too low temperature may cause non-uniform coating of aggregate grains with bitumen, which may cause poorer quality of the asphalt mixture and e.g. increase its susceptibility to water and frost;
- **the temperature of the beginning and end of compaction** – the temperatures of the compaction are crucial for the paving team; during paving and compaction the bituminous binder acts as a lubricant, i.e. it must ensure proper workability or, in other words, slip of the aggregate to facilitate its correct movement in the paver and their compaction under the roller;
 - too high binder temperature may cause segregation of the mixture (separation of components) as the binder viscosity will not provide adequate “hold” of the aggregate (mixture cohesion);
 - too low binder temperature is associated with increased stiffness, which can result in aggregate moving more slowly through the mixture and making it more difficult to compact.

5.1.1. PAVING GRADE BITUMEN PROCESS TEMPERATURES

Paving grade bitumens are relatively simple binders to evaluate, due to the fact that they are Newtonian liquids at high temperature, for which viscosity does not depend on the shear rate.

temperature for paving grade bitumens is to use the equivalent viscosity method. By adopting appropriate viscosity values for each technological process described in 5.1, it is possible to determine from the viscosity–temperature dependency the appropriate temperature range in which a given binder will have the desired properties – an example is shown in Fig. 5.1.

The simplest and longest used way to determine the process

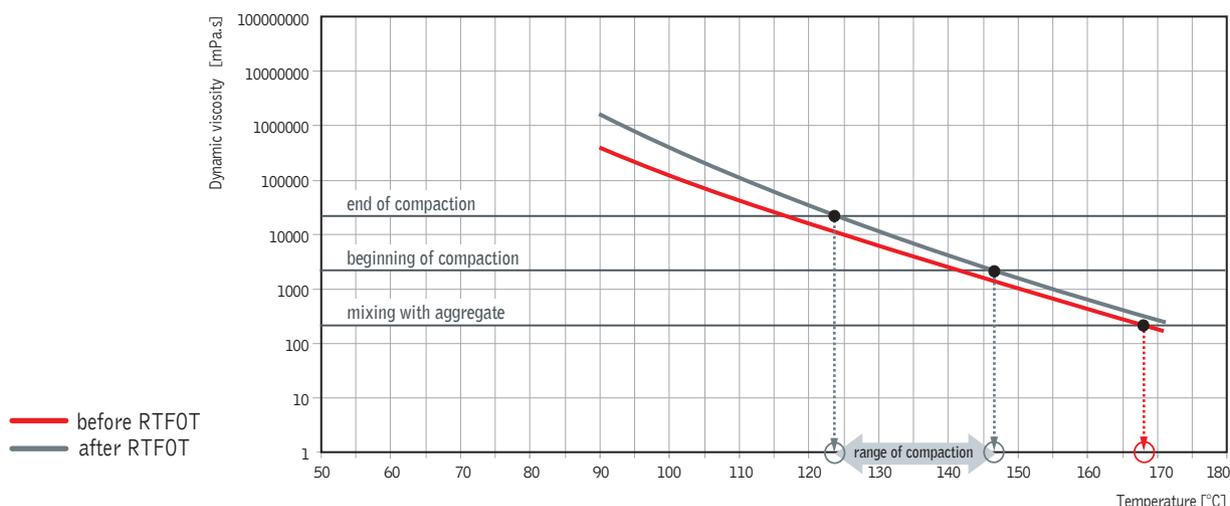


Fig. 5.1. Graphical presentation of the interpretation of process temperature ranges for paving grade bitumens acc. to equivalent viscosities [fig. by ORLEN Asphalt]

The dynamic viscosity limits used to determine the process temperature of paving grade bitumens vary in technical documentation. Table 5.1. shows viscosity ranges for process temperatures acc. to data from Shell [1],

Superpave [2] and ORLEN Asphalt.

The viscosity–temperature relation curves for each paving grade bitumen are presented in Chapter 2.

Table 5.1. Viscosity ranges acc. to different concepts for paving grade bitumens

| | EQUIVALENT VISCOSITY RANGE [Pa·s] | | |
|--|--|-----------------|------------------------|
| | SHELL BITUMEN HANDBOOK 2015 [1] | SUPERPAVE [2] | ORLEN ASFALT |
| Pumping temperature | 2,0 | – | 2,0 |
| Temperature of mixing with aggregate (asphalt mixture production) | 0,2 (or $T_{R\&B} + 110^{\circ}C$) | $0,17 \pm 0,02$ | 0,2 |
| Lower compaction temperature | 30,0 (or $T_{R\&B} + 50^{\circ}C$) | $0,28 \pm 0,03$ | 20,0 (after RTFOT)* |
| Upper compaction temperature | 5,0 | | 2,0 (after RTFOT)* |

* for explanation, see point 5.1.3

5.1.2. MODIFIED BITUMEN PROCESS TEMPERATURES

Determination of the process temperature ranges for bitumen containing polymer modifiers (of various types) and other additives is more complicated. In this case, not only the nature of the bituminous binder but also the characteristics of the additives influence the change in viscosity–temperature dependency. The behaviour of a modified binder can therefore deviate significantly from the typical relations in the paving grade bitumens. The SBS modified bitumens (that are used i.e in Poland) are a pseudoplastic liquids of a non-Newtonian behaviour, which viscosity depends on the shear rate – in other words, they are shear-thinned binders. When the bitumen is mixed with the aggregate in the mixing plant, the binder is subjected to a very high shear rate (a similar process, although in lower range, also occurs when the mixture is being paved), so the process temperatures do depend on the viscosity under the given shear conditions.

Consequently, the method of determining the technological temperature of the modified bitumen using the viscosity equivalent temperature is criticised and considered inadequate for modified binders. As observed, the use of the equivalent viscosity method for PMB/HiMA leads to an overestimation of process temperatures, which in turn contributes to overheating of the bitumen at the mixing plant [2, 3], and other problems related to that fact.

It should be added that the behaviour of the modified binder is not always the same because – as mentioned earlier, it depends on the nature of the modifier. Some of the additives

do not change the viscosity, some decrease and other increase its value. Furthermore, the effect of the modifier may not be uniform in the temperature range 90–230°C.

In conclusion, the application of the concept of equivalent viscosities to the determination of process temperatures for modified bitumen meets a number of obstacles. Therefore, for many years, with the increasing popularity of modified bitumen, research has been carried out aimed at developing a method suitable for this type of binders. In recent years, several new methods have been tested [3], such as:

- *High Shear Rate Viscosity* (HSRV) in several variants,
- *Zero Shear Rate Viscosity* (ZSRV) in several variants,
- *Low Shear Rate Viscosity* (LSRV),
- *Steady Shear Flow Test* (SSFT),

all of which use shear thinning to capture changes in viscosity or stiffness of the modified bitumen. However, these methods also generate very divergent results; an example of a comparative analysis for PMB 25/55-65 and PMB 45/80-55 is shown in Figure 5.2. (Figure based on publication [3]).

In conclusion, the present calculation methods do not allow to precisely determine optimum values of process temperature of the production of asphalt mixture (mixing of bitumen with aggregate) and correct temperature ranges of compaction. It is therefore necessary to refer to the experience gained at construction sites and test sections. The data shown in Tables 5.7 and 5.8 are based on the experience and observations gained in this way.

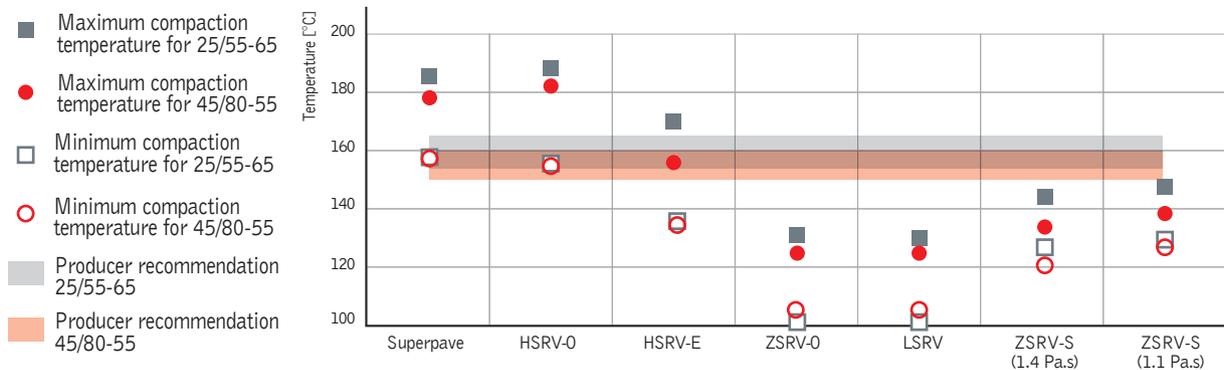


Fig. 5.2. Comparison of calculated compaction temperature values for PMB 25/55-65 and PMB 45/80-55 (Figure based on [3])

5.1.3. BITUMEN AGEING VS. PROCESS TEMPERATURES

The viscosity range of bituminous binders differs in the temperature range of 60–200°C depending on their consistency, origin, type, and additive content. Furthermore, the viscosity values for refinery-produced, non-aged bitumen will always differ from viscosity values after ageing. After ageing, bitumen hardens and its viscosity increases. Simulation of this phenomenon in the laboratory in terms of:

- short-term ageing is carried out in an RTFOT apparatus,
- long-term ageing is carried out in a PAV¹ apparatus after previous conditioning in the RTFOT apparatus.

Due to a certain degree of binder hardening, the viscosity–temperature curve after ageing (RTFOT or RTFOT+PAV) does not overlap with the characteristic curve for the non-aged bitumen and is shifted towards higher viscosity ranges. This means that the correct process temperatures should be determined based on bitumen viscosity testing, both before and after ageing. Processes of interest include:

- pumping and aggregate coating – applies to non-aged bitumen,

- compaction of the binder and aggregate mixture – applies to short-term aged bitumen (after RTFOT).

In order to determine the pumping and aggregate coating temperatures, bitumen test results before ageing should be used, because those processes occur before the contact of a thin binder layer with hot aggregate surface (before the main short-term ageing process starts).

In turn, to determine the beginning and end temperature of asphalt mixture compaction, viscosity values obtained after RTFOT should be used. In the asphalt mixture production process, “wet” mixing of the 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 several hours. Over that time, bitumen spreads over hot aggregate and ages – lighter components evaporate and, in effect, the bitumen hardens. Therefore, when the mixture spreading and compaction starts, the binder in the mix has usually already undergone the short-term ageing.

5.1.4. SAMPLE PREPARATION TEMPERATURE IN LABORATORY

In the discussed process temperature values, particular attention should be paid to the appropriate selection of sample compaction temperatures in the laboratory (according to the method chosen from EN 13108-20 [4]).

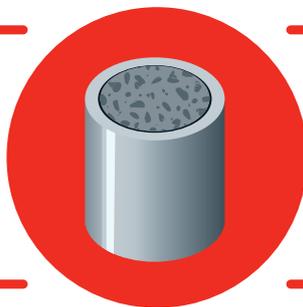
Temperature of asphalt mixture sample preparation should correspond not only to the binder type but also to the specific conditions on the construction site. If the temperature adopted at the lab is too high, the volumetric density of the asphalt mixture in the samples will be high and the void content will be underrated. If the on-site conditions markedly differ from those

adopted at the lab, i.e. the asphalt mixture temperature during the compaction is significantly lower, in practical terms, this will prevent the achievement of the required layer compaction indices insofar as the index calculation is based on data from the asphalt mixture Type Test. Conversely, if the temperature adopted by the lab is too low, it will result in overcompaction on the site – compaction index in excess of 100% will be achieved and void content in the course will be too low, which will increase the risk of rutting. That is why choosing the appropriate sample compaction temperature during the laboratory mixture design is very important².

1) The effectiveness of simulating actual long-term ageing of bitumen in a PAV (Pressure Ageing Vessel) apparatus has been questioned for some time, especially in the context of ageing modified bitumen.

2) It is also worth remembering about a certain incomparability of results of volumetric parameters of asphalt mixtures in tamped Marshall samples with the rolled mix, resulting e.g. from a different arrangement of aggregate grains. More information can be found e.g. in [2].

5.2. BITUMEN SAMPLES IN LABORATORY



The way in which bitumen samples are handled has a great influence on the test results obtained, both for the bitumens themselves and for the asphalt mixtures, therefore it is extremely important to adhere to the rules given below.

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

It should be remembered that a bitumen sample heated and/or overheated in the oven multiple times may significantly harden, and thus obtained results will significantly differ from the results for an non-aged bitumen. Therefore, when using samples of bituminous binders, repeated heating must be strictly 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 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 subsequent pouring into several smaller labelled containers for later use. Please note that the bitumen container must not be sealed during heating. The handling of bitumen samples for tests is specified in EN 12594.

Paving grade bitumen sample heating at the laboratory acc. to the standard procedure:

- under no circumstances should the samples be heated 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 should be raised adequately for the last 2 hours.

In the case of heating up a **conventional modified bitumen**, the procedure provided by the sample supplier should be implemented. ORLEN Asphalt recommends to set the temperature in the oven within the range of 160°C to 180°C, irrespective of the softening point of the bitumen.

In the case of the **highly modified bitumen HiMA** samples, due to their special properties, the procedure differs slightly – Table 5.2 shows maximum temperatures for heating up samples of these bitumens in the laboratory.

Table 5.2.

Heating-up temperature of ORBITON HiMA samples in the laboratory

| SAMPLE SIZE IN A CONTAINER | ORBITON 25/55-80 HIMA | ORBITON 45/80-80 HIMA | ORBITON 65/105-80 HIMA |
|--|--------------------------|--------------------------|---------------------------|
| Container volume up to 1 litre – sample heating time max. 2 hours | max. 170°C | max. 170°C | max. 170°C |
| Container volume 1–2 litres – sample heating time max. 3 hours | max. 170°C | max. 170°C | max. 170°C |
| Container volume 2–3 litres – sample heating time max. 3.5 hours | max. 170°C | max. 170°C | max. 170°C |
| Container volume 3–5 litres – sample heating time max. 4 hours | max. 170°C | max. 170°C | max. 170°C |
| Container volume over 5 litres – sample heating time max. 8 hours | max. 160°C | max. 150°C | max. 140°C |

Handling samples after heating

- binders for preparation of asphalt mixture samples – 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,
- if samples of bituminous binders are intended to be tested for their properties, ORLEN Asphalt, as the supplier recommends, in accordance with the principles given in PN-EN 12594 p. 7.1, **that once the samples are heated and homogenised, the material should be poured through a heated metal sieve with a mesh of 0.5 mm** to eliminate any possible impurities affecting the test results.

Bitumen samples obtained from:

- the extraction of asphalt mixtures (acc. to EN 12697-1, EN 12697-2, and EN 12697-4) should be tested promptly upon extraction in order to avoid reheating,
- tests for resistance to hardening or ageing (acc. to EN 12607-1, EN 12607-2 and EN 12607-3 or EN 14769 or any other standard which deals with hardening or ageing) should be prepared and tested in accordance with adequate extraction and testing methods.

5.3. 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 a high or low content of silica (SiO₂) 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. However, before the application of adhesion promoters, they should be tested at the lab, as certain chemical agents cause bitumen and aggregate bonding (adhesion) to deteriorate.

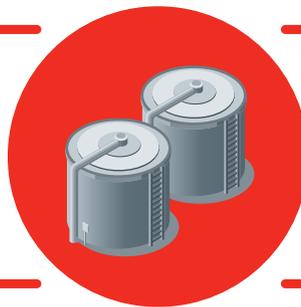
Adhesion agents available on the market and their content in bitumen should be selected for a specific bituminous binder and aggregate, and it should be remembered that all-purpose products that perform well with each bitumen–aggregate combination are a rarity.

Currently, the following two standards are used to test the adhesion of bitumen to aggregate and, in general, the water and frost resistance of asphalt mixtures: EN 12697-11 and EN 12697-12.

Evaluation of adhesion can be carried out on a selected fraction of the mineral mixture, for example, based on the methods described in EN 12697-11, of which the most popular is method A (rolling bottle method). The adhesion of a binder to an aggregate should be at least 80% after 6 hours of rolling (requirement in Poland).

The final check of the asphalt mixture resistance to water and frost is the ITR test, as per EN 12697-12. Different versions of the test procedure are used, depending on the technical documentation.

5.4. STORAGE OF BITUMINOUS BINDERS



5.4.1. GENERAL NOTES

Bituminous binders should be stored in tanks designed specifically for that purpose.

Bitumen in the storage 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 storage 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.

According to the requirements of EN 13108-21 on asphalt mixtures Factory Production Control, the temperature of the in the tank should be recorded once a day.

Long-term storage of bitumen batches at temperatures close to the maximum storage temperature may after some time cause a deposit build-up at the bottom of the tank, 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 a 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 gradual 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 build-up at the bottom of the tank and deterioration of the binder

properties. Similarly, intensive mixing in contact with air can contribute to excessive ageing of the binder.

Examples of factors affecting the ageing of bituminous binders in storage tanks and ways to reduce them are shown in Table 5.3.

Detailed information on the storage of all bituminous binder types are shown in subsequent points of the chapter.

Table 5.3.
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 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 prolonged time, circulation should be limited or activated periodically only. Circulation is particularly useful for the storage of modified bitumen if the tank is not fitted with an agitator. It 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 favourable situation is when the ratio of bitumen surface and its volume in the tank is small. That is why storage tanks for bitumen should be vertical – high tank height-to-diameter ratio. |

5.4.2. STORAGE OF PAVING GRADE BITUMEN

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

Table 5.4.

Paving grade bitumen storage temperatures

| BITUMINOUS BINDER | RECOMMENDED MAXIMUM TEMPERATURE FOR SHORT-TERM STORAGE (UP TO 10 DAYS) OF BITUMEN, [°C] |
|------------------------------|---|
| Paving grade bitumen 20/30 | ≤ 185°C |
| Paving grade bitumen 35/50 | ≤ 185°C |
| Paving grade bitumen 50/70 | ≤ 185°C |
| Paving grade bitumen 70/100 | ≤ 180°C |
| Paving grade bitumen 100/150 | ≤ 180°C |
| Paving grade bitumen 160/220 | ≤ 180°C |

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

Paving grade bitumen storage at high temperatures over prolonged periods of time should be avoided. If it is necessary to store bitumen in the tank at high temperatures (over 150°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.

The following should be tested:

- penetration at 25°C acc.to EN 1426,
- softening point acc. to EN 1427.

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

In normal storage for long periods (>10 days) the temperature of the bitumen should be reduced below 150°C.

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

If it is necessary to store paving grade bitumen for a much longer period than 10 days, it is recommended to reduce the temperature of bitumen to 80–100°C and reheat it before reuse.

If a very long storage period is expected without any production of asphalt mixture, paving grade bitumen storage at the ambient temperature is allowed. A precondition for such storage is fitting the tank with a heating equipment of sufficient capacity to ensure subsequent bitumen heating without running the risk of local binder overheat during prolonged heating.

Before the bitumen is used to produce an asphalt mixture, at least the tests listed above should be carried out. In this case, consideration should be given to the possibility of safely taking samples of the bitumen for testing from the installation (e.g. via dedicated valves).

5.4.3. STORAGE OF POLYMER MODIFIED BITUMEN

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

Table 5.5.

Storage temperatures for polymer modified bitumens ORBITON

| BITUMINOUS BINDER | RECOMMENDED BITUMEN STORAGE TEMPERATURE [°C] | GUARANTEED PERIOD OF BITUMEN SERVICE LIFE FOR ASPHALT MIXTURE PRODUCTION |
|--|--|--|
| Polymer modified bitumen ORBITON 10/40-65 | 160 ÷ 180°C | 7 days |
| Polymer modified bitumen ORBITON 25/55-60 | 160 ÷ 180°C | 7 days |
| Polymer modified bitumen ORBITON 45/80-55 | 160 ÷ 180°C | 7 days |
| Polymer modified bitumen ORBITON 40/80-65 | 160 ÷ 180°C | 7 days |
| Polymer modified bitumen ORBITON 65/105-60 | 160 ÷ 180°C | 7 days |

It is recommended to conduct basic inspection tests of modified bitumen properties after 5 days 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 acc.to EN 1426,
- softening point acc.to EN 1427,
- elastic recovery at 25°C acc.to EN 13398.

If the mixing plant is equipped 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 polymer modified bitumen for longer 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 earlier). It is desirable to mix bitumen in the tank for at least 6 hours per day.

Recommended storage temperature: 130–160°C.

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

The storage of polymer modified bitumen cooled down to ambient temperature (e.g. over winter) is not recommended because of the significant difficulties with its fluidisation and, in consequence, change in performance properties.

5.4.4. STORAGE OF HIGHLY MODIFIED BITUMEN

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

Table 5.6.

Storage temperatures for highly modified bitumens ORBITON HiMA

| BITUMINOUS BINDER | RECOMMENDED BITUMEN STORAGE TEMPERATURE [°C] |
|--|--|
| Highly modified bitumen ORBITON 25/55-80 HiMA | 160 ÷ 170°C |
| Highly modified bitumen ORBITON 45/80-80 HiMA | 160 ÷ 170°C |
| Highly modified bitumen ORBITON 65/105-80 HiMA | 160 ÷ 170°C |

Recommended storage temperature for ORBITON HiMA is 160–170°C.

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

Storage of highly modified bitumen for more than 3 days at high temperatures (over 170°C) is not recommended.

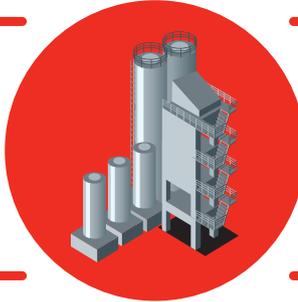
If longer storage is required, the bitumen temperature should be lowered to 160°C and it should be homogenised by periodical mixing. Therefore, it is recommended that the tank is fitted with an agitator. Excessive storage time (more than 3 days) at high temperatures (above 180°C) may lead to gradual increase of the viscosity of the highly modified bitumen, which may reduce the possibility of its application.

5.4.5. 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, e.g. paving grade bitumen with polymer modified bitumen. The mixing would markedly downgrade the binder performance and pavement life.
- Mixing of bitumen of the same type but different grades, such as 50/70 with 70/100, is at the sole responsibility of the contractor. The process requires an effective mixing system in the tank and a laboratory control. Binders from different manufacturers should not be mixed.
- Multiple heating and cooling cycles of modified bitumens ORBITON and highly modified bitumens ORBITON HiMA are not recommended.
- If a paving grade bitumen will 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 tons of bitumen may be lengthy in spring and depend on the efficiency and structure of the tank heating system. Binder properties must be tested after heating. Note: not every type and grade of binder can be stored in this way (see information above).

- Bitumen temperature during storage should not exceed values indicated in Tables 5.7. and 5.8.
- HiMA binders should not be mixed with other binders. It would markedly downgrade the performance of the binder and affect the durability of the pavement.

5.5. ASPHALT MIXTURE PRODUCTION



The bitumen supplied to the mixing plant should be at the right temperature for problem-free unloading from the road tanker.

Bitumen viscosity is strictly related to its temperature:

- for paving grade and polymer modified bitumen – the higher the temperature of the bitumen, the lower the viscosity,
- for the highly modified bitumen HiMA, – the viscosity decreases up to a certain temperature above which it starts to increase rapidly with time of storage (the process is particularly visible from the 3rd day of storage at a temperature above 180°C), therefore, all the guidelines regarding maximum storage temperature and process temperatures given in Table 5.8 should be strictly observed.

In cold seasons, the temperature of the bitumen in the tanker truck should be monitored during the transport from the refinery.

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

The application of too hot asphalt mixture for production may have other adverse effects, especially in the case of the production of mixtures of non-continuous granulation (SMA, BBTM, AUTL or PA) which face 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 the draindown

using the Schellenberg's method in an increased temperature, acc. to EN 12697-18.

The ORBITON HiMA binders should not be overheated and the indications in Table 5.8. should be followed. Increasing the temperature above the recommended values may cause the opposite reaction, i.e. a significant stiffening of the binder due to an increase in viscosity.

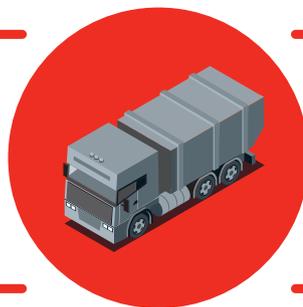
Temperatures provided in Tables 5.7. and 5.8. do not apply to asphalt mixtures containing the additives for production and paving temperature reduction, e.g. for WMA. If such agents are used, their use should be preceded by tests in the laboratory of the asphalt mixture manufacturer.

If the freshly produced mixture is stored in a silo, attention should be paid to its temperature. The cooling and stiffening of the mixture is dependent on the following factors:

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

Tables 5.7 and 5.8 at the end of the chapter give the recommended process temperatures for the use of bituminous binders and asphalt mixtures.

5.6. TRANSPORT OF ASPHALT MIXTURE



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

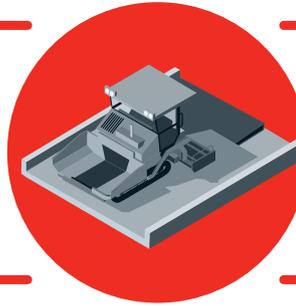
Cargo compartments must be always covered by tarpaulin during asphalt mixture transport. Whenever temperatures are low or during other adverse weather conditions, it is recommended to use vehicles with isolated cargo compartments.

If it is necessary to work under adverse weather conditions (temperature $<+5^{\circ}\text{C}$, strong wind >10 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 truck should be considered. The transport work should be arranged so as to ensure the continuity of deliveries to the construction site (no paver stops).

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

- blue smoke above the mixture – indicates its excessive overheating in the course of mixing with an aggregate (over 200°C). The mixture is essentially destroyed (overburnt) and will ravel after paving 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 mixture – 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 asphalt mixture forms a sharp cone, it is matt – this may suggest that the mixture’s temperature is too low or the bitumen content is too small; in effect, the mixture may not have the required workability and compactibility 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 mixture (design flaw),
 - b. bitumen feeder damage (mineral mixture production error),
 - c. too low bitumen temperature during mixing with an aggregate,
 - d. “wet” mixing time in the paving 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 is an excessive dampness of the aggregate, which the mixing plant drier was not able to eliminate. This effect occurs more frequently with highly-absorbable aggregate types and after long-lasting rainfall.

5.7. PAVING



High stiffness modulus asphalt concrete mix (AC EME) combined with hard bitumens should be paved in the form of the thickest technologically and by-design permitted course. This will improve the temperature conditions of compaction.

When mixtures are paved on the base having an increased temperature (just-paved courses, i.e. "hot on warm"), the temperature at mid-thickness of the paved course should be thoroughly controlled. Non-contact (infrared) thermometers are not recommended, unlike thermometers with a steel spindle allowing for immersion into the course. If the temperature of the paved mixture is very high (mixture cools down very slowly – it is heated from the bottom), rolling should not commence until the temperature drops to the point enabling the compaction to proceed. The recommendations do not apply to the *Kompaktasphalt* technology (two courses built in at the same time with a special paver).

Mastic asphalt (MA) mixture sometimes cannot be paved manually due to its high viscosity. Mechanical equipment is recommended for such paving, along with additives improving workability. Attention must be paid to the temperature and time of storage of a mastic asphalt mixture, detailed guidelines can be found in the tables 5.7. and 5.8.

During the paving of the asphalt mixtures containing ORBITON HiMA, the same rules as for conventional modified bitumen apply. However, the number and type of rollers and the number of passes may be increased, and the final parameters should be chosen on a trial sections taking into account the thickness of the layer, the ambient temperature and the type of asphalt mixture. A key factor that requires attention is the temperature of mixture production and paving.

During the paving 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 asphalt mixture (Autumn). During compaction, the mixture may behave in an elastic way and move slightly under the rollers, especially in the first phase of compaction at high temperatures. Experience has shown no problems with compacting layers containing ORBITON 65/105-80 HiMA.

After completion of the pavement work, it is recommended that the equipment (mainly the paver) be cleaned of residuals immediately while the asphalt mixtures is still hot – this applies particularly to highly modified bitumens.

5.8. **SORBENTS FOR COLLECTING OF OIL SPILLED ON A SURFACE**



By definition, sorbents are substances that have the ability to absorb other substances. They are also used in the road industry as substances that are very helpful for collecting oil or fuel spills on the surface of pavement. It should be remembered that the speed with which such a stain is removed from the road is one of the most important factors for the durability of the asphalt pavement. The oil-derived substances dissolve bitumen, penetrate successive courses and cause permanent damage.

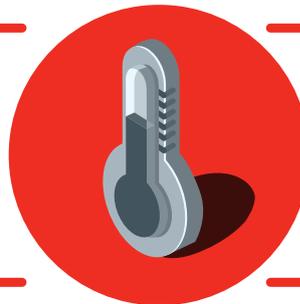
In the past, sand or sawdust was used to remove oil-derived substances from the surface. Cellulose sorbents: sawdust, wood, paper, due to their low density (quite light and sensitive to wind gusts), can be used to absorb oil spills but only in windless weather. It should be remembered that these types of sorbents also absorb water. Specially treated cellulose sorbents with no water absorption problems are also available on the market.

Synthetic polymeric sorbents (e.g. polyurethane sorbents) are also available for use on roads. There are also light sorbents which behave like the previously mentioned cellulose sorbents but there are also specially processed higher-density sorbents which can be used under different weather conditions (wind).

The following factors should determine the use of a particular sorbent on a road surface:

- high and fast absorption,
- no negative impact on the asphalt pavement,
- versatility,
- usefulness in all weather conditions:
 - hydrophobicity (rain, snow)
 - appropriate weight (not susceptible to wind)
- easy to remove after application (does not form a smear),
- anti-slip, if possible.

5.9. **PROCESS TEMPERATURES – SUMMARY**



Tables 5.7. and 5.8. show the minimum and maximum process temperature for the use of bitumens and asphalt mixtures in the laboratory, at the mixing plant and at the construction site.

Temperatures provided do not apply to asphalt mixtures containing the "warm-mix" additives for production and paving temperature reduction.

Table 5.7.

Minimum and maximum temperatures for bitumens and asphalt mixtures depending on the type of binder

| BITUMEN TYPE | PAVING GRADE BITUMEN | | | POLYMER MODIFIED BITUMEN | | | | |
|---|----------------------|---------------------------------|---------------------------------|--------------------------|---------------------|---------------------|---------------------|----------------------|
| | BITUMEN 20/30 | BITUMEN 35/50 | BITUMEN 50/70 | ORBITON 10/40-65 | ORBITON 25/55-60 | ORBITON 45/80-55 | ORBITON 45/80-65 | ORBITON 65/105-60 |
| TEMPERATURE, [°C] | | | | | | | | |
| Laboratory: | | | | | | | | |
| Marshall/gyratory press sample compaction temperature | 155-160 | 140-145 | 135-140 | 150-155 | 145-150 | 145-150 | 150-155 | 145-150 |
| Ingredient temperature at the mixing plant: | | | | | | | | |
| Pumping of bitumen | > 145 | > 140 | > 130 | > 150 | > 150 | > 150 | > 150 | > 150 |
| Short-term binder storage at the mixing plant (up to 3 days) | < 185 | < 185 up to 200 ^d | < 185 up to 200 ^d | < 185 | < 185 | < 185 | < 185 | < 185 |
| Binder storage at the mixing plant (3–7 days) | < 175 | < 175 | < 175 | < 170 | < 170 | < 170 | < 170 | < 170 |
| Asphalt mixture temperature in the mixing plant's mixer: | | | | | | | | |
| Asphalt concrete, AC | < 185 | < 180 | < 175 | < 185 | < 185 | < 185 | < 185 | < 185 |
| SMA, BBTM, AUTL | – | – | < 175 | – | < 185 | < 185 | < 185 | < 185 |
| Porous asphalt, PA | – | – | – | – | – | < 185 | < 185 | < 185 |
| Mastic asphalt, MA | < 220 ^{a)} | < 220 ^{a)} | – | – | < 230 ^{b)} | – | – | – |
| Temperature on site: | | | | | | | | |
| Minimum temperature of the supplied asphalt mixture in the paver's hopper | 150 | 145 | 140 | 160 | 155 | 155 | 160 | 160 |
| End of effective layer compaction temperature | > 120 | > 115 | > 110 | > 125 | > 125 | > 120 | > 125 | > 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 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

c) maximum storage temperature of 200°C only in exceptional cases for deliveries from a bitumen refinery with such a temperature

Table 5.8.

Minimum and maximum temperature of the binder and asphalt mixture in relation to the type of highly modified bitumen

| BITUMEN TYPE | ORBITON 25/55-80 HIMA | ORBITON 45/80-80 HIMA | ORBITON 65/105-80 HIMA |
|--|--------------------------|--------------------------|---------------------------|
| Laboratory: | | | |
| Recommended Marshall/gyratory press sample compaction temperature | 145-150°C* | 145-150°C* | 145°C* |
| Ingredient temperature at the mixing plant: | | | |
| Pumping of bitumen | >150°C | > 140°C | > 130°C |
| Short-term binder storage at the mixing plant (up to 3 days) | recommended < 170°C | recommended < 170°C | recommended <170°C |
| Long-term (more than 3 days) storage of bitumen at a mixing plant, with periodic mixing or circulation | 150-160°C | 150-160°C | 150-160°C |
| Asphalt mixture temperature in the mixing plant's mixer: | | | |
| Asphalt concrete, AC | < 180°C | < 180°C | < 175°C |
| SMA | < 180°C | < 180°C | < 175°C |
| Porous asphalt, PA | < 180°C | < 180°C | < 175°C |
| Mastic asphalt, MA | – | < 180°C | < 180°C |
| Temperature on site: | | | |
| Minimum temperature of the supplied asphalt mixture (in the paver's hopper) | 160°C | 160°C | 150°C |
| End of effective layer compaction temperature | > 130°C | > 125°C | > 120°C |

* does not apply to SMA 16 and SMA 22 mixtures for which a compaction temperature of 160°C should be adopted

NOTE

The temperature data given in Table 5.8 have been determined based on findings from various applications of HiMA bitumens. They may change with further experience.

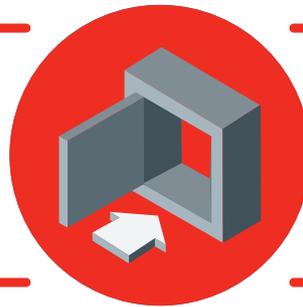
6. RECOMMENDATIONS FOR DESIGN OF ASPHALT MIXTURES WITH ORBITON HiMA

In 2017–2019¹, the Research, Development and Innovation Department of ORLEN Asphalt carried out a research to capture the differences in the process of designing asphalt mixtures with highly modified bitumens ORBITON HiMA, compared to traditional binders. In the study were used the most popular mixtures for courses of the conventional road pavement construction for heavy traffic: AC 22 base, AC 16 bin, SMA 8 surf.

All the tests were carried out in cooperation with the laboratory of the Road and Bridge Research Institute, the laboratory of the Faculty of Construction and Environmental Sciences of Białystok University of Technology and the Road Laboratory W. Bogacki in Rzgów, Poland.

Below is a synthesis of information and conclusions from the research work. The conclusions have been prepared based on research conducted by ORLEN Asphalt as well as observations and experience gathered at full-scale sections.

6.1. INTRODUCTION



Highly modified binders ORBITON HiMA, although similar in appearance and consistency to conventional modified bitumen, are actually a completely different material. The use of the polymer SBS (styrene-butadiene-styrene) in amounts above 7% (m/m) causes a phase inversion in the bitumen-polymer mixture, which gives the highly modified binders superspecial properties. Fig. 6.1 shows the evolution of the polymer network formation in the bitumen during the addition of increasing amounts of SBS elastomer.

Structurally, pavement layers with ORBITON HiMA show a very high tolerance for increasing tensile deformation (so-called

fatigue), which, while maintaining the same thickness as structures designed with conventional binders, allows to extend the fatigue life of structures with ORBITON HiMA or, alternatively, enables the reduction of the asphalt packet thickness in the pavement to some extent while maintaining the fatigue life of a thicker structure with conventional bitumens.

Properly-designed asphalt mixtures with highly modified bitumen ORBITON HiMA are materials with above-standard properties. However, to get the most out of ORBITON HiMA, a slightly different approach to the design and use of these bitumens in asphalt mixtures is required.

1) For more information see [1].

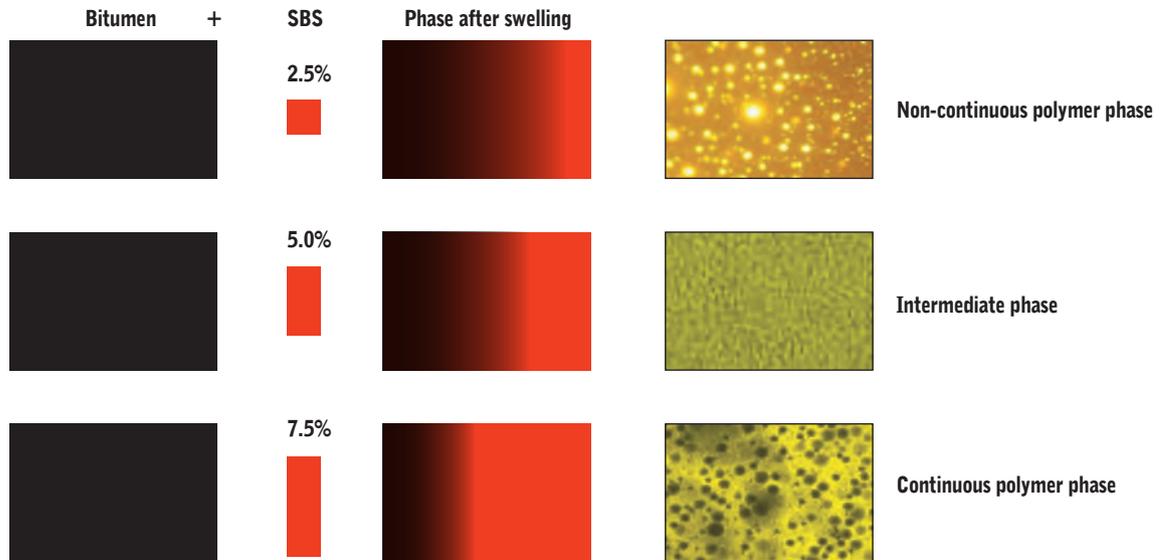


Fig. 6.1. Volume proportions between bitumen and polymer in a typical polymer modified bitumen (2.5–5.0% m/m) and in highly modified bitumen HiMA (7.5% m/m) [2]

6.2. TEST PROGRAMME



In the research programme conducted by ORLEN Asfalt, the properties of asphalt mixtures with ORBITON HiMA were tested using:

- asphalt concrete for the base course – AC 22 base,
- asphalt concrete for the binder course – AC 16 bin,
- stone mastic asphalt for the wearing course – SMA 8 surf.

The following binders were used to test the properties of the asphalt concretes for binder and base courses:

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

For testing the properties of stone mastic asphalt SMA 8 for the wearing course, the following were used:

- paving grade bitumen 50/70,
- polymer modified bitumen ORBITON 45/80-55,
- highly modified bitumen ORBITON 65/105-80 HiMA.

For each asphalt mixture, a set of tests was performed as a function of binder content:

- Density acc. to EN 12697-5, method A,
- Volumetric density acc. to EN 12697-6, method B,
- Void content V_m acc. to EN 12697-8,
- Void content in the mineral mixture – VMA acc. to EN 12697-8,
- Void content in the mineral mixture filled with binder – VFB acc. to EN 12697-8,
- Rutting acc. to EN 12697-22, small apparatus, method B in air,
- ITSR acc. to EN 12697-12 (conditioning procedure with one freezing cycle acc. to WT-2 2014),
- Additionally, for SMA 8 – TSRST acc. to EN 12697-46.

Samples were prepared using the tamping method, with the energy of 2x75 blows for AC 16 bin and AC 22 base, and for SMA 8 with the energy of 2x50 blows.

6.3. SUMMARY AND CONCLUSIONS OF TESTS



- When designing asphalt mixtures with highly modified bitumens, it is worth using the Marshall's method and analysing the volumetric parameters of the asphalt mixture. It is worth remembering that the minimum bitumen contents (B_{min}) given in the technical documentation have been developed based on research and experience with paving grade or modified bitumens and not with highly modified bitumens (unless explicitly stated).
- Based on observation, mixtures with highly modified bitumens require more binder (mixtures on road sections appear "too dry"). It is hypothesised that HiMA binders are absorbed by the mineral mixture to a much greater extent than conventional modified bitumens and paving grade bitumens – this is confirmed by the results for the three asphalt mixtures tested: AC 22 base, AC 16 bin and SMA 8 surf.
- The increased binder absorption of HiMA in asphalt mixtures leads, in consequence, to the need to increase the amount of the binder to achieve an appropriate level of effective bitumen content:
 - in asphalt concrete mixtures for binder and base courses (AC 16 and AC 22), it is necessary to increase the content of highly modified bitumen by 0.2–0.3 pp. compared to the content of conventional binders, which is visible in the values of optimum voids content in the VMA mineral mixture (Fig. 6.2. – example data for AC 16 W);
 - in the mixture with a non-continuous granulation (SMA 8) for the wearing course, it is necessary to increase the content of highly modified bitumen by 0.1–0.2 pp. compared to the asphalt mixtures with conventional binders; no differences in optimum VMA values for the asphalt mixtures with individual binders were observed for this mixture.

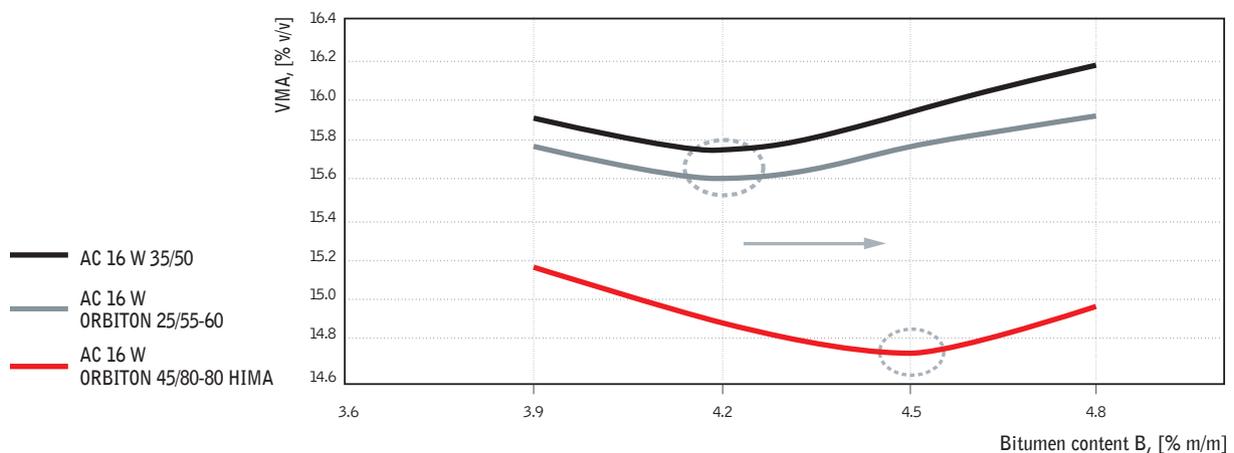


Fig.6.2. VMA void content in mm as a function of bitumen content for AC 16 bin [1]

- Sample compaction temperature for testing of highly modified bitumens should be:
 - for ORBITON 65/105-80 HiMA: $T = 145^{\circ}\text{C}$,
 - for ORBITON 45/80-80 HiMA: $T = 145\text{--}150^{\circ}\text{C}$,
 - for ORBITON 25/55-80 HiMA: $T = 150^{\circ}\text{C}$.
- The standard temperature of $T = 160^{\circ}\text{C}$ for PMB HiMA is too high and, as a result, the samples may reach too high a density impossible to achieve on a real road section; this may cause problems with achieving an appropriate compaction ratio required for acceptance of the completed course.
 - for SMA 16 and SMA 22 mixtures with ORBITON HiMA binders, the sample compaction temperature of $T = 160^{\circ}\text{C}$ should be adopted.
- Asphalt mixtures with polymer modified binders are characterised by higher water and frost resistance (ITSR) in comparison to asphalt mixtures with paving grade bitumens in each of the three types of tested mixtures (which is illustrated by the data for AC 22 base in Fig. 6.3). In the study, the dependence of the ITSR index on the binder content in the mixture was obtained.

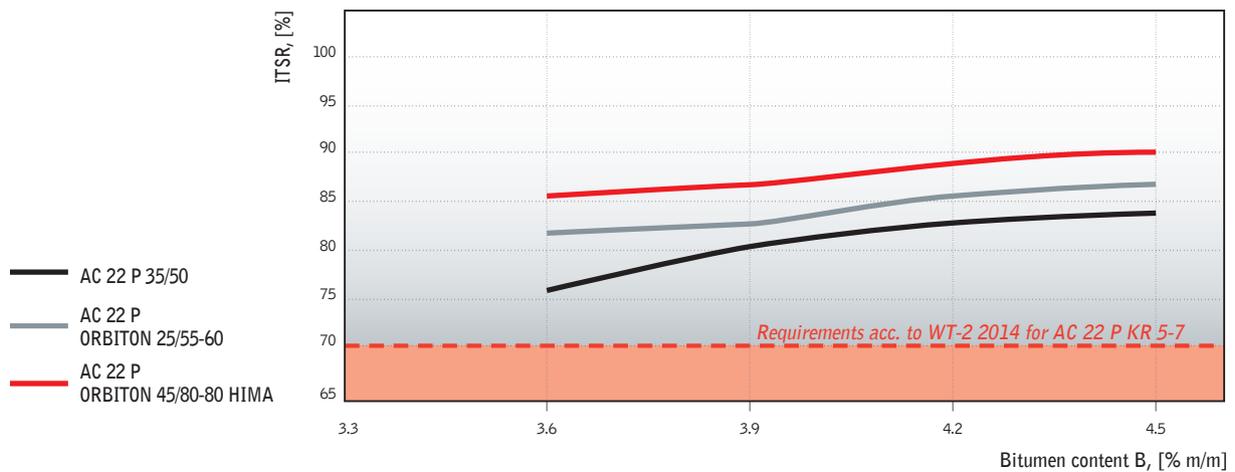


Fig. 6.3.
ITSR as a function of bitumen content for AC 22 base [1]

- The design of SMA 8 surf mixture, as well as other mixtures with non-continuous granulation, should be based on the understanding of the effect of the granulation curve on the aggregate arrangement of the mixture, which translates, for example, into the water permeability of the asphalt mixture.
- Asphalt mixtures with polymer modified binders are more resistant to permanent deformation than mixtures with paving grade bitumens. When PMB HiMA bitumens are used, it can be seen that the WTS and PRD parameters are not very sensitive to the binder content – all mixtures, regardless of the bitumen content in the asphalt mixture, were characterised by very good WTS and PRD results.
- The SMA 8 surf mixture with highly modified binder is characterised by the lowest thermal shrinkage cracking temperature in relation to the tested asphalt mixture with modified and paving grade bitumen (Fig. 6.4.).
- The resistance to thermal shrinkage cracking increases with increasing the amount of polymer modified bitumen in the mixture, while no such relation was observed for the paving grade bitumen (Fig.6.4.).

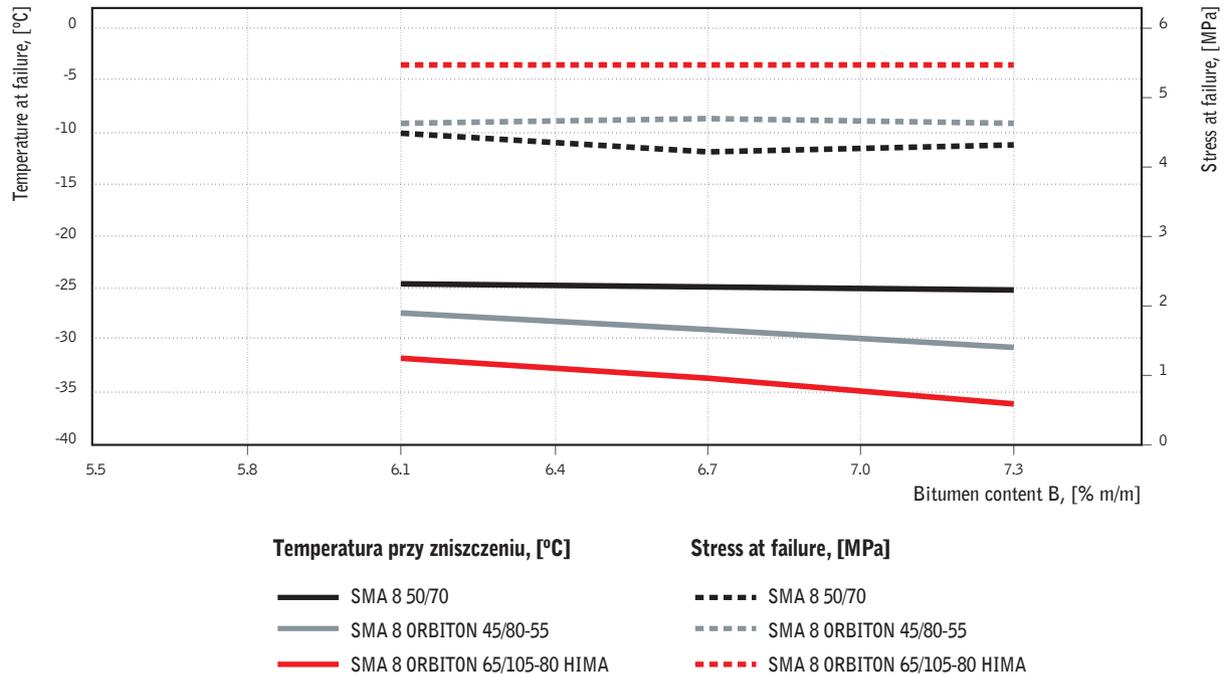


Fig. 6.4.
TSRST Low temperature cracking resistance as a function of bitumen content [1]

- Mixtures containing highly modified bitumens form a much more durable coating on the surface of the aggregate, which protects the mineral mixture, but, on the other hand, requires sprinkling of aggregate on the surface of the wearing course to ensure an adequate coefficient of friction during the service phase of the pavement immediately after its construction.

Readers interested in detailed research results are encouraged to consult the information in the book entitled "Mieszanki i nawierzchnie z ORBITON HiMA" [Asphalt mixtures and Pavements with ORBITON HiMA] distributed by ORLEN Asphalt sp. z o.o. The publication (in Polish) in PDF version is available on the website: www.orlen-asfalt.pl

7. POLISH REQUIREMENTS AND NEW PROPERTIES FOR PMB/HIMA BINDERS

Paving grade and polymer modified bitumens produced by ORLEN Group meet the requirements of standards **EN 12591** and **EN 14023**. These standards are part of a package of European standards for bituminous binders.

7.1. NEW POLISH NATIONAL ANNEX TO PN-EN 14023



In the course of the work on Bitumen Handbook 2021, activities in the Subcommittee for Bitumen of Technical Committee 222 of the Polish Committee for Standardisation related to the preparation of amendments to the Polish National Annex NA to PN-EN 14023 were ongoing. This annex was published together with the original standard in 2011, then it was extended in 2014. (Ap1:2014) and a further revision was prepared in 2019–2020. According to the standardisation rules, the National Annex to the EN standard contains the agreed properties and levels of requirements for polymer modified bitumens (conventional and

highly modified) used in Poland. This work culminated in the publication of an amendment to Ap2:2020-02 (see Chapter 1 for more information).

The amendment to the 2020 National Annex drew on conclusions from the research results from various research centres and bituminous binder manufacturers in Poland.

The new 2020 National Annex is presented in Chapter 1, in Tables 1.7. (modified bitumen) and 1.11. (highly modified bitumen).

7.2. NEW PROPERTIES IN FUTURE SPECIFICATIONS



The TC336 CEN committee is considering changes to the tables for the PMB property specifications. The agreed and voted changes to EN 14023 will be published in the future, according to CEN procedures and deadlines.

The changes aim at a better specification of the performance of PMBs, e.g. for rutting resistance (MSCR test), low temperature cracking (BBR test), or at the progressive introduction of new

methods to replace old ones (e.g. BBR instead of Fraass, $|G^*|$ instead of Softening Point) etc.

The new rheological parameters considered for the future include, among other things: viscoelastic properties ($|G^*|$, δ), the MSCR test and the BBR test. The results of tests of binders produced by ORLEN Group, based on testing parameters according to the publication [1], are presented in the following section.

7.3. VISCOELASTIC PROPERTIES



Properties related to the temperature sensitivity of bitumens and viscoelastic properties, tested using a dynamic shear rheometer acc. to EN 14770, at a constant frequency of 1.59 Hz, are labelled as temperature T0...T4.

Example results of rheological parameters – temperatures T0–T4 and δ at (T0–T4) for modified bitumens supplied by ORLEN Asphalt are shown in Table 7.1.

Table 7.1.

Properties of modified bitumens acc. to prEN 14023 T0–T4 [°C] and δ at (T0–T4) [°] [research by ORLEN Asphalt of 2020]

| BITUMEN TYPE | REOLOGICAL PARAMETERS prEN 14023 – T0-T4 AND δ AT (T0-T4) | | | | | | | | | |
|------------------------|--|-------------------|---------------------|-------------------|--------------|-------------------|-------------------------|-------------------|--------------|-------------------|
| | G* =15 [kPa] | | G* =5 [MPa] | | G* =15 [kPa] | | G* =5 [MPa] | | G* =15 [kPa] | |
| | BITUMEN BEFORE AGEING | | BITUMEN AFTER RTFOT | | | | BITUMEN AFTER RTFOT+PAV | | | |
| | T0 [°C] | δ (T0) [°] | T1 [°C] | δ (T1) [°] | T2 [°C] | δ (T2) [°] | T3 [°C] | δ (T3) [°] | T4 [°C] | δ (T4) [°] |
| ORBITON 25/55-60 | 46.4 | 65.0 | 22.0 | 46.3 | 52.0 | 62.1 | 26.2 | 41.3 | 61.2 | 59.2 |
| ORBITON 45/80-55 | 40.0 | 65.6 | 16.7 | 48.5 | 43.7 | 64.6 | 21.8 | 44.4 | 51.5 | 62.0 |
| ORBITON 45/80-65 | 41.4 | 62.9 | 17.8 | 48.4 | 44.8 | 61.9 | 22.4 | 44.3 | 51.8 | 59.4 |
| ORBITON 65/105-60 | 35.9 | 65.0 | 12.7 | 49.4 | 39.4 | 63.1 | 17.6 | 46.0 | 48.8 | 61.2 |
| ORBITON 25/55-80 HiMA | 43.5 | 55.4 | 13.9 | 45.6 | 48.1 | 52.5 | 19.0 | 41.6 | 57.6 | 49.2 |
| ORBITON 45/80-80 HiMA | 35.4 | 61.2 | 12.6 | 47.7 | 39.9 | 58.7 | 16.4 | 43.8 | 48.8 | 53.5 |
| ORBITON 65/105-80 HiMA | 31.8 | 61.7 | 8.9 | 50.5 | 38.6 | 57.2 | 14.7 | 45.9 | 47.0 | 53.5 |

7.4. MSCR TEST



Properties obtained from the MSCR test (EN 16659), also performed using a dynamic shear rheometer (see Chapter 3 for more information on the MSCR test). After performing the MSCR test acc. to EN 16659, the following properties, as defined in the standard, are determined at the selected temperature on the RTFOT short-term aged sample:

- percent recovery [%] – R
- non-recoverable creep compliance [kPa^{-1}] – J_{nr}

Both parameters are related to the flow and strain resistance of the binder and can provide valuable information on the influence

of a given bitumen on rutting of asphalt mixtures, especially when determined at high temperature values.

Table 7.2 shows the results of the MSCR test at reference temperature 60°C for modified bitumens supplied by ORLEN Asphalt. It can be seen that at least some modified bitumens should be tested at a higher temperature than 60°C , as the result of $J_{nr,3.2}$ is below the limit value of 0.1 kPa^{-1} . More results of tests performed using the MSCR method are given in Chapter 3.

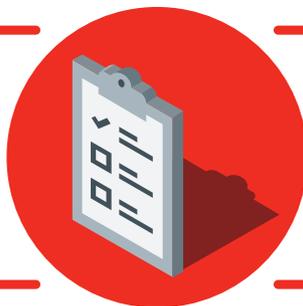
Table 7.2.

MSCR test results – parameters: J_{nr} [kPa^{-1}] and R [%], obtained at the stress of 3.2 kPa at 60°C , samples after RTFOT

Interpretation: the lower the J_{nr} value, the higher the resistance to rutting and the higher the recovery R, the more elastic the binder [research by ORLEN Asphalt]

| BITUMEN TYPE | MSCR ACC. TO prEN 14023 | |
|-----------------------|------------------------------------|----------------------|
| | 60°C, SAMPLES AFTER RTFOT | |
| | $J_{nr,3.2}$ [kPa^{-1}] | R 3.2 [%] |
| Interpretation | <i>less = better</i> | <i>more = better</i> |
| ORBITON 25/55-60 | 0.1 | 67.9 |
| ORBITON 45/80-55 | 0.2 | 72.6 |
| ORBITON 45/80-65 | 0.1 | 82.3 |
| ORBITON 65/105-60 | < 0.1 | 92.1 |
| ORBITON 25/55-80 HiMA | < 0.1 | 96.0 |
| ORBITON 45/80-80 HiMA | < 0.1 | 98.5 |

7.5. BBR TEST



Properties concerning the low-temperature behaviour of bituminous binders are tested using a bending beam rheometer BBR acc. to EN 14771 on previously aged bitumen samples using the RTFOT method acc. to EN 12607-1 and the PAV method (conditioning at 100°C for 20 h ± 10 min) acc. to EN 14769. The parameters obtained from the BBR rheometer are:

- Temperature [°C] at $S = 300$ MPa – $T(S_{(60)} = 300 \text{ MPa})$
- Parameter m [-] at the temperature $T(S_{(60)} = 300 \text{ MPa})$

The temperature $T(S_{(60)} = 300 \text{ MPa})$ is determined by interpolation of the creep stiffness–temperature relation curve based on data from at least three measuring points (three temperature values differing by a minimum of 6°C, which will cover the expected temperature $T(S_{(60)} = 300 \text{ MPa})$). Analogically, the value of the parameter m must be determined for each sample.

Table 7.3.

Test results of low-temperature properties of modified and highly modified bitumens acc. to prEN 14023 [research by ORLEN Asphalt]

| BITUMEN TYPE | REOLOGICAL PARAMETERS prEN 14023 – BBR | |
|------------------------|---|---------------------------------------|
| | TEMPERATURE T AT WHICH CREEP STIFFNESS IS 300 MPa | PARAMETER m -value AT TEMPERATURE T |
| | $T(S_{(60)}=300 \text{ MPa}), [^{\circ}\text{C}]$ | $m T(S_{(60)}=300 \text{ MPa}), [-]$ |
| Interpretation | <i>less = better</i> | <i>more = better</i> |
| ORBITON 25/55-60 | -18.1 | 0.283 |
| ORBITON 45/80-55 | -18.6 | 0.290 |
| ORBITON 45/80-65 | -18.5 | 0.284 |
| ORBITON 65/105-60 | -20.7 | 0.295 |
| ORBITON 25/55-80 HiMA | -21.1 | 0.280 |
| ORBITON 45/80-80 HiMA | -21.8 | 0.288 |
| ORBITON 65/105-80 HiMA | -21.6 | 0.304 |

8. OCCUPATIONAL, HEALTH, SAFETY AND ENVIRONMENT PROTECTION

Bitumen in its solid form is not classified as a hazardous substance but its viscous liquid form may pose some hazards when working with it due to the high temperature of storage, handling and transport.

This chapter describes key issues related to the occupational health and safety at work with bituminous binders. The discussed issues concern bitumen of petroleum origin, used in road construction and distributed by ORLEN Asphalt.

8.1. BITUMEN – GENERAL INFORMATION



Bitumen is a non-volatile material obtained from the processing of heavy crude oil fractions. It is a substance of very high viscosity or almost constant consistency at ambient temperature. It is hydrophobic and completely or almost completely soluble in toluene. Bitumen is used as a material for road construction or industrial applications such as production of membranes and other waterproofing materials.

Bitumens for road construction are construction products and, therefore, are subject to Regulation 305/2011 of the European

Parliament and of the Council (so-called CPR Regulation) which sets out harmonised conditions for placing them on the European market [1].

The CPR Regulation, imposes on producers of bituminous binders the obligation to use the 2+ conformity assessment system, including maintenance of the Factory Production Control (FPC) confirmed by appropriate certificates issued by a notified body. The numbers of FPC certificates for all production centres in ORLEN Group are included in Chapter 1.

8.2. CLASSIFICATION OF BITUMEN ACC. TO REACH



The REACH Regulation (ang. **R**egistration, **E**valuation, **A**uthorisation and **R**estriction of **C**hemicals [2]), imposes a number of obligations on companies to ensure maximum safety during the use of the chemical substances they produce or distribute.

According to the requirements of the REACH Regulation, manufacturers of chemicals are obliged to register their substances with the **E**uropean **C**hemicals **A**gency (ECHA).

Bitumens distributed by ORLEN Asphalt in accordance with the above regulations has been registered with ECHA. Registration data are presented in Table 8.1.

Table 8.1.
REACH registration data

| | PRODUCTION PLANT IN PŁOCK MANUFACTURER: PKN ORLEN S.A | PRODUCTION PLANT IN TRZEBINIA MANUFACTURER: ORLEN ASFALT SP.Z O.O. |
|--|--|---|
| Paving grade bitumen Pen25 < 160 [0,1mm] | | |
| No. CAS | 64742-93-4 | 64742-93-4 |
| No. WE | 265-196-4 | 265-196-4 |
| No. REACH | 01-2119498270-36-0067 | 01-2119498270-36-0005 |
| Paving grade bitumen Pen25 > 160 [0,1mm] | | |
| No. CAS | 8052-42-4 | 8052-42-4 |
| No. WE | 232-490-9 | 232-490-9 |
| No. REACH | 01-2119480172-44-0080 | 01-2119480172-44-0010 |

According to REACH, bituminous binders are not classified as environmentally hazardous substances.

Taking this into account, there is e.g. no formal requirement to prepare and provide all users in the supply chain with current safety data sheets for bituminous binders (MSDS – **M**aterial **S**afety **D**ata **S**heet)¹.

Despite that, ORLEN Asphalt, in accordance with the binding industry practice, makes available to all the interested parties up-to-date safety data sheets and information sheets for its products on the company's website – www.orlden-asfalt.pl.

1) Article 31 par. 5 of the REACH Regulation

8.3. CLASSIFICATION OF BITUMEN ACC. TO CLP



The CLP (ang. *C*lassification, *L*abelling, *P*ackaging), Regulation applies to a uniform system of classification, packaging and labelling of hazardous substances and mixtures thereof in the European Union.

According to the CLP Regulation, bituminous binders are not classified as hazardous substances, so all storage areas as well as their transport do not have to be marked with special pictograms indicating specific hazards [3].

8.4. BITUMEN TRANSPORT ACC. TO RID AND ADR



The transport of bitumen is subjected to international regulations on the carriage of dangerous substances, in which bitumen is classified as dangerous due to its high temperature during transport. For that reason, pictograms should be displayed on the means of transport to warn against high temperatures.

Most ORLEN Asphalt products are transported by road or rail tankers.

Road transport of hazardous substances is regulated by the international agreement **ADR** (from the French *L'Accord européen relatif au transport international des marchandises Dangereuses par Route*) [4].

The transport of dangerous goods by rail, on the other hand, is governed by the RID Regulations (from the French *Règlement concernant le transport Internationale ferroviaire des marchandises Dangereuses*) [5].



Fig. 8.1.
ORLEN Asphalt's road tanker (photo by ORLEN Asphalt sp. z o.o.)

8.5. POTENTIAL HEALTH HAZARDS DURING APPLICATION OF BITUMINOUS BINDERS



The following are the most dangerous and likely hazards that may occur when working with **hot bituminous binders**.

8.5.1. BITUMEN FIRE

PREVENTIVE MEASURES

To prevent a fire in storage tanks, bituminous binders should be stored at a temperature of min. 30°C below their flash point.

The flash point of paving grade bitumen, tested in an open cup using the Cleveland's method, is generally above 300°C. 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.

When operating storage tanks, it is important to be aware of the possibility of bitumen deposits on walls and roofs, which, in the presence of oxygen, may be a source of self-ignition. It is recommended that the condition of tanks be periodically inspected and, if deposits are found, they should be cleaned by a specialist company.

If bitumen in the tank is overheated, flammable decomposition products are likely to occur, which increases the risk of fire or even explosion. To minimise the risk of vapour generation, bitumen overheating should be avoided because then the bitumen loses the manufacturer-declared product properties.

Open flames should also not be used in the immediate vicinity of storage tanks or during loading and unloading of the bitumen – such actions may also contribute to fire or explosion.

However, it is worth noting that **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 [7].

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

Bitumen fires should be extinguished with extinguishing agents in such a way as to cut off the supply of oxygen. Suitable extinguishing agents are therefore:

- fire blanket,
- carbon dioxide extinguisher,
- powder extinguisher,
- foam extinguisher,
- sand,
- water spray.

It is important to remember that during a bitumen fire, the emitted gases and vapours are heavier than air and can collect in depressions in the ground, spread just above the ground at a distance from the fire source and thus pose a risk of re-ignition.

The fire environment also produces harmful carbon oxides, a complex mixture of organic bitumen decomposition products and small amounts of sulphur oxides, nitrogen oxides and metal oxides.

PROCEDURE IN THE CASE OF BITUMEN FIRE:

- in any case, immediately notify the nearest fire service,
- if there is no risk to safety:
 - turn off bitumen heating,
 - turn off the circulation pumps and other electrical equipment,
 - shut off the valves, which may contribute to limiting fire spread.

8.5.2. HOT BITUMEN BURNS

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 the third degree).

Burns may occur in different situations: during routine work (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 or unloading hose integrity loss or if shut-off valves work defectively.

To minimise the risk of burns, personal protective equipment should always be worn and health and safety procedures followed.

The basic personal protective equipment that must be used absolutely in all situations is shown in Figure 8.2.

FIRST AID IN CASE OF BURNS FROM HOT BITUMEN:

- burn should be immediately cooled down with cold running water for at least 15 minutes to avoid further skin damage,
- under no circumstances attempt to remove the bitumen from the burned area, as this may lead to further skin damage with subsequent serious complications,
- contaminated clothing can be removed as long as it is not stuck to the skin,
- medical assistance should be immediately sought in each case.

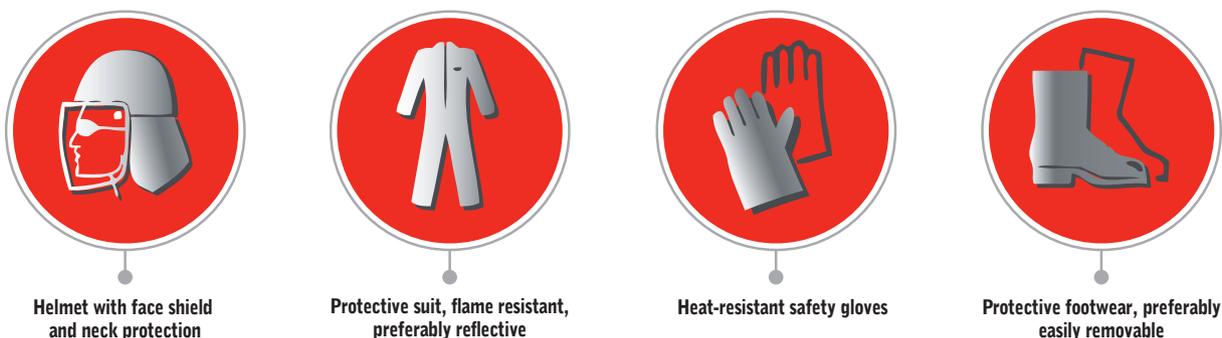


Fig. 8.2.
Basic personal protective equipment [source: <http://www.eurobitume.eu>]

8.5.3. EXPOSURE TO BITUMEN FUMES

Bituminous binders heated to temperatures above 100°C may emit vapours. Bitumen vapours consist of a gas phase and an aerosol phase. For many years, the bitumen industry has been supporting scientific research on the potential occupational hazards resulting from worker exposure to bitumen vapours. The results of such research are published on an ongoing basis on the Eurobitume website [8].

The Regulation of the Polish Minister of Labour and Social Policy [9, 10] on the highest permissible concentrations and intensities of factors harmful to health in the work environment includes the limit values of the highest permissible concentrations of harmful substances. They are marked as:

- **maximum permissible concentration** (*Polish abbreviation NDS*) – the weighted average value of the concentration which impact on an employee during an 8-hour daily and average weekly working time for the period of their professional activity, should not cause negative changes in

their health condition and on the health condition of their future generations;

- **maximum permissible momentary concentration** (*Polish abbreviation NDSch*) – the average value of the concentration that should not cause negative changes in the health condition of an employee if it occurs in the working environment for not longer than 15 minutes and not more frequently than 2 times during the working shift, with a minimal interval of 1 hour;
- **maximum permissible ceiling concentration** (*Polish abbreviation NDSP*) – the concentration value which, due to the risk to the employee's health or life, must not be exceeded at any time in the working environment.

Based on the REACH Regulation and the Regulation of the Polish Minister of Labour and Social Policy [9, 10], maximum exposure limits have been developed for workers in contact with bitumen vapours at the workplace. The data are presented in Table 8.2. and 8.3.

Table 8.2.

Limits of maximum exposure to bitumen fumes, as per the Regulation of the Polish Minister of Labour and Social Policy [9]

| HARMFUL FACTOR | MAXIMUM PERMISSIBLE CONCENTRATION DEPENDING ON EXPOSURE TIME DURING A WORK SHIFT | | |
|--|--|---|--|
| | Maximum permissible concentration (NDS) | Maximum permissible momentary concentration (NDSch) | Maximum permissible ceiling concentration (NDSP) |
| Petroleum bitumen – inhalable fraction [8052-42-4] | 5 mg/m ³ | 10 mg/m ³ | — |

Table 8.3.

Limits of maximum exposure to bitumen fumes, as per the REACH Regulation [2]

| PARAMETER | EXPLANATION | PERMISSIBLE LIMIT |
|--|---|--|
| DNEL_{worker} (<i>derived no-effect level</i>) | Predicted Exposure Level to a chemical, below which no adverse effect on human health is expected | 2.9 mg/m ³ /8h |
| DNEL_{consumer} (<i>derived no-effect level</i>) | | 0.6 mg/m ³ /24h |
| PNEC (<i>predicted no-effect concentration</i>) | Predicted Exposure Level to a chemical, below which no adverse effect on the environmental of concern is expected | None – the substance presents no risk to the environment |

One of the harmful substances that could theoretically be found in bitumen vapours are aromatic hydrocarbons. In 2018/2019, ORLEN Asphalt carried out a research programme on determination of aromatic hydrocarbons from the BTEX group in paving grade and industrial bitumens produced by ORLEN Group.

BTEX is an abbreviation used for a group of volatile organic compounds such as: **benzene, toluene, ethylbenzene** and **xylene**. BTEX aromatic hydrocarbons are considered to be particularly dangerous to human health and pose a serious threat to the environment. Concentration limits of particular compounds at workplaces specified in the Regulation of the Polish Minister of Labour and Social Policy [9] are shown in Table 8.4.

Table 8.4.

Limits of maximum exposure to BTEX hydrocarbons, as per the Regulation of the Polish Minister of Labour and Social Policy [9]

| HARMFUL FACTOR | MAXIMUM PERMISSIBLE CONCENTRATION DEPENDING ON EXPOSURE TIME DURING A WORK SHIFT | | |
|---|--|---|--|
| | Maximum permissible concentration (NDS) | Maximum permissible momentary concentration (NDSch) | Maximum permissible ceiling concentration (NDSP) |
| Benzene [71-43-2] | 1.6 mg/m ³ | — | — |
| Toluene [108-88-3] | 100 mg/m ³ | 200 mg/m ³ | — |
| Ethylbenzene [100-41-4] | 200 mg/m ³ | 400 mg/m ³ | — |
| Xylene (mixture of isomers) [95-47-6], [108-38-3], [106-42-3], [1330-20-7] | 100 mg/m ³ | 200 mg/m ³ | — |

The content of BTEX hydrocarbons in bituminous binders was analysed by gas chromatography coupled with mass spectrometry – GC/MS, Fig. 8.3. The research was performed at the ORLEN UniCRE Research and Development Centre in Czech Republic. The obtained results are shown in Table 8.5.

Table 8.5.

Test results for BTEX content in samples of bituminous binders [source: ORLEN Asphalt internal research]

| BITUMINOUS BINDER | BENZENE | TOLUENE | ETHYLBENZENE | XYLENE (MIXTURE OF ISOMERS) |
|-------------------------------------|-----------|-----------|--------------|-----------------------------|
| Paving grade bitumen 20/30 | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg |
| Paving grade bitumen 50/70 | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg |
| Paving grade bitumen 160/220 | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg |
| Oxidised (Industrial) bitumen 95/35 | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg | < 2 mg/kg |

* Limit of Quantification LOQ² = 2 mg/kg

2) Limit of Quantification (LOQ) – the smallest amount or the lowest concentration of a substance that can be quantified using a given analytical procedure with an adopted accuracy and precision.



Fig. 8.3.

Gas chromatograph – general view (photo by ORLEN Asphalt sp. z o.o., with permission of ORLEN UniCRE, a.s.)

In the course of the conducted tests, no BTEX hydrocarbons were found in the tested samples of bituminous binders.

In each case, the results were below the method's limit of quantification (i.e. below 2 mg/kg).

Additionally, to confirm the absence of BTEX aromatic hydrocarbons in the tested bituminous binders, a very accurate technique was used for both the identification (qualitative determination) and quantitative analysis of volatile and medium-volatile organic compounds – stationary phase microextraction SPME-GC/MS. **As in the case of the GC/MS analysis, no BTEX was found in the tested bitumen samples.**

Nevertheless, when working with hot bitumen, inhalation of vapours and mist of the heated product should be avoided. Prolonged exposure to high concentrations of vapours/smoke from hot bitumen may irritate the respiratory system or eyes, or even cause breathing problems or nausea.

However, most bitumen-related road works are carried out in the open where exposure to fumes is usually lower. On the other hand, when carrying out roadworks in tunnels,

it is advisable to analyse exposure of workers and apply an appropriate solution.

Employees' exposure to bitumen vapours/smoke should be minimised through the application of the so-called best practices [8]:

- keep process temperatures as low as possible,
- work in well-ventilated areas,
- job rotation around the work site,
- use of 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 care if problems with breathing persist.

However, it should be noted that if the process temperature on site is strictly controlled, the work area is open and well ventilated, there is no evidence that bitumen fumes can pose a health risk to workers.

8.5.4. POLYCYCLIC AROMATIC HYDROCARBONS (PAH)

Polycyclic aromatic hydrocarbons abbreviation: PAH are a large group of organic compounds containing from two to several or even more than a dozen aromatic rings per molecule. They are very toxic and harmful to human health and life. There are also 100 compounds in this group but due to their toxicity, effects on humans and the amount of information available, not all of them have been studied. Studies indicate that nine of them have particularly dangerous effects, including carcinogenic effects. These are:

- anthracene,
- benzo[a]anthracene,
- benz[a]pyrene,
- benzo[b]fluoroanthene,
- benzo[k]fluoroanthene,
- benzo[g,h,i]perylene,

- dibenzo[a,h]anthracene,
- chrysene,
- indeno[1,2,3-c,d]pyrene.

The best known hydrocarbon from the group of PAHs is benzo[a]pyrene, which due to its carcinogenicity and prevalence in the environment has been considered as an indicator of the whole group of PAHs, in relation to which have been established carcinogenicity coefficients of individual polycyclic aromatic hydrocarbons [13].

Table 8.6. shows the maximum permissible limits of workers exposure to PAH (including separately benzo[a]pyrene and dibenzo[a,h]anthracene), included in the Regulation of the Polish Minister of Labour and Social Policy [9, 10].

Table 8.6.

Limits of maximum exposure to PAHs as per the Regulation of the Polish Minister of Labour and Social Policy [9, 10]

| HARMFUL FACTOR | MAXIMUM PERMISSIBLE CONCENTRATION DEPENDING ON EXPOSURE TIME DURING A WORK SHIFT | | |
|--|--|---|--|
| | Maximum permissible concentration (NDS) | Maximum permissible momentary concentration (NDSCh) | Maximum permissible ceiling concentration (NDSP) |
| Polycyclic aromatic hydrocarbons (PAH) (sum of products of concentrations and carcinogenicity factors of nine carcinogenic PAH)* | 0.002 mg/m ³ | — | — |
| Benzo[a]pyrene [50-32-8] | 0.002 mg/m ³ | — | — |
| Dibenzo[a,h]anthracene [92-84-2] | 0.004 mg/m ³ | — | — |

* The values of the carcinogenicity factors (k) are:

| | |
|-------------------------------|-------------|
| anthracene | 0.01 |
| benzo[a]anthracene | 0.10 |
| benzo[a]pyrene | 1.00 |
| benzo[b]fluoroanthene | 0.10 |
| benzo[k]fluoroanthene | 0.10 |
| benzo[g,h,i]perylene | 0.01 |
| dibenzo[a,h]anthracene | 5.00 |
| chrysene | 0.01 |
| indeno[1,2,3-c,d]pyrene | 0.10 |

The analysis of air pollution at workplaces during the use of bituminous binders is the subject of many research works [14, 15, 16]. The problem of occurrence of polycyclic aromatic hydrocarbons in bitumen vapours concerns mainly workers exposed to direct contact of bitumen fumes with the skin and respiratory system. However, studies conducted among

workers performing jobs in the area of direct exposure to bitumen fumes do not give conclusive results. Authors of these studies pay particular attention to many other factors affecting the final results, which makes it impossible to unequivocally assess the impact of the bitumen fumes on human health [15, 18, 20].

This results both from the complexity of interactions to which the human organism is subjected in everyday life as well as the methodological approach used to analyse the composition of emitted vapours or insufficiently sensitive analytical methods.

ORLEN Asphalt periodically monitors PAH content in bituminous binders produced by ORLEN Group. In 2020, tests were

carried out to determine the content of polycyclic aromatic hydrocarbons (PAH) in samples of paving grade, industrial, modified and highly modified bitumens. The measurements were made with the gas chromatography technique with a coupled mass spectrometer – GC/MS in the Oil and Gas Institute – National Research Institute in Cracow, Poland. The results are shown in Table 8.7.

Table 8.7.

Results of tests of benzo[a]pyrene content and total quantity of PAH in samples of bituminous binders [source: ORLEN Asphalt internal research]

| BITUMINOUS BINDER | BENZO[A]PYRENE CONTENT | TOTAL POLYCYCLIC AROMATIC HYDROCARBONS (PAH) CONTENT* |
|---|------------------------|---|
| | [mg/kg] | [mg/kg] |
| Paving grade bitumen 20/30 | 0.2 | 2.1 |
| Paving grade bitumen 50/70 | 0.1 | 1.9 |
| Paving grade bitumen 160/220 | 0.9 | 8.9 |
| Industrial bitumen 95/35 | 0.2 | 2.8 |
| Polymer modified bitumen PMB 45/80-55 | <0.1 | 1.0 |
| Highly modified bitumen PMB 45/80-80 HiMA | <0.1 | 1.0 |

* Tests on sum of PAHs: benzo(a)anthracene, chrysene, benzo(e)pyrene, benzo(b)fluoranthene, benzo(j)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, dibenzo(ah)anthracene

Carcinogenic effects of benzo[a]pyrene is in effect when its content in the binder exceeds 50 mg/kg [11]. However, the benzo[a]pyrene content in the tested bitumen samples was in the range of 0.1–0.9 mg/kg. On the other hand, the total PAH content did not exceed 8.9 mg/kg.

Thus, it can be concluded that as a **result of the research, no exceedance of PAH content was recorded in any of the tested samples.**

Nevertheless, it is advisable to be cautious when working with hot bitumen and, if possible, avoid inhaling bituminous binder vapours.

8.5.5. HYDROGEN SULPHIDE

The elemental composition of bituminous binders varies, depending among other things on the chemical nature of the crude oil from which they are produced. Most bitumens contain small amounts of sulphur in their elemental composition. 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.

Hydrogen sulphide (H_2S) is a poisonous and flammable gas, heavier than air, which can accumulate in low and confined spaces. Typical symptoms of hydrogen sulphide poisoning include eye irritation, nausea, vomiting, dizziness and headaches. Data

published on the website of the Polish Central Institute for Occupational Protection says that long-term exposure to H_2S concentrations above 10 mg/m^3 causes irritation of the eyes, nose, throat and lungs. Exposure to concentrations above 300 mg/m^3 can cause severe pulmonary oedema. H_2S levels above 750 mg/m^3 cause loss of consciousness within seconds and can be fatal. Hydrogen sulphide in high concentrations exceeding 7000 mg/m^3 causes death within a few to several seconds [22].

Table 8.8. shows the maximum permissible limits of the exposure to hydrogen sulphide, included in the Regulation of the Polish Minister of Labour and Social Policy [9].

Table 8.8.

Limits of maximum exposure to hydrogen sulphide as per the Regulation of the Polish Minister of Labour and Social Policy [9]

| HARMFUL FACTOR | MAXIMUM PERMISSIBLE CONCENTRATION DEPENDING ON EXPOSURE TIME DURING A WORK SHIFT | | |
|---|--|---|--|
| | Maximum permissible concentration (NDS) | Maximum permissible momentary concentration (NDSch) | Maximum permissible ceiling concentration (NDSP) |
| Hydrogen sulphide [7783-06-4] | 7 mg/m^3 | 14 mg/m^3 | — |

With this in mind, special precautions must be taken before entering an emptied bitumen storage tank, in accordance with safety regulations. These include lowering the temperature of the tank, ventilating it, then leaving it under a constant flow of fresh air. 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. A worker entering the tank should be properly equipped with personal protective equipment and have a personal H_2S concentration detector.

In the case of poisoning or overexposure to hydrogen sulphide vapour, it is necessary to [22]:

- remove the affected person from the area of exposure and ensure absolute calm in a semi-sitting or sitting position (any physical exertion may trigger pulmonary oedema),
- if the victim is conscious, administer oxygen through the breathing mask,
- if the victim is not breathing, start cardiopulmonary resuscitation,
- in either case, call for medical assistance.

8.5.6. BITUMEN FOAMING IN THE PRESENCE OF WATER

When hot bitumen comes into contact with water, the so-called bitumen foaming process occurs. It results from a rapid increase in the volume of water and its conversion into water vapour. It generates a real hazard of bitumen boiling over the tank. Bitumen foaming may be accompanied by hot material 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 the unloading 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.

The foaming process is very dangerous and can cover quite a large area around the tank or tanker. Always keep a safe distance when pumping hot binder.

8.5.7. IMPACT OF BITUMEN ON ENVIRONMENT

According to REACH, bitumen is not classified as an environmentally hazardous substance. REACH requires, among other things, that the properties of substances be assessed for persistence, bioaccumulation and toxicity (PBT) and whether they are very persistent and very bioaccumulative (vPvB). **Bituminous binders are not classified as PBT or vPvB substances.**

Bitumen also poses no risk to soil or the aquatic environment due to its relatively rapid solidification and very low solubility in water. Bitumen spills can be cleaned up easily using standard, readily available equipment (i.e.: rakes, shovels, loaders, etc.).

Bitumen at ambient temperature, i.e. when built into the pavement, does not emit any chemical compounds into the atmosphere.

It should also be noted that the bituminous binder in the asphalt mixture is solid and poses no risk to health or the environment. What is more, bitumen is one of very few construction products that is considered as ecological, as it can be 100% recyclable and then re-used for road pavement construction.

Considering the above, it can be concluded that bituminous binders are environmentally safe materials.

8.5.8. OTHER HAZARDS

One potential source of risk in the use of bituminous binders is that they can be mixed with other substances or additives. Such mixtures can generate additional hazards that are unspecified and unknown to bitumen producers.

The manufacturers of those mixtures are responsible for the changes that may cause bitumen to become a substance hazardous to human life or the environment.



Reduced road construction costs through reduced course thickness



Increased resistance to rutting, potholes and cracks



Reduced traffic noise

Improved durability due to new polymer content



ORBITON HiMA pavements are fully recyclable



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Member of the Management Board

RESEARCH, DEVELOPMENT AND INNOVATION DEPARTMENT

Company department at ORLEN Asphalt. Active from the company's foundation in 2003. 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 Department's achievements include patent applications and numerous awards and distinctions, e.g. a distinction for modified bitumen ORBITON in the EUROPRODUCT competition organised under the auspices of the Polish Minister of Economy and the Polish Agency for Enterprise Development. Modified bitumens ORBITON 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. Multigrade bitumen BITREX won the Gold Medal at the International Invention Fair IWIS 2007. In 2014, highly polymer modified binders ORBITON HiMA 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.

Technical consultancy is available for the company's customers at: doradztwotechnologiczne@orlen-asfalt.pl

WE LOOK FORWARD TO POTENTIAL COOPERATION WITH YOU

Although many people associate research and development with work in a laboratory, ORLEN Asphalt's activities in this area are much broader.

For the company's customers, we mainly provide technical advisory services on the application and use of bituminous binders. Moreover, we also cooperate with road administration, design companies, road construction companies and research institutes in the area of road construction technology,

research of asphalt mixtures, as well as new applications and search for non-standard ideas and solutions.

We are open to cooperation with all those who seek to create new initiatives to better manage the road network, strengthen their company or want to develop individually. We invite people with and without connections to the road industry, who have ideas we could implement together.

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- [2] Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH), establishing a European Chemicals Agency, amending Directive 1999/45/EC and repealing Council Regulation (EEC) No 793/93 and Commission Regulation (EC) No 1488/94 as well as Council Directive 76/769/EEC and Commission Directives 91/155/EEC, 93/67/EEC, 93/105/EC and 2000/21/EC
- [3] Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006
- [4] European Agreement concerning the International Carriage of Dangerous Goods by Road - ADR (in French: Accord européen relatif au transport international des marchandises dangereuses par route), together with the Government Statement of 28 February 2017 on the entry into force of the amendments to Annexes A and B to the European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR), made at Geneva on 30 September 1957.
- [5] Regulations for the International Carriage of Dangerous Goods by Rail - RID (French: Règlement concernant le transport international ferroviaire des marchandises dangereuses), together with the Government Declaration of 29 May 2017 on the entry into force of the amendments to the Regulations for the International Carriage of Dangerous Goods by Rail (RID), constituting Appendix C to the Convention concerning International Carriage by Rail (COTIF), done at Berne on 9 May 1980.
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LIST OF STANDARDS

AASHTO M 320-17 Standard specification for Performance-Graded Asphalt Binder

AASHTO M 332 Standard Specification for Performance-Graded Asphalt Binder Using Multiple Stress Creep Recovery (MSCR) Test

AASHTO MP1 Standard specification for performance graded asphalt binder

AASHTO PP 42 Standard practice for determination of low-temperature performance grade (PG) of asphalt binders

AASHTO T 315 Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

AASHTO T 350 Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

AASHTO TP 101-14 Estimating Damage Tolerance of Asphalt Binders Using the Linear Amplitude Sweep

AASHTO TP 70 Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

ASTM D 4402 Standard Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer

ASTM D 7175 Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer

ASTM D 7405 Standard Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer

EN 12595 Bitumen and bituminous binders. Determination of kinematic viscosity

EN 12596 Bitumen and bituminous binders. Determination of dynamic viscosity by vacuum capillary

EN 13302 Bitumen and bituminous binders. Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus

EN 13702 Bitumen and bituminous binders. Determination of dynamic viscosity of modified bitumen by cone and plate method

EN 12593 Bitumen and bituminous binders. Determination of the Fraass breaking point

EN 12594 Bitumen and bituminous binders. Preparation of test samples

EN 12595 Bitumen and bituminous binders. Determination of kinematic viscosity

EN 12596 Bitumen and bituminous binders. Determination of dynamic viscosity by the vacuum capillary method

EN 12607-1 Bitumen and bituminous binders. Determination of the resistance to hardening under influence of heat and air Part 1: RTFOT method

EN 12607-2 Bitumen and bituminous binders. Determination of the resistance to hardening under influence of heat and air Part 2: TFOT method

EN 12607-3 Bitumen and bituminous binders. Determination of the resistance to hardening under influence of heat and air Part 3: RFT method

EN 12697-1 Bituminous mixtures. Test methods for hot mix asphalt. Part 1: Soluble binder content

EN 12697-2 Bituminous mixtures. Test method. Part 2: Determination of particle size distribution

EN 12697-4 Bituminous mixtures. Test methods. Part 4: Bitumen recovery - Fractionating column

EN 12697-8 Bituminous hot mixtures. Test methods. Part 8: Determination of void characteristics of bituminous specimens

EN 12697-11 Bituminous mixtures. Test methods for hot mix bituminous mixtures. Part 11: Determination of the affinity between aggregate and bitumen

EN 12697-12 Bituminous mixtures. Test methods for hot mix bituminous mixtures. Part 12: Determination of the water sensitivity of bitumen sample

EN 12697-18 Bituminous mixtures. Test methods. Part 18: Binder drainage

EN 12697-22 Bituminous mixtures. Test methods for hot mix asphalt. Part 22: Wheel tracking

EN 12697-24 Bituminous mixtures. Test methods. Part 24: Resistance to fatigue

EN 13108-1 Bituminous mixtures. Material specifications. Part 1: Asphalt concrete

EN 13108-9 Bituminous mixtures. Material specifications. Part 9: Asphalt for Ultra-Thin Layer (AUTL)

EN 13108-20 Asphalt mixtures. Material specifications. Part 20: Type Testing

EN 13108-21 Bituminous mixtures. Material specifications. Part 21: Factory Production Control

EN 13302 Bitumen and bituminous binders. Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus

EN 13398 Bitumen and bituminous binders. Determination of elastic recovery of modified bitumen

EN 13589 Bitumen and bituminous binders. Determination of tensile properties of modified bitumen by the force ductility method

EN 13632 Bitumen and bituminous binders. Visualisation of polymer dispersion in polymer modified bitumen

EN 13702:-1 Bitumen and bituminous binders. Determination of dynamic viscosity of modified bitumen. Part 1: Cone and plate method

EN 13703 Bitumen and bituminous binders. Determination of deformation energy (standard withdrawn)

EN 1426 Bitumen and bituminous products. Determination of needle penetration

EN 1427 Bitumen and bituminous products. Determination of the softening point. Ring and Ball method

EN 14769 Bitumen and bituminous binders. Accelerated long-term ageing conditioning by pressure ageing vessel(PAV)

EN 14770 Bitumen and bituminous binders. Determination of complex shear modulus and phase angle. Dynamic Shear Rheometer (DSR)

EN 14771 Bitumen and bituminous binders. Determination of flexural creep stiffness. Bending Beam Rheometer (BBR)

EN 15326 Bitumen and bituminous binders. Measurement of density and specific gravity. Capillary-stoppered pycnometer method

EN ISO 4259-1 Petroleum and related products. Precision of measurement methods and results. Part 1: Determination of precision data in relation to methods of test.

EN ISO 4259-2 Petroleum and related products. Precision of measurement methods and results. Part 2: Interpretation and application of precision data in relation to methods of test

EN ISO 3838 Crude petroleum and liquid or solid petroleum products. Determination of density or relative density. Capillary-stoppered pycnometer and graduated bicapillary pycnometer methods PN-EN 12591:2010 Asfalty i lepiszcza asfaltowe. Wymagania dla asfaltów drogowych [EN 12591:2009 Bitumen and bituminous binders. Specifications for paving grade bitumen]

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PN-EN 14023:2011/Ap2 2020-02 Bitumen and bituminous binders. Specification framework for polymer modified bitumen (Polish National Annex)

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