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Quantitative and qualitative characteristics of industrial oils

Operation in a continuous oil system

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Original article

Abstract

The subject of this publication is research carried out on two industrial oils which were used in a continuous oil system, ensuring lubrication of machine elements. In addition, the aspect of the physicochemical properties of the oil and the method of assessing its suitability for operational purposes are discussed. In the paper are presented quantitative and qualitative methods of analysis of industrial oils, according to applicable standards and method of their implementation. The research was carried out over a period of 5 months, so it was possible to determine what parameters of the oils changed during their use. This aspect was the basis for the conclusion on the evaluation of the suitability of the oil as a lubricant in the further operation of the machines. The selected oils that were analyzed differed in purpose, as one of them was a mineral turbine oil and the other a mineral compressor oil.

Keywords

- industrial oil
- continuous oil system
- viscosity
- impurity content in oil

Authors contributions

A – Preparation of the research project
B – Assembly of data for the research undertaken
C – Conducting of statistical analysis
D – Interpretation of results
E – Manuscript preparation
F – Literature review
G – Revising the manuscript

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Introduction

There are many classifications of industrial oils. This is due to the different industrial and utility applications. The most fundamental classification is that which includes chemical origin and method of production. Oils are divided into mineral, semi-synthetic and synthetic [1]. Mineral oils are obtained from crude oil in a distillation process. This distillate purification and enrichment technology consists mainly of three stages: refining using selective solvents, dewaxing, and hydrorefining. Synthetic oils are produced by chemical reactions, the most important of which are polymerization and esterification. Polyolefins are used to produce many synthetics, using Zigler-Natta catalysts during the polymerization reaction [1-6].

Industrial oils are divided into application groups according to the PN-EN ISO 6743:2009 standard [7]. In compressors and turbines, oils are mainly used to lubricate the working components. A good compressor oil is characterized by high resistance to oxidation and low susceptibility to sedimentation. Its viscosity is selected depending on the purpose of the compressor and its construction. It should not contain additives such as viscosifiers (increasing the viscosity index value) or depressants (responsible for the fluidity of the oil at low temperatures) due to the susceptibility of these substances to coking. The correct oil for turbines is characterized by high resistance to oxidation. It usually has low kinematic viscosity. It is important that the turbine oil has good demulsifying properties. Small amounts of water in the oil can form an emulsion, which can cause local increases in viscosity [1,5,8].

The right quality of oil is essential to ensure the correct operation of many industrial devices. Systematic performance of comprehensive analyses allows for early detection of lubrication problems, which minimizes the risk of failure and production downtime. Based on the conducted research, an attempt was made to answer the question of whether the analyzed oils are appropriately selected for the type of their operating environment and whether they are suitable for further use, based on the obtained results. For this purpose, parameters such as density, kinematic viscosity, viscosity index, acid value, water content, purity and emulsification tendency were determined over a period of 5 months according to methods specified by standards and literature. [9-16]. In addition, a formaldehyde test (Nastyukov reaction) was performed for samples of both oils and the aniline point was determined [17-18].

Materials and methods

For the purpose of the research, the tested oils were marked as Oil 1 and Oil 2 respectively. The first one (Oil 1) is a turbine oil used in the turboset, while the second one (Oil 2) is intended and used to lubricate the compressor elements. Samples of both oils were collected from the same devices over a 5-month period (from December 2023 to April 2024) and analyzed. Samples were collected once a month and analyzed on an ongoing basis. The oils were used under the following conditions: Oil 1: temperature: 54°C, pressure: 1.5 bar, Oil 2: temperature: 42–45°C, pressure: 3.5–4 bar. Samples of both oils were taken from the installations after the oil had passed through the system filters. A detailed list of apparatus and reagents used for individual determinations is provided in Appendix 1.

The density of the samples was determined using the areometric method according to the PN-EN ISO 3675:2004 standard [9]. Kinematic viscosity was tested using the Ubbelohde viscometer, with capillary type I, in accordance with PN-EN ISO 3104:2024-01 [12]. This parameter was determined at 40°C and 100°C. The results obtained from this test were used to determine the viscosity index (VI). Water content in oil samples was determined using the coulometric Karl Fischer method according to PN-EN ISO 12937:2000 [11]. The apparatus used during the research is shown in Figure 1.

Figure 1. Coulometer for the determination of water content in oil samples by the Karl Fischer method

The acid value was determined by potentiometric titration in accordance with the PN-ISO 6619:2011 standard [10]. The Purity of oil samples was tested using an automatic particle counter and by filtration and assessment of the purity class under a microscope according to PN-ISO 4406:2005 [14]. The automatic laser particulate counter used to determine oil purity is shown in Figure 2.

Figure 2. The laser particulate counter

Each sample was also tested for its ability to form a stable emulsion with water based on the procedure described in ISO-6614:1994 [19]. The aniline point was determined for selected samples based on the PN-ISO 2977:2012-03 standard [20]. In addition, a qualitative test with formaldehyde, also known as the Nastyukov reaction, was performed for both oil samples. All the analyses mentioned above make up a comprehensive set of tests to determine the properties of the working oil. Some of the determined parameters have a range of permissible values. These levels are shown in Table 1.

Table 1. Permissible parameter values

Determined parameter	Permissible value	
	Oil 1	Oil 2
Kinematic viscosity in 40°C	41.4–50,6 mm^2/s	90–110 mm^2/s
Water content	≤0.01%	≤0.01%
Acid value	≤0.5 mg KOH/g	≤1.5 mg KOH/g
Purity determined by automatic particle counter (class in accordance with PN-ISO 4406)	16/14/11	18/16/13
Purity determined by microscope method (class in accordance with PN-ISO 4406)	14/11	16/13

Results and discussion

The oil used in the machine should be frequently checked for its rheological properties. This applies not only to oils but also to other lubricants, such as greases. Information on rheology is easiest to obtain from the analysis of density, kinematic viscosity and viscosity index. The data obtained for these parameters are presented in Table 2.

Table 2. The results of density, kinematic viscosity and viscosity index

Month of oil sampling	Density [g/cm^3]		Kinematic viscosity in 40°C [mm^2/s]		Kinematic viscosity in 100°C [mm^2/s]		Viscosity Index [–]	
	Oil 1	Oil 2	Oil 1	Oil 2	Oil 1	Oil 2	Oil 1	Oil 2
December	0.852	0.883	44.87 ± 0.02	125.33 ± 0.02	7.48 ± 0.02	13.04 ± 0.02	132.14	97.06
January	0.854	0.885	44.62 ± 0.02	116.88 ± 0.02	7.39 ± 0.02	12.36 ± 0.02	129.80	95.89
February	0.856	0.885	44.62 ± 0.02	120.78 ± 0.02	7.40 ± 0.02	12.48 ± 0.02	130.18	93.74
March	0.851	0.884	44.62 ± 0.02	125.03 ± 0.02	7.39 ± 0.02	12.59 ± 0.02	129.80	91.14
April	0.859	0.884	44.66 ± 0.02	116.88 ± 0.02	7.43 ± 0.02	12.31 ± 0.02	131.16	95.20

Based on the analysis of the oil density results, no major changes were noticed in individual months. These fluctuations did not indicate excessive degradation of oils or danger to lubrication systems. The results indicate that both oils continue to be in good condition and have proper rheological properties. In this situation, with only the marked density of samples available, it is not recommended to replace the oil. The noticeable difference in density between the tested oils may result primarily from their chemical composition, which translates into the degree of their concentration. Furthermore, Oil 2 may have a higher density because it has a higher kinematic viscosity than Oil 1.

The stability that characterized the kinematic viscosity of Oil 1 proved the good quality of this lubricant and its resistance to negative impact that occurred during operation in the installation. Based on the results obtained, there was no need to partially replace the used oil with fresh oil. It can also be concluded that Oil 1 contained additives in the form of viscosifiers, allowing it to maintain a constant viscosity over time. In the case of Oil 2, changes in viscosity were observed, which confirmed its partial consumption. The results from December and March are the basis for partial oil replacement with fresh oil.

The viscosity indexes determined for Oil 1 were noticeably higher than those for Oil 2. This shows that, the viscosity of Oil 1 changes less with increasing temperature than that of Oil 2. The refining additives present in its composition may be to some extent responsible for this character of the oil. Oil 1 operates in a system with a higher temperature, unlike the installation with Oil 2. Therefore, its fluidity must be maintained at a better level. Consequently, the values of the determined viscosity indexes must be higher for Oil 1. Nevertheless, the rheological stability of both oils at a given temperature is correct, and confirms the validity of the results of the remaining parameters.

The results of the Karl Fischer water content determination in the oil samples are included in Table 3.

Table 3. Results of Karl Fischer water content determination in oil samples

Month of oil sampling	Water content [%]	
	Oil 1	**Oil 2**
February	0.0060 ± 0.0001	0.0030 ± 0.0001
March	0.0094 ± 0.0001	0.0030 ± 0.0001
April	0.0082 ± 0.0001	0.0036 ± 0.0001

This test was performed over a period of three months, as instrument failure prevented it from being carried out in December and January. The highest water content in Oil 1 was determined in March (0.0094%). Compared to the result in February (0.0060%), there was a significant increase. In the last month of the study, the parameter determined reached a value of 0.0082%. In the case of Oil 2, the water content was 0.003% in the first two months, but increased slightly to 0.0036% in April. The determined water content levels in the tested oils were low. In Oil 1, the water content was slightly higher because the temperature in the system in which it was used was slightly higher than in the system in which Oil 2 was used. The temperature conditions may have favoured the condensation of water from the air. Oil 1 was also characterised by a low and constant water content during the tests, which may be indicative of a good tightness of the installation. Water did not enter through sensitive points in the system, e.g. pipe joints, seals. The results did not indicate the need for additional cleaning of the oil or the need to remove excess water from the oil.

Table 4 presents the results obtained when determining the acid value in oil samples.

Table 4. Results of the acid value determination in the oil samples

Month of oil sampling	Acid value [mg KOH/g]	
	Oil 1	**Oil 2**
December	0.071	0.090
January	0.082	<0.010
February	<0.010	<0.010
March	0.121	0.203
April	0.035	<0.010

Some of the analytical results presented in Table 4 were marked as <0.01 mg KOH/g oil. This is due to the fact that the lower value of the measuring range of the titrator was 0.01 mg KOH/g. There was no need to determine an exact result because this value is certainly lower than the permissible values for both oils. Oil 1 and Oil 2 were characterised by low acid values throughout the analysis period. The permissible values were not exceeded in any month. Purification of the oils took place in the months when the highest acid numbers were recorded. It should be noted that this approach was effective and very conservative, as the processes were carried out when the determined acid number value was not even 25% of the limit value. The results obtained for both oils were very good and indicated a low state of overwork.

The purity of the oils tested was determined in accordance with the classes described in PN-ISO 4406. Table 5 presents the results of the purity (class) of samples of both tested oils.

Table 5. Results of purity class (ISO 4406) of tested oils

Month of oil sampling	Results of purity class from automatic particulate counter method		Results of purity from microscope method	
	Oil 1	Oil 2	Oil 1	Oil 2
December	17/15/12	19/16/12	15/12	17/14
January	17/15/12	19/17/12	15/12	17/14
February	17/15/12	21/19/14	15/12	18/15
March	18/17/12	19/16/13	15/12	18/15
April	18/16/12	20/17/12	15/12	14/14

Comparing the results from the microscopic method in Table 5, it can be seen that the same level of solid contaminants was maintained in Oil 1 throughout the tests. The first number in the code 15/12 indicates that in 1 ml of the sample the amount of solid impurities greater than or equal to 5 μm is in the range 160–320, while the second indicates that the amount of solid impurities greater than or equal to 15 μm is in the range 20–40. In each month in which samples were taken and analysed, the purity class was exceeded by 1 degree. In the case of the results obtained for Oil 2, it was noted that the class values changed during the period of analysis. In December and January, the class was 17/14. The number 17 in the code means that the number of particles ≥5 μm present in 1 ml of the sample is in the range 640–1300. The number of impurities relating to particles ≥15 μm (number 14 in the code) is in the range 80–160. In the following two months, a doubling of the number of impurities in the oil with regard to both sizes, thus increasing the grade by 1 degree, was observed. In the case of this oil, the assumed standard was also exceeded throughout the test period. When the contamination level reached 18/15, the working oil was filtered and refined in February and March. This resulted in a grade drop of 1 grade.

Figure 3 shows the appearance of the filters on microscope images taken for Oil 1 and Oil 2 samples from February.

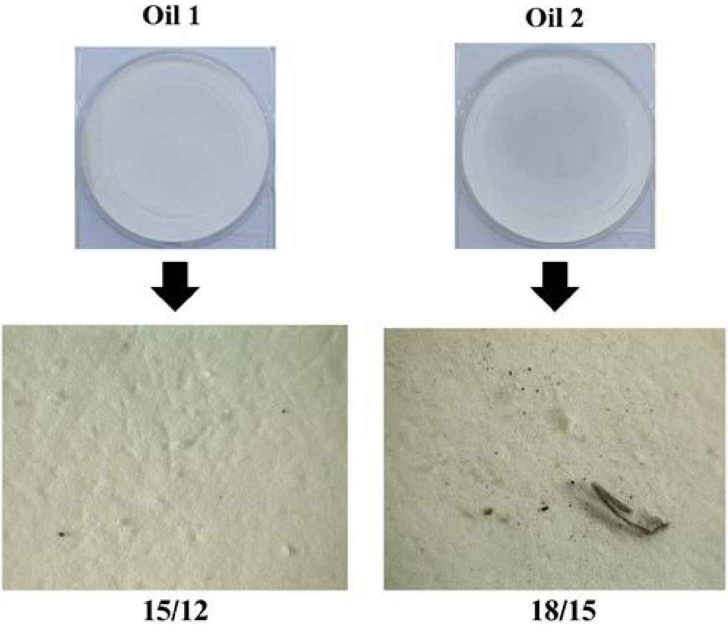

Figure 3. The appearance of the filters after filtrating oil samples from February

FThe results for Oil 1 from the automatic particle counter method in the first three months were the same. A grade of 17/15/12 means that in 1 ml of oil sample, particles with a size ≥4 µm ranged between 640 and 1300, the number of particulate contaminants with a size ≥6 µm ranged between 160 and 320, while ≥14 µm ranged between 20 and 40. In March, the analysis showed that the number of contaminants with the smallest size (4 µm to 6 µm) and medium size (6 µm to 14 µm) approximately doubled. In April, the cleanliness class indicated a decrease of 1 degree in the number of medium-sized particles, while other levels maintained the result from March. The result in March, however, indicated that the oil needed to be cleaned, as the amount of small contaminants had increased dramatically. The automatic counter gives the exact amount of contamination, so despite the relatively good result under the microscope, it was decided to make a recommendation to refresh the oil. The April class proved that this treatment had succeeded in removing some of the contaminants (sized from 6 µm to 14 µm). In the case of Oil 2, the analyses showed that there were large changes in purity over a period of five months. In December and January, the content of individual particles was practically at the same level. In February, a sharp deterioration in oil quality was registered. The content of contaminants of all sizes increased dramatically. In March, a significantly lower level of particles was achieved compared to the February sample. In April, the cleanliness class increased by one number for each type of contaminant. The class defining the content of the smallest contaminant particles was exceeded in each month.

Purity analyses of the oils tested provided some valuable information about their operating environment. It was observed that there was a problem in maintaining acceptable levels of solid contaminants in both oils. However, in the case of Oil 1, the amount of contaminants, despite exceeding the standard, did not reach a level that would be dangerous to the components of the lubrication system or the machine. Only in March and April did the levels require more urgent filtering of the oil in the system. Systematic filtering carried out with greater frequency would certainly have brought it up to an acceptable cleanliness class. In Oil 2, greater fluctuations in purity classes were observed when analyses were performed over a period of 5 months. In its case, purity classes were also exceeded throughout this period. Class 18/15 required filtering to be carried out. Maintaining the 17/14 value is still relatively safe, as the amount of contamination does not drastically affect the efficiency of the lubrication process and the wear of plant parts, e.g. filters. Nevertheless, further efforts should be made to achieve an acceptable level. The results of the automatic particle counter method followed the trend of the microscope-based results. It was observed that one of the problems of maintaining the desired purity class was the dynamic increase in content, especially of the smallest impurities, when the purity of the oil deteriorated. Their percentage, above all in Oil 2, was significant. This is a signal in the event of further large disturbances to check the technical condition of the lubrication system.

The results of the water separation test from the oil samples are shown in Table 6.

Table 5. The results of the water separation test for the oil samples

Month of oil sampling	Oil 1		Oil 2	
	Result according to ISO 6614	Total phase separation time of the oil/ water emulsion [min]	Result according to ISO 6614	Total phase separation time of the oil/ water emulsion [min]
December	40-40-0 (10)	10	40-40-0 (21)	21
January	40-40-0 (9)	9	40-40-0 (19)	19
February	40-40-0 (8)	8	40-40-0 (16)	16
March	40-40-0 (9)	9	40-40-0 (18)	18
April	40-40-0 (11)	11	40-40-0 (18)	18

Analysing the results, it was noted that the phase separation times for Oil 1 emulsions were considerably shorter compared to those measured for Oil 2 samples. In most cases, it was essentially twice as long. The results were recorded in the form of the code described in ISO 4416. All analyses resulted in complete phase separation of the oil/water emulsion samples. A 40 ml oil sample and 40 ml distilled water were used. The water separation tests have shown that both oils do not form permanent emulsions with water. From the point of view of working in a system composed of metal components, this is good news. The oils do not retain water, which directly eliminates the chances of excessive corrosion of lubrication system parts and machinery. Oil 2 had a higher viscosity than Oil 1, which may have had an effect on slower phase separation. The water molecules remained suspended longer in the emulsion solution, that's why the rate of separation was slower. The shorter separation times for Oil 1 may also have been related to the higher content of deemulsifying agents in its formulation. The differences in phase separation times in the months in question are also a result of the oil purification processes carried out, e.g. filtering and partial replacement with fresh oil.

The aniline point determination was carried out over a period of three months, as its purpose was to determine the overall content of aromatic compounds in the composition of the oils. The results are presented in Table 7.

Table 7. Aniline point results of oil samples

Month of oil sampling	Aniline point [°C]	
	Oil 1	**Oil 2**
December	119.0	108.0
January	119.0	107.5
February	119.0	108.0

Determination of the aniline point was a test to see how many aromatic compounds were present in the compositions of the oils tested. The results obtained are typical of mineral oils (aniline point of approx. 97–117°C), confirming that the lubricants tested have a mineral base. Oil 1 was characterised by a higher temperature at the aniline point than Oil 2. The composition of Oil 1 thus contained fewer aromatic compounds than Oil 2. The results had virtually no differences from one month to the next, so the possibility that arene would be formed in significant amounts during oil use was excluded. Their content was related to the presence of a fresh, untreated system already in the formulation. This is good information, as a significant amount of aromatic compounds formed during oil operation could have contributed to an increased risk of oxidation or reduced thermal resistance. However, arenes did not make up the majority of the compounds present in the oil formulation, because if they had, the temperature of the aniline points would have been much lower.

The formaldehyde test was, in addition to the aniline point, a qualitative determinant of the content of aromatic compounds in the compositions of the oils tested. For the samples using both oils, the observations were identical. A black sediment was observed to precipitate, as well as a change in the colour of the solution to dark brown. For both oils tested, the result was indicative of a sizable proportion of aromatic compounds in their compositions. The black precipitate consists of large organic compounds, formed by condensation. Both oils have a mineral base, making the positive result of the Nastyukov test unsurprising. The results of the formaldehyde test were in line with the conclusions regarding the interpretation of the aniline score values of the oils tested.

Conclusion

Oil samples were tested from various angles to ensure that the results were consistent with reality. Diagnostics cannot be carried out based on the determination of one parameter, ignoring the others.

The obtained results suggest that both Oil 1 and Oil 2 are perfect for operation in the systems in which they are used.

Oil 1, used in the turbine, both in the lubrication system and in its operation regulation, was characterized by appropriate kinematic viscosity. It was able to keep it at a constant level throughout the period of research. The temperature and pressure in the system did not result in a rapid degradation of its rheological properties. The problem that arose after performing comprehensive tests was the high content of solid impurities. Their level should be constantly reduced by performing more frequent filtering (among other methods). Perhaps the solution to this problem could be the use of other filters in the lubrication system.

Oil 2 also has very good properties in the context of compressor operation. Throughout the entire testing period, it maintained the appropriate density despite the fact that it worked under significantly higher pressure than Oil 1. The viscosity fluctuated slightly over time, so its determination should continue to be carried out regularly. Solid impurities were a drawback that often resulted in the need to filter or even refresh some of the oil. If their level is still not reduced and their content increases more and more often, it will be necessary to carefully check the condition of the installation.

Both of the tested oils are characterized by good physicochemical properties in the place where they are used. The viscosity of Oil 2 was significantly higher than that of Oil 1, which is typical for lubricants used in compressors compared to those used in turbines. In the case of Oil 2, changes in viscosity were observed, which confirmed its partial consumption. The results from December and March are the basis for partial oil replacement with fresh oil. Additionally, oil filtering should be performed frequently to get as close to the allowable range as possible. The lower results in January and April were certainly the result of the oil regeneration and refreshment processes carried out in the installation. The results for Oil 2 at a temperature of 100°C directly reflected the recommendation of actions that should be taken to maintain its proper properties. Overly high viscosity of Oil 2 could be caused by the negative impact of the pressure and temperature in the system in which it worked. Its rheological parameters could also deteriorate as a result of catalysing the oxidation reaction of oil components through impurities that could contain metals.

Moreover, the water separation test showed that Oil 1 is separated from water in a much shorter time, which is extremely satisfactory in the context of operation in a turbine. This aspect directly contributes to the longer life of this device. It is also worth noting that both oils have a low water content and have the ability to quickly release water from the emulsion. This is safe for the system and does not cause rapid wear of its metal elements.

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Appendix 1

The following equipment was used in the individual analyses:

1. Density determination according to PN-EN ISO 3675
    - Thermoareometer for mineral oils, manufactured by GOMAR WARSZAWA, measuring range: 0.750–0.840 g/cm^3, thermometer range: from −10°C to −30°C, density measurement accuracy: 0.001 g/cm^3
2. Acid value determination according to PN-ISO 6619
    - Titromatic S2 titrator, manufactured by CRISON/Labindex
    - CRISON glass electrode, electrolyte: LiCl solution with a concentration of 0.1 mol/dm3 in ethanol.
    - AB204/A laboratory balance, manufactured by METTLER TOLEDO, measuring range: 0.0001–−200 g, measuring accuracy: 0.0001 g.
3. Water content determination according to PN-EN ISO 12937
    - Cou-Lo Aquamax KF coulometric titrator, manufactured by GRSCIENTIFIC/Labindex.
4. Kinematic viscosity determination according to PN-EN ISO 3104.
    - WLM ME viscosimetric bath
    - WKT thermometer (measuring range: −50°C to 200°C, accuracy: ±0,5°C)
5. Purity determination according to PN-ISO 4406
    - MAHLE PiC 9300 automatic particle counter
    - MOTIC B1-223A biological microscope
    - AGA LABOR TL 2/4 vacuum pump
    - CDS-100 ultrasonic washer
    - Filters manufactured by Millipore S-Pak Membrane Filters MCE, gradation: 1.2 µm, diameter: 47 mm, 41
6. Water separation test according to ISO 6614
    - CAT E50 mechanical stirrer, speed range 50–−1600 rpm.
    - IKA C-MAG HS7 magnetic stirrer with compact thermometer.
7. Aniline point determination according to PN-ISO 2977
    - CAT E50 mechanical stirrer, speed range 50–−1600 rpm.
    - CHEMLAND 98-I-B heating jacket with power regulator, 150W.

The following reagents were used in the individual analyses:

1. Acid value determination according to PN-ISO 6619
    - Potassium hydroxide (KOH) solution in propan-2-ol at a concentration of 0.1 mol/dm^3
    - Mixture of solvents: toluene, propan-2-ol, distilled water from Chempur.
    - Buffer solution with pH = 7.00 from Hach Polska
    - Buffer solution with pH = 9.21 from Hach Polska
2. Determination of water content according to PN-EN ISO 12937
    - Commercial catholyte: Aquagent Coulometric CG, catholyte for coulometric Karl Fischer titration (5 ml) from Scharlau
    - Commercial anolyte: Aquagent Coulometric Oil (100 ml) from Scharlau
3. Determination of kinematic viscosity according to PN-EN ISO 3104
    - Methyl silicone oil for viscosimetric bath Polsil OM-100
4. Determination of aniline point according to PN-ISO 2977
    - Aniline 99,5% from POL-AURA.