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Thermal oxidation stability of lubricating greases – can a rapid small-scale method replace the classical method?

Stabilność termooksydacyjna smarów plastycznych – czy szybki test w małej skali może zastąpić metodę klasyczną?

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ABSTRACT: The article presents the results of tests of the oxidation resistance of 26 samples of selected lubricating greases available on the market. Various types of lubricant samples (according to the type of thickener), produced with oils of different chemical nature and viscosity, were tested. The basic parameters of the greases were determined: the worked penetration and the dropping point. Two different test methods were used to determine the thermal oxidation stability: the classical oxidation method according to PN-C-04143 and the rapid small-scale test method according to ASTM D 8206. The method of determining the correlation between these methods was presented. A correlation was found between the two methods of testing the resistance to oxidation, which can be roughly described using the exponential dependence. For the quick method, better compliance with the classical method was obtained at 140°C than at 160°C, which is confirmed by the determination coefficients determined by the ranking method. Based on the results of the quick method, using the determined exponential dependency, the results of the classical method can be estimated. However, the determined correlation between the PN-C-04143 and ASTM D 8206 methods is insufficient to use these methods interchangeably.

Key words: thermal oxidation stability, lubricating grease, correlation, resistance to oxidation.

STRESZCZENIE: W artykule przedstawiono wyniki badań odporności na utlenianie 26 próbek wybranych smarów plastycznych dostępnych na rynku. Przebadano różne rodzaje próbek smarów (w zależności od rodzaju zagęszczacza) wyprodukowanych na bazie olejów o różnym charakterze chemicznym i o różnej lepkości. Określono podstawowe parametry smarów: penetrację po ugniataniu i temperaturę kroplenia. Do wyznaczenia stabilności termooksydacyjnej zastosowano dwie różne metody badawcze: klasyczną metodę utleniania według PN-C-04143 oraz szybką metodę badań w małej skali według ASTM D 8206. Przedstawiono metodę określania korelacji pomiędzy tymi metodami. Stwierdzono korelację między dwiema metodami badania odporności na utlenianie, którą w przybliżeniu można opisać za pomocą zależności wykładniczej. W przypadku metody szybkiej lepszą zgodność z metodą klasyczną uzyskano w 140°C niż w 160°C, co potwierdzają współczynniki determinacji wyznaczone metodą rankingową. Na podstawie wyników metody szybkiej, wykorzystując wyznaczoną zależność wykładniczą, można oszacować wyniki metody klasycznej. Wyznaczona korelacja pomiędzy metodami PN-C-04143 i ASTM D 8206 jest jednak niewystarczająca, aby stosować te metody zamiennie.

Słowa kluczowe: stabilność termooksydacyjna, smar plastyczny, korelacja, odporność na utlenianie.

Introduction

Under operating conditions lubricating greases are subject to the action of a number of factors, causing their damage – shear stresses, pressure, loads, varying operating conditions – in particular temperature changes. The grease degradation may be divided into:

- physical degradation, comprising all physical changes of the grease during its use, being an irreversible process related to permanent changes in the grease structure;
- and chemical degradation, comprising all chemical reactions proceeding inside the grease, that is the base oil oxidation, thickener oxidation, and depletion of additives, which is more sensitive to the operating temperature (Rezasoltani and Khonsari, 2016).

The oxidation is the prevailing process of ageing, affecting directly the period of lubricant use. The choice of appropriate improvers is one of methods to prevent the oxidation, both of base oils and lubricants. The thermal oxidation stability of lubricating greases may be modified by the introduction

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of appropriate antioxidants, the selection of which depends on the thickener type of lubricating grease and on the operating temperature of the grease (Trzaska et al., 2016, 2017; Krasodomski et al., 2018; Skibińska and Żółty, 2018).

The task of oxidation inhibitors (antioxidants) consists in slowing down the process of base oil oxidation, through decomposition of hydroperoxides, formed in a reaction of oxygen with hydrocarbons, or of free peroxide radicals. The lubricating grease during the operation is in contact with oxygen from the air, while a high temperature and catalytic properties of the friction surface metal create very good conditions for the base oil oxidation. Complex reactions of autocatalytic oxidation proceed inside it, resulting in the origination of new substances, such as peroxides, alcohols, aldehydes, ketones, water, and acids. The oxidation products increase the oil viscosity, and acid pollutants originate, featuring corrosive properties, as well as deposits of resin, sealing-wax, and polymer types (Khamidullina et al., 2016).

To test the oxidation resistance of greases, a method of classical oxidation in a pressurised vessel is applied, under specified temperature conditions during a preset time, and the value of the oxygen pressure decline is given as the result of the test. Standardised test methods for the assessment of the thermal oxidation stability of greases include: BS 2000-142, FTM 791.3453, IP 142, ASTM D942, DIN 51808, and PN-C-04143. A new standardised method for greases appeared for the first time in 2018, the so-called Rapid Small-Scale Oxidation Test, in which the time to reach an induction period (oxygen pressure decline by 10%) is given as the result – ASTM D 8206.

In the article by Edinger (Edinger, 2016) the presented results of oxidation resistance tests for 11 grease samples, performed by means of a RapidOxy instrument (Rapid Small Scale Oxidation Test, RSSOT) at a temperature of 160°C according to ASTM D 525 and by a classical method for greases ASTM D 942 (at 99°C, 100 and 400 h test) were presented. The comparison of obtained results allowed the author to state that there was a good correlation between these testing methods. Nolan, based on the test results presented in the paper (Nolan and Savin, 2016) confirmed the existence of a correlation between the RSSOT method and the traditional grease oxidation method ASTM D 942. The Anton Paar company, the manufacturer of the RapidOxy 100 instrument dedicated to rapid grease oxidation, specifies in their materials (Rapidoxy Report) that there is a good linear correlation between the results of oxidation resistance by the classical method at 99°C during 100 and 400 hours and the rapid one at a temperature of 160°C.

The tests of grease oxidation resistance, carried out at the INiG – PIB (Skibińska et al., 2018) for produced samples of various types of greases with antioxidant additives, did not allow to state a linear relation between the classical oxidation method and the rapid small-scale method.

Materials

A wide range of greases available on the market, belonging to various types (by thickener type), based on oils of a diverse chemical nature and various viscosities, were used in this study. Table 1 presents the information on the samples.

Testing methods

The ASTM D 8206-18 method is used to determine the oxidation stability of greases, on a small scale under accelerated conditions (Rapid Small – Scale Oxidation, RSSOT) via the measurement of the induction period. The method consists in placing a grease sample in a glass cell at an ambient temperature (Fig. 1), placing the cell with grease in the reaction chamber of the instrument (Fig. 2), and filling the chamber with oxygen up to a pressure of 700 ±5 kPa. The reaction chamber is heated up to a preset temperature (140 or 160°C).

The pressure in the chamber increases with heating and then declines with the consumption of oxygen for sample oxidation. The pressure in the cell is recorded in 1s intervals until achieving a breakdown point. The time passing from the start of determination till the breakdown point is the induction period at the test temperature.

Fig. 1. Glass cell (photo INiG – PIB)
Rys. 1. Naczynko szklane (fot. INiG – PIB)

The oxidation resistance testing method according to PN-C-04143:1956 (similar to IP 142, ASTM D 942, BS 200-142, FTM791.3453), is a classical grease oxidation method in a pressurised cell, the only method widely used for more than half a century, referred to in requirements for greases, in particular the military ones. This method consists in oxidising the tested sample under precisely determined pressure and temperature conditions. The tests are carried out in a pressurised vessel, the so-called bomb. Grease is placed in each of five cells,

Table 1. Characteristic of grease samples, based on the information and technical sheets of grease manufacturers/suppliers
Tabela 1. Charakterystyka próbek smarów, na podstawie informacji i kart technicznych producentów/dostawców smarów

Sample number	Application temperature range, from/to [°C]	Base oil	Kinematic viscosity [mm²/s]		Thickener	Worked penetration [mm/10] NLGI class	Dropping point [°C]
			at 40°C	at 100°C			
1	–30/120	mineral	90.0	10.50	lithium soaps	220–250	>190
2	–20	mineral	115.0	12.20	poliurea	NLGI 2	260
3	–30/60	rapeseed oil and PAO	38.0	8.00	calcium soaps	405–445	>120
4	–51/179	PFPE	60.0	9.00	PTFE	NLGI 2	no
5	–30/250	synthetic	–	–	PTFE	NLGI 2	no
6	–35/160	mineral	–	–	lithium complex	NLGI 2	>240
7	–30/60	rapeseed oil and PAO	38.0	8.00	calcium soaps	385–400	>120
8	–	ether	103.0	13.00	urea	NLGI 2	>230
9	–50/120	synthetic ester	11.0	3.30	lithium soaps	NLGI 1.5	180
10	–40/180	ester oil	100.0	11.00	poliurea	250–280	>250
11	–28/200	mineral	133.0	13.00	calcium sulfonate	NLGI 2	>280
12	–50/150	polyoester diester	26.0	5.10	lithium soaps	250	190
13	–50/120	PAO	30.0	5.90	barium complex	NLGI 2	>240
14	–20/140	mineral	115.0	–	lithium complex	NLGI 3	230
15	–40/150	PAO	46.7	7.75	diurea	235	>270
16	–40/180	polyoesters	30.5	5.40	diurea	NLGI 2	>250
17	–50/80	mineral, naphtenic	9.1	2.30	aluminium complex	NLGI 2	>220
18	–30/120	white petrolatum oil	18.0	3.70	aluminium complex	NLGI 2	>220
19	–20/200	mineral	500.0	–	bentonite with copper	310–340	>280
20	100/200	mineral	591.0	34.50	bentonite	NLGI 2	>280
21	–30/180	mineral	460.0	–	calcium sulfonate	302	>300
22	–40/150	mineral and PAO	80.0	–	lithium complex	356	>240
23	–50/120	ester and mineral	15.5	3.50	lithium soaps	NLGI 2	190
24	–40/120	biodegradable synthetic ester	120.0	–	calcium-lithium soaps	NLGI 1	>120
25	–25/120	mineral	68.0	–	lithium soaps	NLGI 1	>180
26	–50/180	PAO	46.7	7.80	lithium complex	285	290

Fig. 2. RapidOxy 100 instrument for the oxidation stability determination (photo INiG – PIB)
Rys. 2. Aparat RapidOxy 100 do oznaczania stabilności oksydacyjnej (fot. INiG – PIB)

and spread with a spatula, to obtain a uniform layer of grease with a smooth surface. Cells with the grease prepared in this way are placed on a stand (Fig. 3). The stand is placed in the pressurised vessel (Fig. 4). The bomb is then filled with oxygen and placed in a thermostat heated up to a temperature of 100 ±0.1°C. From this moment the oxygen pressure in the bomb is recorded every 2 hours until reaching a constant maximum pressure, maintaining in the bomb for a period not shorter than 2 hours. This moment is considered the measurement start point. From the moment of reaching the maximum pressure a reading is taken not less frequently than every 18 hours. The test is carried out over a period of 100 hours.

Fig. 3. Set of cells on the stand (photo INiG – PIB)
Rys. 3. Zestaw naczynek na podstawce (fot. INIG – PIB)

Fig. 4. Instrument for oxidation resistance determination acc. to PN-C-04143 (photo INiG – PIB)
Rys. 4. Aparat do oznaczania odporności na utlenianie według PN-C-04143 (fot. INIG – PIB)

The oxidation resistance of grease samples was tested applying standardised testing methods acc. to the following conditions:

- Classical oxidation method acc. to PN-C-04143:
  - temperature: 100 ±0.5°C,
  - oxygen pressure at 20 ±5°C: 625 ±5 kPa,
  - amount of grease sample in the glass cell: 4.00 ±0.01 g,
  - number of cells: 5,
  - end of testing: after 100 h of oxidation;
- Rapid small-scale method acc. to ASTM D 8206:
  - temperature: 140 ±0.5°C and 160 ±0.5°C,
  - oxygen pressure at 20 ±5°C: 700 ±5 kPa,
  - amount of grease sample in the glass cell: 4.00 ±0.01 g,
  - number of cells: 1,
  - end of testing: pressure decline by 10% (from the maximum).

Tests were carried out twice. An arithmetic mean of 2 measurements was given as the result. Results of the tests are compared in Table 2 and in Figs. 5 and 6.

Table 2. Results of oxidation resistance tests for grease samples
Tabela 2. Wyniki badań odporności na utlenianie próbek smarów

Sample number	PN-C-04143 [MPa]	ASTM D 8206 [min]	
	100°C	140°C	160°C
1	0.007	1270	357
2	0.006	1489	647
3	0.717	100	39
4	0.015	2698	1209
5	0.010	2152	1506
6	0.008	607	160
7	0.704	77	33
8	0.020	1805	1005
9	0.019	1797	657
10	0.019	762	296
11	0.019	1056	315
12	0.011	2379	1308
13	0.004	1938	857
14	0.016	1786	623
15	0.030	1321	777
16	0.021	1296	639
17	0.023	619	175
18	0.015	1883	309
19	0.215	243	176
20	0.252	213	141
21	0.088	884	431
22	0.038	634	248
23	0.004	2431	1193
24	0.172	215	121
25	0.023	1431	363
26	0.020	1795	827

Determination of correlation between the methods

Based on the results of the grease oxidation resistance testing, an attempt was made to determine correlations between the results obtained by the methods: ASTM D 8206 and PN-C-04143, and hence between the methods themselves.

To this end, correlograms were drawn – point graphs of $\{(x_i, y_i)\}$ pairs, where x_i and y_i are the results of sample "i" testing by two methods. If a set of dispersed points is observed,

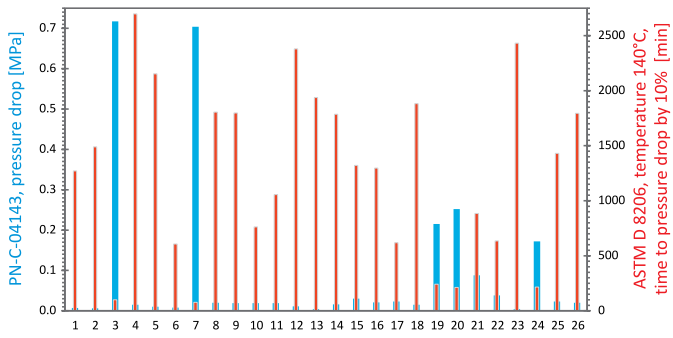

Fig. 5. Comparison of oxidation resistance results acc. to PN-C-04143 (100°C) and ASTM D 8206 (140°C)

Rys. 5. Porównanie wyników odporności na utlenianie metodami PN-C-04143 (100°C) i ASTM D 8206 (140°C)

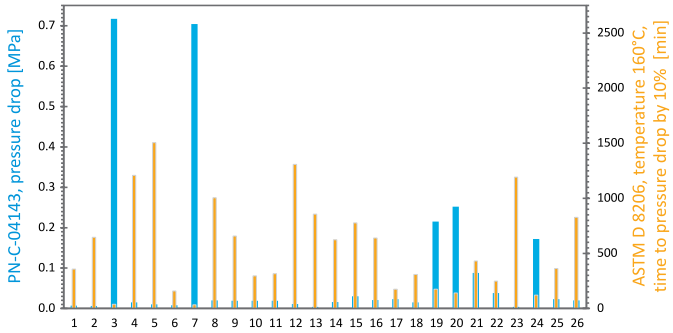

Fig. 6. Comparison of oxidation resistance results acc. to PN-C-04143 (100°C) and ASTM D 8206 (160°C)

Rys. 6. Porównanie wyników odporności na utlenianie metodami PN-C-04143 (100°C) i ASTM D 8206 (160°C)

which does not remind one of a shape of graph known from a functional relationship, it is considered that there is no relationship between features X and Y. The situation is opposite if the observed set of points seems to arrange themselves in a characteristic way – the existence of a relationship between variables is considered. This feature is confirmed next by calculations of the coefficient of determination, R^2.

The coefficient of determination R^2 is a measure of the strength of the relationship between the variables (it specifies what part of one feature variability is explained by the other feature). The coefficient of determination is defined as:

$$R^2 = \frac{\sum_{i=1}^{n}(\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \geq 0$$

y_i – results of the i-th sample measurement (by the classical method),

$\hat{y}_i$ – theoretical value of the i-th sample measurement (by the classical method) calculated based on the model (curve fitting),

$\bar{y}$ – arithmetic mean of all samples results (obtained by the classical method).

The coefficient of determination R^2 differs from the frequently used Pearson r^2 coefficient. The Pearson r^2 coefficient is used to study a linear relationship between variables. Because of the strongly non-linear relationship presented in Figure 7, the application of the Pearson coefficient would be unjustified in this case.

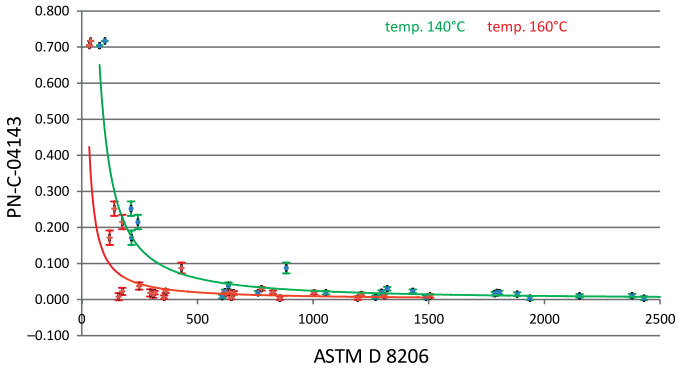

Fig. 7. Correlogram between results of oxidation resistance for grease samples obtained by the classical method at 100°C and by the rapid method at 140°C and 160°C with marked error bars resulting from the PN-C-04143 method repeatability

Rys. 7. Korelogram pomiędzy wynikami odporności na utlenianie próbek smarów uzyskanymi metodą klasyczną w 100°C i metodą przyśpieszoną w 140°C i 160°C z naniesionymi słupkami błędów wynikającymi z powtarzalności metody PN-C-04143

The degree of fitting, depending on the value of the coefficient of determination R^2, may be described as:

- 0.0–0.5 – unsatisfactory fitting;
- 0.5–0.6 – poor fitting;
- 0.6–0.8 – satisfactory fitting;
- 0.8–0.9 – good fitting;
- 0.9–1.0 – very good fitting.

Figure 7 presents a correlogram between the results of the oxidation resistance of grease samples obtained by the classical method at 100°C and the rapid method at 140°C and 160°C. The obtained set of points is arranged for results of both rapid methods separately, on a curve being approximately an exponential curve. This is the curve with the best fitting among the studied relationships: exponential, linear, logarithmic, and multinomial ones. The obtained values of the coefficient of fitting are as follows:

- $R^2 = 0.788$ for the temperature of 140°C;
- $R^2 = 0.614$ for the temperature of 160°C;

which, according to the above range of fitting, may be considered the satisfactory fitting. Nevertheless, noticeable deviations of numerous measurement points from curves (Fig. 7):

- $y = 172.25x^{-1.284}$ for the temperature of 140°C;
- $y = 20.556x^{-1.115}$ for the temperature of 160°C;

substantially exceeding the repeatability of measurement results:

- 15 measurements out of 26 made (which is ~58%) for the temperature of 140°C;

- 18 measurements out of 26 made (which is ~69%) for the temperature of 160°C;

show a need for further analysis of the fitting. The above deviations are presented in Tables 3 and 4.

Also the so-called ranking method was used to analyse more precisely the correlations between the results of oxidation resistance obtained by the ASTM D 8206 (at 140°C and 160°C) and the PN-C-04143 (100°C) methods. The method of fractional ranking was applied in the case of the existence of results with equal ranking values. Such a method of tied ranks assigning is widely used in statistics, e.g. to calculate the Spearman's rank correlation coefficient. To this end:

- the obtained 3 groups of test results for grease samples were ordered, from the smallest to the highest;
- a rank was assigned to each sample, i.e. their place in ranking: 1 for the grease most resistant to oxidation, 26 to the least resistant;
- if the test results were identical for two samples (e.g. in place 1 the result is 0.004 and in place 2 also 0.004), both samples were assigned an average place, that is 1.5.

These data are specified in Table 5. Next, pairs of ranking places for results obtained in these three tests were specified in graphs (4–6) and the coefficient of determination was determined for them.

In the case of a linear relationship, the coefficient of determination R^2 is equal to the square Pearson correlation coefficient r^2, that is for $R^2 = r^2 = 1$ we obtain exactly a linear relationship.

Table 3. Conversion of test results obtained by the ASTM D 8206 rapid method at a temperature of 140°C into results of the classical method, acc. to the formula $y = 172.25x^{-1.284}$

Tabela 3. Konwersja wyników badań uzyskanych metodą przyśpieszoną ASTM D 8206 w temperaturze 140°C na wyniki metody klasycznej, według wzoru $y = 172{,}25x^{-1{,}284}$

Sample number	Method 1	Conversion results method 1 to 2	Method 2	Repeatability (r) according to PN-C-04143 [MPa]	Range of results [MPa]	
	ASTM D 8206 average (X) from two measurements [min]		PN-C-04143 average (X) from two measurements [MPa]		X–0.5r	X+0.5r
1	1270	0.018	0.007	0.01	0.002	0.012
2	1489	0.015	0.006	0.01	0.001	0.011
3	100	0.466	0.717	–	–	–
4	2698	0.007	0.015	0.01	0.010	0.020
5	2152	0.009	0.010	0.01	0.005	0.015
6	607	0.046	0.008	0.01	0.003	0.013
7	77	0.651	0.704	–	–	–
8	1805	0.011	0.020	0.01	0.015	0.025
9	1797	0.011	0.019	0.01	0.014	0.024
10	762	0.034	0.019	0.01	0.014	0.024
11	1056	0.023	0.019	0.01	0.014	0.024
12	2379	0.008	0.011	0.01	0.006	0.016
13	1938	0.010	0.004	0.01	–0.001	0.009
14	1786	0.012	0.016	0.01	0.011	0.021
15	1321	0.017	0.030	0.01	0.025	0.035
16	1296	0.017	0.021	0.01	0.016	0.026
17	619	0.045	0.023	0.01	0.018	0.028
18	1883	0.011	0.015	0.01	0.010	0.020
19	243	0.149	0.215	0.04	0.195	0.235
20	213	0.176	0.252	0.04	0.232	0.272
21	884	0.028	0.088	0.03	0.073	0.103
22	634	0.043	0.038	0.02	0.028	0.048
23	2431	0.008	0.004	0.01	–0.001	0.009
24	215	0.174	0.172	0.04	0.152	0.192
25	1431	0.015	0.023	0.01	0.018	0.028
26	1795	0.011	0.020	0.01	0.015	0.025

Table 4. Conversion of test results obtained by the ASTM D 8206 rapid method at a temperature of 160°C into results of the classical method, acc. to the formula $y = 20.556x^{-1.115}$

Tabela 4. Konwersja wyników badań uzyskanych metodą przyśpieszoną ASTM D 8206 w temperaturze 160°C na wyniki metody klasycznej, według wzoru $y = 20.556x^{-1.115}$

Sample number	Method 1	Conversion results method 1 to 2	Method 2	Repeatability (*r*) according to PN-C-04143 [MPa]	Range of results [MPa]	
	ASTM D 8206 average (X) from two measurements [min]		PN-C-04143 average (X) from two measurements [MPa]		X–0.5*r*	X+0.5*r*
1	357	0.029	0.007	0.01	0.002	0.012
2	647	0.015	0.006	0.01	0.001	0.011
3	39	0.351	0.717	–	–	–
4	1209	0.008	0.015	0.01	0.010	0.020
5	1506	0.006	0.010	0.01	0.005	0.015
6	160	0.072	0.008	0.02	–0.002	0.018
7	33	0.424	0.704	–	–	–
8	1005	0.009	0.020	0.01	0.015	0.025
9	657	0.015	0.019	0.01	0.014	0.024
10	296	0.036	0.019	0.02	0.009	0.029
11	315	0.034	0.019	0.01	0.014	0.024
12	1308	0.007	0.011	0.01	0.006	0.016
13	857	0.011	0.004	0.01	–0.001	0.009
14	623	0.016	0.016	0.01	0.011	0.021
15	777	0.012	0.030	0.01	0.025	0.035
16	639	0.015	0.021	0.01	0.016	0.026
17	175	0.065	0.023	0.02	0.013	0.033
18	309	0.034	0.015	0.02	0.005	0.025
19	176	0.065	0.215	0.04	0.195	0.235
20	141	0.083	0.252	0.04	0.232	0.272
21	431	0.024	0.088	0.03	0.073	0.103
22	248	0.044	0.038	0.02	0.028	0.048
23	1193	0.008	0.004	0.01	–0.001	0.009
24	121	0.098	0.172	0.04	0.152	0.192
25	363	0.029	0.023	0.01	0.018	0.028
26	827	0.011	0.020	0.01	0.015	0.025

Table 5. Ranking places of results for each of grease samples in three tests at different temperatures

Tabela 5. Miejsca w rankingu wyników dla każdej z próbek smarów w trzech badaniach w różnych temperaturach

Sample number	PN-C-04143 temperature 100°C		ASTM D 8206 temperature 140°C		ASTM D 8206 temperature 160°C	
	result [MPa]	rank	result [min]	rank	result [min]	rank
1	0.007	4	1270	15	357	15
2	0.006	3	1489	11	647	10
3	0.717	26	100	25	39	25
4	0.015	8.5	2698	1	1209	3
5	0.010	6	2152	4	1506	1
6	0.008	5	607	21	160	22
7	0.704	25	77	26	33	26

cont. Table 5 / cd. Tabela 5

Sample number	PN-C-04143 temperature 100°C		ASTM D 8206 temperature 140°C		ASTM D 8206 temperature 160°C	
	result [MPa]	rank	result [min]	rank	result [min]	rank
8	0.020	14.5	1805	7	1005	5
9	0.019	12	1797	8	657	9
10	0.019	12	762	18	296	18
11	0.019	12	1056	16	315	16
12	0.011	7	2379	3	1308	2
13	0.004	1.5	1938	5	857	6
14	0.016	10	1786	10	623	12
15	0.030	19	1321	13	777	8
16	0.021	16	1296	14	639	11
17	0.023	17.5	619	20	175	21
18	0.015	8.5	1883	6	309	17
19	0.215	23	243	22	176	20
20	0.252	24	213	24	141	23
21	0.088	21	884	17	431	13
22	0.038	20	634	19	248	19
23	0.004	1.5	2431	2	1193	4
24	0.172	22	215	23	121	24
25	0.023	17.5	1431	12	363	24
26	0.020	14.5	1795	9	827	7

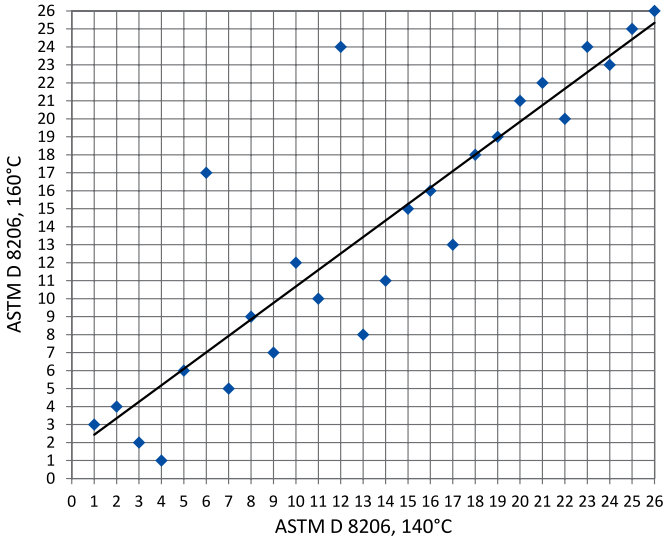

Fig. 8. Pairs of ranking places in tests by the ASTM D 8206 method

Rys. 8. Pary miejsc rankingowych w badaniach metodą ASTM D 8206

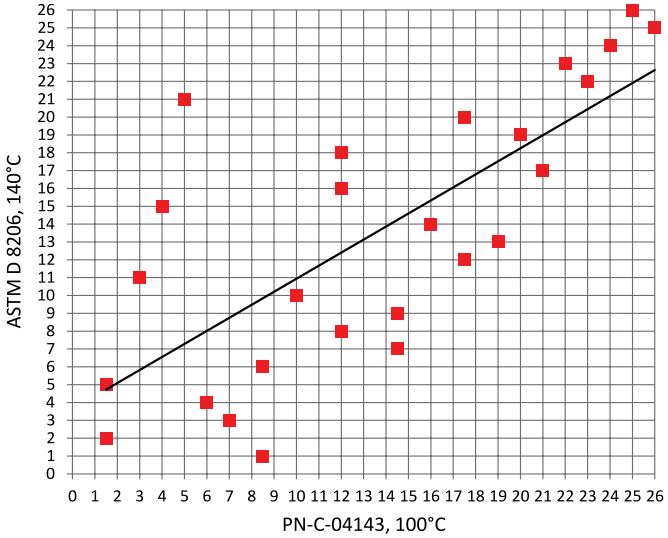

Fig. 9. Pairs of ranking places in tests by the method ASTM D 8206 at 140°C and PN-C-04143 at 100°C

Rys. 9. Pary miejsc rankingowych w badaniach metodą ASTM D 8206 w 140°C i PN-C-04143 w 100°C

Based on Fig. 8 it is possible to state that a linear arrangement of points proves the existence of a consistency of results obtained acc. to ASTM D 8206 at both temperatures. The analysis of data presented in Figs. 9 and 10 shows that the obtained sets of points do not remind one of a shape of a linear relationship graph, expected in the case of both methods consistency. This leads to a conclusion that there is no strong relationship between the results of oxidation resistance of 26 grease samples carried out at a temperature of 100°C acc. to PN-C-04143 and at temperatures 140°C and 160°C acc. to ASTM D 8206.

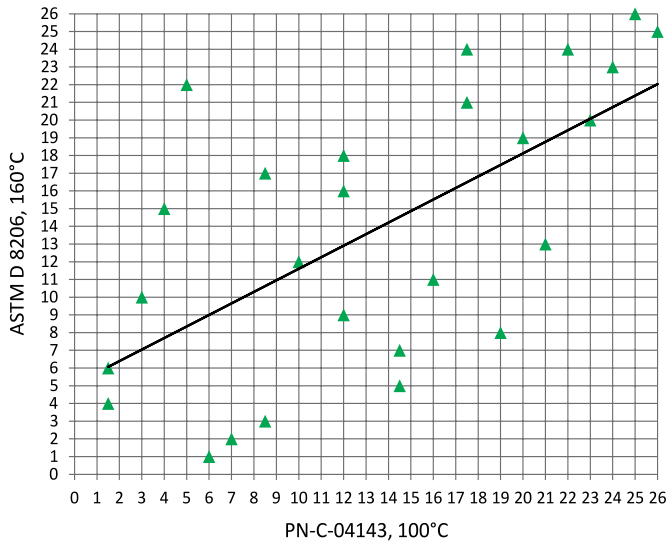

Fig. 10. Pairs of ranking places in tests by the method ASTM D 8206 at 160°C and PN-C-04143 at 100°C

Rys. 10. Pary miejsc rankingowych w badaniach metodą ASTM D 8206 w 160°C i PN-C-04143 w 100°C

Based on the determined coefficients of determination, it is possible to state that:

- there is a relationship between the results of grease oxidation resistance obtained by the rapid method acc. to ASTM D 8206 at two temperatures 140°C and 160°C – the coefficient of determination R^2 of approx. 0.78 means a satisfactory consistency of both methods;
- there is no strong relationship between the results of grease oxidation resistance obtained by the rapid method acc. to ASTM D 8206 (test at a temperature of 140°C), and the classical method acc. to PN-C-04143 (test at a temperature of 100°C) – the coefficient of approx. 0.53 means a poor consistency of both methods;
- there is no strong relationship between the results of grease oxidation resistance obtained by the rapid method acc. to ASTM D 8206 (test at a temperature of 160°C), and the classical method acc. to PN-C-04143 (test at a temperature of 100°C) – the coefficient of approx. 0.39 means a very poor consistency of both methods.

Conclusions

Summarising the attempt to determine the correlation between methods – the classical PN-C-04143 and rapid small scale ASTM D 8206 – it was found that:

- there is a correlation between the two testing methods; it may be roughly described by an exponential relationship;
- an approximate nature of the exponential relationship is proved by the fact that many measurement points, within the method repeatability, are not situated on the exponential curve;
- for the rapid method a better consistency with the classical method (albeit still poor) is obtained at a temperature of 140°C than at 160°C, which is confirmed by coefficients of determination determined by the ranking method;
- based on the results of the rapid method, by means of the determined exponential relationship, it is possible to estimate the results of the classical method;
- however, the determined correlation between PN-C-04143 and ASTM D 8206 methods is insufficient to apply these methods interchangeably.

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