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Interlaboratory tests of paving grade bitumen and polymer modified bitumen

In the paper the results of interlaboratory round-robin tests are presented. The RR program was executed for paving grade 50/70 bitumen and polymer modified bitumen 45/80-55. For both binders similar test programs have been prepared, starting from fundamental properties like Pen@25, Softening Point, Softening Point after RTFOT, Fraass breaking point, elastic recovery etc. and with use of more contemporary test methods as complex modulus G*, phase angle δ (60°C, f = 1.59 Hz (10 rad/s), PMB microstructure etc. Finally 8 properties for unmodified bitumen were tested and 9 for PMBs. In the program 24 laboratories from three european countries participated representing different kinds of labs – private owned, technical universities, road administration and refinery control labs. The final results were evaluated with statistical and z-score method. Surprisingly results showed that the best methods with high reproductibility are complex modulus G* and phase angle δ.

Key words: interlaboratory tests, round-robin tests, paving grade bitumen, polymer modified bitumen, statistical evaluation.

Badania porównawcze asfaltów drogowych i modyfikowanych polimerami (PMB)

W artykule zaprezentowano wyniki badań międzylaboratoryjnych zorganizowanych przez ORLEN Asfalt w 2012 roku. Program badań obejmował dwa rodzaje lepiszczy asfaltowych: asfalt drogowy 50/70 według PN-EN 12591 oraz asfalt modyfikowany polimerami PMB 45/80-55 według PN-EN 14023. Wśród wybranych parametrów oceny asfaltu drogowego 50/70 i modyfikowanego PMB 45/80-55 znalazły się badania obligatoryjne (zawarte w normach, w oparciu o które produkowane są asfalty) oraz badania funkcjonalne oznaczane w reometrze dynamicznego ścinania DSR. Finalnie do porównań wybrano 8 właściwości asfaltu drogowego i 9 właściwości asfaltu modyfikowanego. W badaniach międzylaboratoryjnych wzięły udział 24 laboratoria reprezentujące zarówno inwestorów z branży drogowej jak i typowe jednostki badawcze. Otrzymane wyniki badań zostały poddane analizie statystycznej i ocenione zgodnie z ogólnymi zasadami opisanymi w normach: PN-EN ISO/IEC 17043:2011 *Ocena zgodności – Ogólne wymagania dotyczące badania biegłości* oraz ISO 13528:2005 *Statistical methods for use in proficiency testing by interlaboratory comparisons*.

Słowa kluczowe: badania międzylaboratoryjne, badania porównawcze, asfalt drogowy, asfalt modyfikowany, analiza statystyczna.

Introduction

The ORLEN Asfalt Research and Development Department has been making analyses of bitumens and gathering the data of the tests conducted for many years. The results of our own research, as well as that of other research institutions which we have obtained via data exchange during road construction-related conferences and seminars, have led the authors of this article to conduct an analysis of the deviations in the results of bituminous binders achieved by various laboratories, and by different operators (reproducibility). The scope of the project was an assessment of credibility received test results for paving grade and modified bitumen.

In mid-2012, ORLEN Asfalt organised a *proficiency test*[1] *via inter-laboratory comparison*[2], on the basis of guidelines included in standards PN-EN ISO/IEC 17043:2011 and ISO 13528:2005. The statistical processing of the test results was carried out by the ORLEN Laboratorium Sp. z o.o. company of the PKN ORLEN Corporate Group [4, 5, 6].

Purpose, Object and Scope of Performed Tests

The purpose of the performed test project was an objective and impartial assessment of the quality of parameters of selected paving grade and modified bituminous binders by various road laboratories.

The tests were carried out on **paving grade bitumen 50/70** (sample A) as per PN-EN 12591, and **polymer modified bitumen PMB 45/80-55** (sample B), as per PN-EN 14023 (National Annexe of 2011). Bitumen samples with a volume of 30 litres were extracted from ready-made products tanks on one of the large-tank installations belonging to ORLEN Asfalt Sp. z o.o., in accordance with PN-EN 58 (pt. 8.1.2. *Sampling valve installed in tank walls*). Each sample was mixed in order to achieve exact homogeneity. Afterwards 30 "sub-samples" were extracted from the sample, with a volume of 1 litre each. The sub-samples were packaged in cardboard boxes lined internally with aluminium foil – specially designed for transporting and storage of bituminous binders. Such prepared and tagged testing materials "Sample A" and "Sample B" were simultaneously handed over to the comparative test participants via a logistics company.

Table 1. Scope of compared selected tests of paving grade bitumen 50/70 (Sample A) and polymer modified bitumen PMB 45/80-55 (Sample B)

No.	Property	Abbreviation	Unit of Measurement	50/70 (Sample A)	45/80-55 (Sample B)
1.	Penetration at 25°C as per PN-EN 1426:2009	Pen25 1) 2)	0.1mm	Yes	Yes
2.	R&B softening point, as per PN-EN 1427:2009	SP 1) 2)	°C	Yes	Yes
3.	Fraass breaking point, as per PN-EN 12593:2009	Fraass 1) 2)	°C	Yes	Yes
4.	Elastic recovery at 25°C as per PN-EN 13398:2012	ER25 2)	%	No	Yes
5.	Paraffin content, distillation method as per PN-EN 12606-1:2009	Paraf 1)	% m/m	Yes	No
6.	Complex module G* in temperature of 60°C, frequency 1.59 Hz (10 rad/s), plate-plate arrangement, plate diam. 25 mm, gap 1 mm, testing in controlled stress mode, with the stress value set at 0.12 kPa (120 Pa) in DSR as per PN-EN 14770:2009	G* 1) 2)	kPa	Yes	Yes
7.	Phase angle δ in temperature of 60°C, frequency 1.59 Hz (10 rad/s), plate-plate arrangement, plate diam. 25 mm, gap 1 mm, testing in controlled stress mode, with the stress value set at 0.12 kPa (120 Pa) in DSR as per PN-EN 14770:2009	Delta 1) 2)	°	Yes	Yes
8.	Softening point R&B after RTFOT hardening as per PN-EN 1427:2009 and PN-EN 12607-1:2009	SP^{RTFOT} 1) 2)	°C	Yes	Yes
9.	Increase of softening point R&B after RTFOT hardening as per PN-EN 1427:2009 and PN-EN 12607-1:2009	ΔSP^{RTFOT} 1) 2)	°C	Yes	Yes
10.	Force ductility. Energy value at 5°C as per PN-EN 13589:2011 and PN-EN 13703:2009	FD5 2)	J/cm^2	No	Yes
11.	Visual assessment of dispergation of polymer in polymer modified bitumen (sample B) as per PN-EN 13632:2010	Image	–	No	Yes

1) Abbreviation of full description of tested characteristic used in Table 2 and 3.
2) Abbreviation of full description of tested characteristic used in Table 4 and 5.

The selected parameters for the assessment of paving grade bitumen 50/70 and polymer modified bitumen PMB 45/80-55 included obligatory tests (admitted in EN standards serving as the basis for bitumen production) and additional functional tests in a Dynamic Shear Rheometer (DSR). The functional tests (currently excluded from the scope of product specification standards) are much better in terms of quality characterisation of bituminous binders than strictly standard tests, which should be considered rather as type-classification tests or commercial quality-defining tests. The scope of the compared selected tests of paving grade bitumen and polymer modified bitumen is shown in Table 1.

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[1] (PT – Proficiency Testing) – determination, via inter-laboratory comparisons, of the ability of a laboratory to perform tests or ratings (sometimes proficiency testing is also called EQA – External Quality Assurance) [1].

[2] (ILC – Interlaboratory Comparisons) – organisation, performance and evaluation of tests or ratings of the same or various objects of testing or rating by at least two different laboratories, in accordance with previously defined conditions [1].

Information about Test Project Participants and Confidentiality of obtained results

24 laboratories participated in the interlaboratory comparison tests, which included facilities representing road construction investors, as well as standard research institutions. The structure of institutions administrating the participating laboratories was as follows:

- State-owned belonging to the national road administration in Poland – 8,
- Technical universities and research institutes – 7,
- Road construction companies – 5,
- Private – 1,
- Bitumen manufacturers – 3.

Among the 24 laboratories, 8 had certificates of accreditation accordant with standard EN ISO 17025. It has to be noted that the scope of accreditation of the laboratories did not fully cover the tests conducted under the proficiency test programme [1].

In view of generally accepted regulations governing comparison tests, the organizer of the tests – ORLEN Asfalt – guaranteed the confidentiality of all information related to the participation in the programme of all participants. Therefore, for the purposes of the project, all names of laboratories have been coded by appropriating to them randomly chosen numbers from 1 to 24, and each participant has been given its own personal code number. Each participant was also obliged to carry out the tests and hand over the results directly and only to the organizer, without contacting any of the other laboratories.

Implemented Methodology of Statistical Evaluation of Results

Test results received from the Participants of interlaboratory tests were subject to standard statistical evaluation, using a MS Excel spreadsheet. The list of obtained results and calculated statistical measures are shown in Table 2 and 3 (for paving grade bitumen 50/70) and in Table 4 and 5 (for polymer modified bitumen PMB 45/80-55). One exception is the evaluation of the results of visual assessment of dispergation of polymer in polymer modified bitumen (PMB), where in accordance with the appropriate standard, the obtained result does not represent a number, but a microscopic image and the implemented code of description of the microstructure. In this case, statistical approach to the evaluation of obtained results, as was done in the case of other test results, is not possible, and the evaluation of variation in test results can only be done in a descriptive way.

In accordance with the implemented methodology of statistical evaluation of test results, the definition of *attributed value* X^*, *standard uncertainty of attributed value* u_x, *standard deviation for the evaluation of proficiency* $\sigma\hat{}$ and the evaluation of the tests results obtained by the participants, was carried out in accordance with the general rules described in standards [3] and [2], with the consideration of the following particular boundary conditions implemented for the purposes of this analysis:

- *Attributed value* X^* was defined as the mean value of laboratory test results received from the participants, with the consideration of techniques minimising the effect of extreme values using the resistant statistical method of Algorithm A [2]. The attributed value is the so-called "*resistant mean value*" [4, 6].
- *The standard uncertainty* u_x *of the attributed value* X^* was estimated only for the purpose of tests, where the number of participating laboratories was less than 10 [4, 6].
- The estimated *standard uncertainty* was compared to *standard deviation applied to the evaluation of proficiency* $s\hat{}$. If the criterion of $u_x \leq 0{,}3\ s\hat{}$ was not fulfilled, the effect of the uncertainty of definition of the attributed value on the evaluation of results of tests obtained by the participants was taken into account, i.e. instead of *z-score*, the value of *z'-score* was applied [4, 6].
- For the purpose of evaluation of test results, *resistant standard deviation* s^*, estimated on the basis of results sent by the participants, was applied instead of the *standard deviation for evaluation of proficiency* $s\hat{}$. For the purpose of estimation of *standard deviation on the basis of test results* s^*, the strong statistical method of Algorithm A was applied [2, 4, 6].
- Only in one case (analysis of the results of elastic recovery at 25°C as per PN-EN 13398:2012) standard deviation calculated on the basis of results sent by the participants s was applied, after the rejection of the extreme value [4, 6].
- For the purpose of statistical evaluation of individual results of tests from each of the laboratories, the index of *z-score*, calculated as per formula (1), or *z'-score*,

calculated as per formula (2), was applied. The *z'-score* index was applied in cases when the boundary condition of $u_x \leq 0{,}3\sigma^{\wedge}$ was not fulfilled [4, 6].

$$z = \frac{x - X^*}{\sigma^{\wedge}} \tag{1}$$

$$z' = \frac{x - X^*}{\sqrt{\sigma^{\wedge 2} + u_x^2}} \tag{2}$$

where:

x – result obtained by the Participant,
X^* – attributed value,
$\sigma^{\wedge}$ – standard deviation,
u_x – standard uncertainty of attributed value X*.

Test results obtained by the laboratories were subject to evaluation with the implementation of the following criteria in accordance with the *z-score* or *z'-score* index [4, 6]:
$|z| \leq 2$ – satisfactory result,
$2 < |z| < 3$ – questionable result,
$|z| \geq 3$ – unsatisfactory result.

For the purposes of complementing the statistical evaluation carried out according to the *z-score* or *z'-score* index, the test results were further expanded with standard statistical measures for data sets, i.e. *arithmetic average, median*[3]*, standard deviation*[4]*, minimum value, maximum value and range*[5].

Obtained Interlaboratory Test Results and their Statistical Evaluation

Table 2. Comparative test results and their statistical evaluation – Sample A – paving grade bitumen 50/70

Method	Pen25		SP		Fraass		Paraf	
	result	evaluation	result	evaluation	result	evaluation	result	evaluation
	[0.1 mm]	*z-score*	[°C]	*z-score*	[°C]	*z'-score*	[% *m/m*]	*z-score*
No. of test participants *p*	24		23		13		7	
Attributed Value *X**		61.750		49.100		–16.130		1.650
Std. Dev. *s**		3.147		0.556		3.474		0.575
Uncertainty of Attr. Val. u_x		0.800		0.100		1.200		–
Arithm. Ave.	61.20		49.10		–16.20		1.70	
Median	62.00		49.00		–17.00		1.70	
Std. Dev.	4.77		0.50		3.16		0.51	
Min. Value	48.20		48.30		–22.00		0.87	
Max. Value	72.00		50.00		–12.00		2.30	
Range	23.80		1.70		10.00		1.43	
Comments to Statistical Evaluation of Results	24 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Nineteen laboratories achieved satisfactory results, three laboratories achieved questionable results, and two laboratories achieved unsatisfactory results.		23 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Laboratories achieved satisfactory results.		13 laboratories participated in the comparison. Results were evaluated on the basis of *z'-score*, because standard uncertainty of the attributed value was greater than 0.3 *s*^. Laboratories achieved satisfactory results.		7 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Laboratories achieved satisfactory results. Due to a small number of participating laboratories, the above assessment has an informative role only.	

Tables 2, 3 and 4, 5 [4, 6] present results of comparative tests received from 24 laboratories participating in the research programme, including their statistical analysis and basic statistical measures. Table 6 [5] presents the results of comparative tests of the visual assessment of dispergation of polymer. Due to the specific character of these results, their analysis is limited to a descriptive comment.

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[3] The value of a characteristic located in an ordered series, with identical number of observations above and below it.

[4] Informs how far the values in a given set are scattered beyond its arithmetic average. The smaller value of standard deviation, the bigger the concentration of observed values around the arithmetic average.

[5] The difference between the lowest and highest value of a statistical characteristic in a given set.

Table 3. Comparative test results and their statistical evaluation – Sample A – paving grade bitumen 50/70 (continued)

Method	G*		Delta		SP^{RTFOT}		ΔSP^{RTFOT}	
	result	evaluation	result	evaluation	result	evaluation	result	evaluation
	[kPa]	*z-score*	[°]	*z-score*	[°C]	*z'-score*	[°C]	*z-score*
No. of test participants *p*	7		7		14		14	
Attributed Value *X**		2.540		84.710		54.330		5.130
Std. Dev. *s**		0.064		0.311		0.525		0.512
Uncertainty of Attr. Val. u_x		–		–		0.200		–
Arithm. Ave.	2.50		84.70		54.30		5.10	
Median	2.50		84.70		54.40		5.20	
Std. Dev.	0.06		0.35		0.59		0.47	
Min. Value	2.45		84.00		52.80		4.40	
Max. Value	2.60		85.10		55.00		6.00	
Range	0.16		1.07		2.20		1.60	
Comments to Statistical Evaluation of Results	7 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Laboratories achieved satisfactory results. Due to a small number of participating laboratories, the above assessment has an informative role only.		7 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Six laboratories achieved satisfactory results, one laboratory achieved a questionable result. Due to a small number of participating laboratories, the above assessment has an informative role only.		14 laboratories participated in the comparison. Results were evaluated on the basis of *z'-score*, because standard uncertainty of the attributed value was greater than 0.3 *s*^. Thirteen laboratories achieved satisfactory results, one laboratory achieved a questionable result.		14 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Laboratories achieved satisfactory results.	

Table 4. Comparative test results and their statistical evaluation – Sample B – polymer modified bitumen PMB 45/80-55

Method	Pen25		SP		Fraass		ER25	
	result	evaluation	result	evaluation	result	evaluation	result	evaluation
	[0.1 mm]	*z-score*	[°C]	*z-score*	[°C]	*z'-score*	[%]	*z-score*
No. of test participants *p*	23		22		13		18	
Attributed Value *X**		56.710		62.20		–19.130		84.430
Std. Dev. *s**		2.706		1.118		3.967		1.392
Uncertainty of Attr. Val. u_x		0.700		0.3		1.400		0.400
Arithm. Ave.	56.80		62.20		–19.10		84.40	
Median	56.00		62.20		–19.00		84.80	
Std. Dev.	5.41		1.06		3.50		1.94	
Min. Value	41.00		60.50		–25.00		82.00	
Max. Value	73.80		64.60		–13.70		90.00	
Range	32.80		4.10		11.30		8.00	
Comments to Statistical Evaluation of Results	23 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Twenty one laboratories achieved satisfactory results, two laboratories achieved unsatisfactory results.		22 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Twenty one laboratories achieved satisfactory results, one laboratory achieved a questionable result.		13 laboratories participated in the comparison. Results were evaluated on the basis of *z'-score*, because standard uncertainty of the attributed value was greater than 0.3 *s*^. Laboratories achieved satisfactory results.		18 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Seventeen laboratories achieved satisfactory results, one laboratory achieved an unsatisfactory result.	

Table 5. Comparative test results and their statistical evaluation – Sample B – polymer modified bitumen PMB 45/80-55 (continued)

Method	G*		Delta		SP^{RTFOT}		ΔSP^{RTFOT}		FD5	
	result	evaluation	result	evaluation	result	evaluation	result	evaluation	result	evaluation
	[kPa]	*z-score*	[°]	*z-score*	[°C]	*z'-score*	[°C]	*z'-score*	[J/cm^2]	*z-score*
No. of test participants p	7		7		14		14		8	
Attributed Value X^*		4.780		66.010		65.810		3.670		8.110
Std. Dev. s^*		0.281		0.311		0.586		1.148		1.107
Uncertainty of Attr. Val. u_x		–		–		0.200		–		–
Arithm. Ave.	4.70		66.00		65.70		3.70		8.00	
Median	4.80		66.00		65.70		3.60		7.80	
Std. Dev.	0.23		0.29		0.76		1.11		0.99	
Min. Value	4.43		65.60		63.80		2.00		6.90	
Max. Value	5.10		66.40		66.60		5.90		9.80	
Range	0.67		0.80		2.80		3.90		2.90	
Comments to Statistical Evaluation of Results	7 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Laboratories achieved satisfactory results. Due to a small number of participating laboratories, the above assessment has an informative role only.		7 laboratories participated in the comparison. Results were evaluated on the basis of *z-score*. Laboratories achieved satisfactory results. Due to a small number of participating laboratories, the above assessment has an informative role only.		14 laboratories participated in the comparison. Results were evaluated on the basis of *z'-score*, because standard uncertainty of the attributed value was greater than 0.3 *s*^. Twelve laboratories achieved satisfactory results, one laboratory achieved a questionable result, one laboratory achieved an unsatisfactory result.		14 laboratories participated in the comparison. Results were evaluated on the basis of *z'-score*, because standard uncertainty of the attributed value was greater than 0.3 *s*^. Laboratories achieved satisfactory results.		10 laboratories participated in the comparison. Two laboratories were not evaluated, because they stated „rupture" in the result column. Results from eight laboratories were evaluated according to *z-score*. Laboratories achieved satisfactory results. One laboratory gave results for a temperature of 10°C only thus the result was not evaluated.	

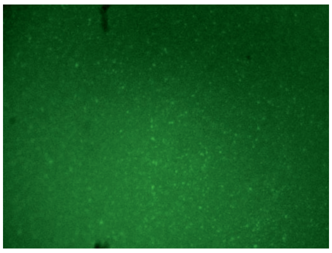
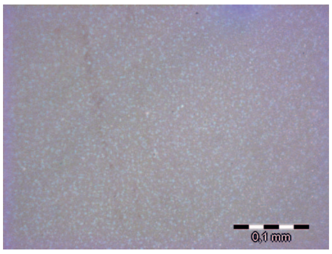
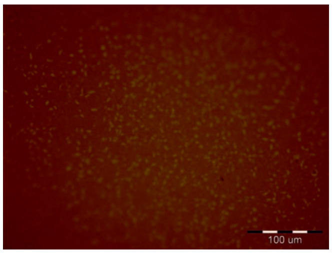
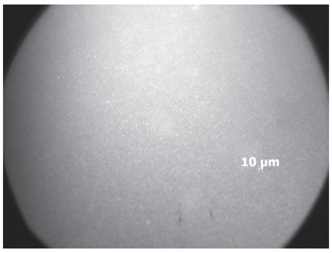
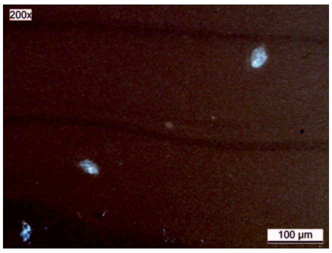
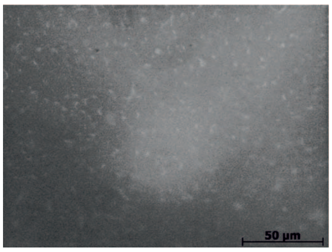
For tests results received from the participants, and for each tested characteristic (apart from the visual assessment of dispergation of polymer in polymer modified bitumen), a statistical analysis of the results was performed on the basis of relevant literature [2] and [3].

All results received from the participants were additionally verified using the *Dixon test*. In order to detect whether the highest or lowest result value is an outlying value, using the available ordered ascending data set x_i for $i = 1, 2, \ldots, p$, the respective Q_{max} and Q_{min} parameters were calculated, and subsequently compared with the *Dixon test* critical parameter. If the calculated Q_{max} and Q_{min} values were higher than the critical value of the *Dixon test*, then the suspected result was the outlying value. Information concerning the detection of outlying values had a strictly informative character, because the programme used a statistical technique limiting the effect of extreme results using the resistant statistical method – Algorithm A [6].

For the purpose of evaluation of test results obtained by the participating laboratories, the *z-score* index was used in the case of 12 parameters. In the case of the definition of the Fraass Breaking Point and the Softening Point R&B after RTFOT hardening, the results were evaluated according to *z'-score*, due to the fact that standard uncertainty of the value attributed to these parameters was greater than $0.3\ s^*$ [6].

The values of *z-score* and *z'-score* were determined using the resistant standard deviation s^* estimated on the basis of results sent by participants. In the case of determination of elastic recovery at 25°C, the spread between the results from different participants was very small (seven laboratories achieved identical results of 85%), and the estimated value of resistant standard deviation s^* was twice lower than the value standard deviation of reproducibility of the PN-EN 13398:2012 method. In view of the above, standard deviation *s* was applied for the purpose of evaluation of this parameter, and calculated on the basis of results sent by participants, following the rejection of outlying value detected via the *Dixon test* [6].

Table 6. Comparative test results of visual evaluation of dispergation of polymer – Sample B – polymer modified bitumen PMB 45/80-55

Code of Participant	Microscopic Image	Microstructure code as per EN 13632:2010
1		*B/H/S/r*
10		*B/I/S/o*
12		*B/H/S/r*
13		*B/H/S/r*
15		*B/H/S/r*
23		*B/H/S/o*

Summary and Conclusions

The project *Testing of Proficiency through Interlaboratory Comparisons* organised by ORLEN Asfalt included the participation of 24 laboratories with major experience and certification in the scope of tests which were undertaken in this project.

The project covered standard and non-standard tests of the properties of bituminous binders – paving grade bitumen 50/70 and polymer modified bitumen PMB 45/80-55. Bitumens designated for testing were extracted from manufacturing installations belonging to ORLEN Asfalt.

Paving grade bitumen (sample A) was subject to comparative tests in terms of 8 parameters, while polymer modified bitumen (sample B) was subject to comparison of 9 parameters.

The results of the described project have shown that the weakest results were achieved for the parameter of penetration at 25°C (as many as 5 cases of questionable or unsatisfactory results), especially for paving grade bitumen. On the other hand, the most striking results were achieved for Fraass Breaking Point, where for paving grade bitumen, results were obtained in the range of –12 to –22°C, and for polymer modified bitumen between –13.7 and –25°C. In view of the fact that in most countries, breaking point represents a control test on road construction projects, this may be a cause for concern.

Attention should also be paid to the results of the complex modulus of stiffness G* and the phase angle δ for 50/70 bitumen and PMB 45/80-55. In view of relatively short experience with DSR tests and differences in testing equipment used, these results can be considered as very good.

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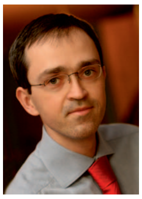
[4] *Proficiency testing by interlaboratory comparisons* complementation to report in *Lepiszcza asfaltowe No. OA/2012*, results of force ductility tests. Energy value at 5°C as per PN-EN 13589:2011 and PN-EN 13703:2009. Płock 17.01.2013.

[5] *Proficiency testing by interlaboratory comparisons* complementation to report in *Lepiszcza asfaltowe No. OA/2012*, visual assessment of polymer dispergation in polymer-modified bitumen (sample B) as per PN-EN 13632:2010. Płock 17.01.2013.

[6] *Proficiency testing by interlaboratory comparisons*. Report „Lepiszcza asfaltowe No. OA/2012", Płock 27.11.2012.



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