Institute of Mathematics, Physics and Chemistry

Department of Chemistry

Laboratory of fuels, oils and lubricants

Laboratory exercise

Measurement and evaluation of operational parameters of plastic lubricants

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Szczecin

EXERCISE SHEET

<table>
<tr><td rowspan="3">1</td><td colspan="3">Relation to subjects: Marine Power Plant Operation/28</td></tr>
<tr><td>Specialty/Subject</td><td>Learning outcomes for the subject</td><td>Detailed learning outcomes for the subject</td></tr>
<tr><td>MPPO – Chemistry of fuels and lubricants.</td><td>EKP3
K_U014, K_U015, K_U016.</td><td>SEKP12 – Performing determinations of selected quality indicators of petroleum products.</td></tr>
<tr><td>2</td><td colspan="3">Purpose of the exercise:
teaching the student how to independently measure the operational parameters of plastic lubricants: penetration, dropping point, resistance of lubricants to water and methods of their identification.</td></tr>
<tr><td>3</td><td colspan="3">Prerequisites:
the student is trained in the occupational health and safety regulations on a laboratory stand, which he confirms with his signature on the appropriate form, knows – the concept of plastic lubricant, chemical composition and types of lubricants, additives, methods of measuring penetration and determining the lubricant consistency classes according to NLGI, method of measuring lubricant dropping and determining safe operating temperature, method of testing water resistance and identification of the type of lubricant.</td></tr>
<tr><td>4</td><td colspan="3">Description of the laboratory workplace:
penetrometer with a lubricant kneader, apparatus for measuring the dropping point of lubricants, standard stains of plastic lubricant, filter paper, timer, plastic lubricant samples.</td></tr>
<tr><td>5</td><td colspan="3">Risk assessment *:
heating the lubricant in a hot oil bath to a temperature of up to 220°C, contact with hot oil – there is a possibility of thermal burns with hot oil.
Final assessment – CONSIDERABLE HAZARD, SERIOUS EFFECTS
Security measures required:
a. lab coats, protective glasses,
b. health and safety cleaning products, cleaning cloths, paper towels,
c. petroleum products waste container (for disposal).</td></tr>
<tr><td>6</td><td colspan="3">The course of the exercise:
a. Read the workplace manual (appendix 1) and familiarize with the laboratory kit for the exercise,
b. Perform a determination of the penetration of the tested lubricant, knead the lubricant in a kneader, measure the penetration after kneading,
c. Determine the drop point of the tested lubricant,
d. Perform a water resistance test of the lubricants,
e. Identify the lubricants by paper analysis.</td></tr>
<tr><td>7</td><td colspan="3">Exercise report:
a. Develop the exercise in accordance with the instructions contained in the workplace manual,
b. On the basis of the marked penetration of the lubricant after kneading, determine the consistency class of the lubricant according to NLGI,
c. Determine the maximum operating temperature of the lubricant on the basis of the dropping point measurement,
d. Determine the water resistance of the lubricant,
e. Identify lubricant samples by paper analysis;</td></tr>
</table>

8	**Archiving of research results:** Submit a written report on the performed exercise to the academic teacher.
9	**Assessment method and criteria:** a. EKP1, EKP2 – tasks given for independent solution and development: mark 2.0 – the student has no basic chemical, physicochemical and operational knowledge concerning the properties and performance parameters of plastic lubricants and the ability to solve simple tasks in this field; mark 3.0 – has basic chemical, physicochemical and operational knowledge concerning the properties and performance parameters of plastic lubricants, as well as the ability to calculate and solve simple tasks in this field; mark 3.5 – 4.0 – has extensive chemical, physicochemical and operational knowledge of the physicochemical and functional properties of plastic lubricants and the ability to solve complex tasks in this field; mark 4.5 – 5.0 – has the ability to apply complex chemical, physicochemical and operational knowledge to assess the quality and usability of the tested plastic lubricants due to the determined performance parameters; b. EKP3 –control works: mark 2.0 – does not have the ability to analyse and evaluate the results of the performed analyses and determinations and to draw conclusions; mark 3.0 – has the ability to analyse the obtained results, interpret the laws and phenomena, transform formulas, and interpret charts and tables; mark 3.5 – 4.0 – has the ability to broaden the analysis of results, apply laws, construct monograms and charts; mark 4.5 – 5.0 –has the ability to comprehensively analyse the obtained results, make generalizations, detect cause-and-effect relationships and make the right operational decisions.
10	**Literature:** 1. Krupowies J., Wiznerowicz Cz.: Pomiar i ocena parametrów użytkowych smarów plastycznych. Instrukcja stanowiskowa do ćwiczenia, AM, Szczecin 2013. 2. Barcewicz K.: Ćwiczenia laboratoryjne z chemii wody, paliw i smarów. Wyd. AM w Gdyni, Gdynia 2006. 3. Podniało A.: Paliwa oleje i smary w ekologicznej eksploatacji. WNT, Warszawa 2002. 4. Przemysłowe środki smarne. Poradnik. TOTAL Polska Sp. z o.o., Warszawa 2003. 5. Czarny R.: Smary plastyczne. WNT, Warszawa 2004. 6. Urbański P.: Paliwa i smary. Wyd. FRWSzM w Gdyni, Gdańsk 1999. 7. PN/EN/ISO standards for the testing of petroleum products. 8. Oil product catalogs of oil companies. 9. Baczewski K., Biernat K., Machel M.: Samochodowe paliwa, oleje i smary. Leksykon, Wydawnictwa Komunikacji i Łączności, Warszawa 1993. 10. Herdzik J.: Poradnik motorzysty okrętowego. Wydawnictwo TRADEMAR, Gdynia 1995.
10	Notes

APPENDIX 1 – MANUAL

1. SCOPE OF THE EXERCISE

- getting acquainted with the workplace instructions for the exercise,
- penetration measurement and determination of the consistency class of lubricants,
- measuring the dropping point of lubricants and determining the safe operating temperature of the lubricant,
- testing of lubricant resistance to water,
- identification of the type of lubricant on the filter paper.

2. THEORETICAL INTRODUCTION TO THE EXERCISE

2.1. Plastic lubricants

Plastic lubricants are colloidal systems in which the dispersing phase is the lubricating base oil, and the dispersed phase is the thickeners.

The most common thickeners are soaps, i.e., salts of higher fatty, resinous, naphthenic acids or solid natural hydrocarbons (e.g., paraffin, ceresin, petrolatum, asphalt) or synthetic hydrocarbons. Soaps used in the production of lubricants are divided into: sodium, potassium, lithium, silver, calcium, lead, barium, strontium, zinc, magnesium, aluminium and other soaps.

Non-hydrocarbon natural substances (e.g., bentonite) or synthetic substances (e.g., aryl urea compounds) are also used as thickeners. For special purposes, lubricants are used with the following inorganic substances as thickeners, e.g., with active clay, bentonite, graphite or molybdenum disulphide.

Due to the wide use of such solid lubricants as graphite, molybdenum disulphide, Teflon, silicones, etc., the used term "solid lubricants" has become obsolete. For this reason, the name "plastic lubricants" was adopted to describe the discussed group of lubricating products.

Lubricating greases are plastic lubricants that have a solid or semi-fluid consistency at normal temperatures. Plastic lubricants are obtained by thickening lubricating oils to a plastic consistency. They are used when the use of more effective oils is pointless or impossible. They are therefore lubricants used for various mechanisms in which liquid oils either cannot hold on or cannot be continuously supplied to them. Plastic lubricants are used to reduce the frictional resistance between mating surfaces (anti-friction lubricants) or to temporarily protect metal surfaces against corrosion (protective lubricants). The consumption of plastic lubricants in different countries ranges from 7 to 12% of the amount of lubricating oil consumption. Plastic lubricants are mainly used to lubricate rolling and sliding bearings in many machines and devices in industry, transport, construction and agriculture.

Depending on the type of soap used as a thickener, we distinguish between lubricants: sodium, potassium, sodium-potassium, lithium, silver, calcium, lead, barium, strontium, zinc, magnesium, aluminium and others.

Plastic lubricants can be modified, e.g., calcium lubricants by adding graphite, and then we obtain graphite lubricants, used to lubricate friction junctions, where there are very high pressures.

Synthetic lubricants are also used, obtained from synthetic oils, such as: silicone oil, ester oil and synthetic thickeners, such as modified silica, arylurea derivatives and others.

The functional properties of plastic lubricants depend on the type of liquid medium thickener (mineral or synthetic oil) used, dispersion conditions and the presence of certain polar substances, so-called structure modifiers, such as: water, fatty acids, alcohols, esters, salts of low molecular weight organic acids and others. Some lubricants also contain enriching additives, such as oxidation and corrosion inhibitors, additives increasing the strength of the lubricant film, increasing adhesion and resistance to high pressures. Different types of lubricants, depending on the type of thickener, have characteristic properties, the knowledge of which allows to determine the scope of their applicability.

Selected, aging-resistant raw materials and the addition of inhibitors of this process in the form of antioxidants are used for the production of lubricants intended for long-term operation without replacement, e.g., in roller bearings. Lubricants intended for operation at low temperatures are produced with the use of base oils with appropriate rheological properties at minus temperatures, while high-temperature lubricants contain oils of higher viscosity and low evaporation.

2.2. Functional properties of plastic lubricants

When it comes to assessing the quality of plastic lubricants, the determination of their properties, such as: dropping point, water content, soaps, free bases and acids, ashing residues, are only relevant as a means of controlling the uniformity of production.

In the conditions of hydrodynamic lubrication, the rheological properties of the lubricant are the most important. The most common way of determining these properties for lubricants is to determine the penetration, i.e., the conventional value expressing the immersion depth of the standard penetration cone in the sample of the tested lubricant after kneading. Based on the penetration, the consistency class of the lubricant is then determined. Like the viscosity of lubricating oils, penetration is the basis for the classification of lubricants according to the so-called grades of consistency. The knowledge of the penetration value enables in practice an approximate assessment of the pressures needed to supply lubricant through lines and the frictional resistance of the lubricant in the bearing, especially when starting machines and devices. Lower penetration lubricants have better sealing properties of the friction junctions. Penetration, however, does not give an idea of the viscosity of the lubricant under operating conditions, because it depends mainly on the thickener content in the lubricant. On the other hand, the viscosity of a lubricant also depends on its structure and the viscosity of the oil used in the production of the lubricant, therefore the standards should specify the viscosity of the oil in a given lubricant.

The class of lubricant consistency based on the worked penetration is obtained from Table 1:

Table 1

Plastic lubricant grades according to NLGI
(National Lubricating Greases Institute)

Consistency class according to NLGI	Penetration after kneading at the temperature of 25 °C in the range	Consistency
000	445 ÷ 475	very fluid
00	400 ÷ 430	fluid
0	335 ÷ 385	semi-fluid
1	310 ÷ 340	very soft
2	265 ÷ 295	soft
3	220 ÷ 250	medium
4	175 ÷ 205	semi-hard
5	130 ÷ 160	hard
6	85 ÷ 110	very hard
7	40 ÷ 70	very hard

Due to their structure, plastic lubricants exhibit the properties of solid and liquid bodies at the same time. Under slight pressure, they behave like solids, undergoing reversible elastic deformation. Under the influence of pressures exceeding their strength range, they deform irreversibly and begin to flow, which makes them good lubricants.

The dropping point of a lubricant is the temperature at which the first drop of lubricant falls when placed in the cup of the Ubbelohde apparatus and heated under strictly defined conditions.

If the tested lubricant does not flow down in drops, but in the form of a cylindrical column, then the dropping point should be the temperature at which the extended column reaches the bottom of the test tube.

The specified method determines the dropping point of plastic lubricants, petroleum jelly and terein.

Based on the dropping point of the lubricant, the safe operating temperature of the lubricant is determined, which is usually lower by 30º to 90ºC than the dropping point of the lubricant, depending on the type of lubricant.

An important functional property of lubricants is their resistance to water. It depends on the type of soap used as a lubricant thickener. Since sodium, potassium and sodium-potassium mixed soaps are water-soluble, therefore, lubricants containing these soaps are not resistant to water. Other lubricants, i.e., lithium, silver, calcium (so-called towoty), lead, barium, strontium, zinc, magnesium and aluminium lubricants are resistant to water.

The influence of the thickener type on the properties of plastic lubricants is illustrated in Table 2.

Table 2

Influence of the type of thickener on the properties of plastic lubricants

Lubricant	Dropping point [ºC]	Application temperature range [ºC]	Water resistance	Mechanical stability	The effectiveness of the additives	Notes
Calcium normal	90 ÷ 125	–20 ÷ 60	high	high	high	very cheap
Calcium anhydrous	140 ÷ 160	–40 ÷ 100	high	high	high	–
Calcium complex	240 ÷ 280	–40 ÷ 150	high	high	good	a tendency to harden at high temperatures
Lithium	180 ÷ 205	–40 ÷ 140	good	high	good	the most widely used
Lithium complex	220 ÷ 310	–60 ÷ 180	high	high	medium	difficult technology, expensive
Aluminium complex	250 ÷ 280	–40 ÷ 150	high	good	medium	simple technology, cheap
Sodic	180 ÷ 210	–40 ÷ 100	none	good	good	washable with water
Bar complex	250 ÷ 270	–40 ÷ 150	high	high	high	excellent adhesion to metals, difficult technology, expensive
Polyurethane	240 ÷ 300	–40 ÷ 150	high	good	high	a tendency to harden during storage
Bentonite	infusible	–60 ÷ 200*	medium	low	low	a tendency to soften at high temperatures
Silicone gel	infusible	–60 ÷ 200*	high	low	medium	a tendency to soften at high temperatures

* – based on synthetic oil

3. PERFORMING THE EXERCISE

3.1. Measurement of the penetration of lubricants

Penetration is a number that indicates the depth to which the penetration cone will plunge into the tested lubricant under a load of 150g, at a temperature of 25°C, during 5 seconds.

The unit of measurement for penetration is a unitless number, corresponding to 0.1 mm of the countersink of the standard penetration cone.

The described method is used for lubricants with a penetration not exceeding 400. Depending on the method of sample preparation, a distinction is made between the penetration of the lubricant without and after kneading.

Measurement of penetration without kneading consists in determining the depth of immersion of the penetration cone in a sample of the same consistency as the tested lubricant. If the test sample is subjected to mechanical kneading in a special kneader, in a strictly defined manner, and then the measurement of this lubricant in the penetration vessel, the obtained result will be penetration after kneading.

3.1.1. Penetration measurement without kneading

After removing the 50 mm thick top layer of lubricant, fill the penetration vessel – 1 with lubricant without kneading, so that it does not change its original consistency (fig. 1).

Then place the open vessel in a water bath at 25°C, taking care that the surface of the lubricant does not come into contact with water. After 1 hour remove the vessel from the bath and smooth the surface of the lubricant. Place the penetration vessel I with the lubricant on the penetration table. Place the penetration cone – 3 in the center of the test sample so that its end touches the surface of the lubricant. This setting is best observed in the mirror – 4. After zeroing the apparatus, start the timer and press the button – 5 at the same time, release the pin – 6 of the penetrometer, holding it in this position for 5 seconds. During this time, the cone should freely sink into the lubricant. Then move the rod – 7 to the pin – 6 and read the penetration indicated on the disc – 8.

Lift the pin – 6, thoroughly clean the cone – 3 and repeat the measurement.

Note!

If the penetration of the tested lubricant is greater than 200, perform only one measurement in one vessel, placing the cone in the centre of the penetration vessel. Carry out further measurements on freshly prepared lubricant samples.

If the penetration of the tested lubricant is less than 200, repeat the measurements three times in the same vessel, placing the penetration cone halfway between the rim and the centre of the vessel, at three points equidistant from each other.

Do not smooth the surface of the lubricant after the previous measurements.

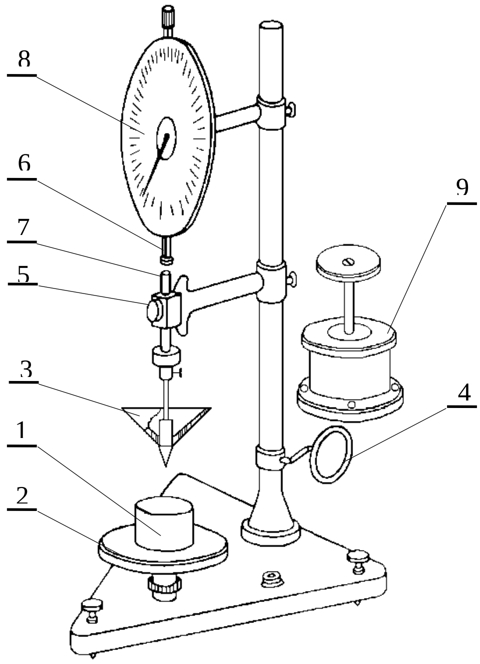


Fig. 1. Penetrometer:
1 – vessel I, 2 – table, 3 – cone, 4 – mirror, 5 – button,
6 – pin, 7 – rod, 8 – disc, 9 – vessel II (kneader)

Elaboration of the results

Take as the final result the average value of the measurements with an acceptable difference between each other by 12 divisions.

3.1.2. Penetration measurement after kneading

After removing the top layer from the tested 50 mm thick sample of lubricant, fill the penetration vessel II (kneader) with it. After closing, put the vessel into a temperature of 25°C. The water level should be at least 10mm above the lid of the vessel. Keep them in the bath until the lubricant reaches 25ºC. Then remove the vessel from the bath and make 60 full (double) strokes of the piston within 60 seconds. Finish kneading when the plunger is at the top of the vessel. In order to remove air bubbles, use a spatula several times to shift the lubricant from the bottom of the vessel onto its surface and press it back into the vessel. Next, transfer the tested lubricant to the penetration vessel – 1 and, after smoothing the surface, start the measurement.

The penetration vessel I with the lubricant should be placed on the penetration table-2, and the penetration cone-3 in the centre of the test sample so that its end touches the surface of the lubricant.

After the apparatus has been reset, start the timer by simultaneously pressing the penetrometer pin release button and hold it in this position for 5 seconds. Then slide the rod against the pin and read the penetration. After the first measurement, you should quickly start the next measurements on the same sample, after mixing, denting and smoothing its surface.

Elaboration of the results

The final result should be the average of the measurements with an acceptable difference between themselves by 8 divisions.

3.2. Measurement of the dropping point of lubricants using the Ubbelohde method

The dropping point according to this method is the temperature at which the first drop of the tested lubricant drops, placed in the cup of the Ubbelohde apparatus and heated under strictly defined conditions.

If the tested product does not flow down dropwise, but in the form of a cylindrical bar, then the dropping point should be taken as the temperature at which the protruding bar reaches the bottom of the test tube.

The specified method determines the dropping point of plastic lubricants, petroleum jelly and terein. The principle of marking is based on the use of a standard Ubbelohd apparatus.

Performing the determination

Use a spatula to fill the vessel-1 of the Ubbelohde apparatus with the tested lubricant (Fig. 2), making sure that no air bubbles remain inside the lubricant. Scrape off excess lubricant with a spatula. Then insert the vessel into the metal cap – 2 so that its edge touches the rivets. Scrape lubricant pressed out by the thermometer with a spatula. Place the Ubbelohde apparatus with the tested product concentrically in the test tube using a stopper.

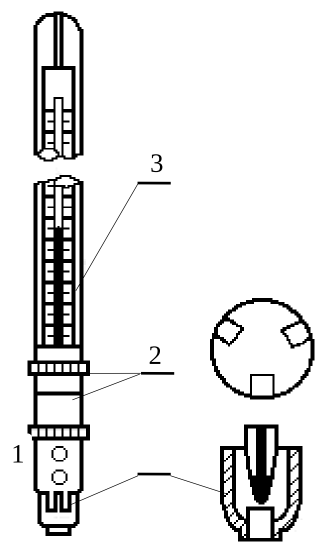


Fig. 2. The Ubbelohde apparatus:
1 – vessel, 2 – metal cap, 3 – thermometer

Place the set prepared in this way with the stopper concentrically in the test tube. The plug should have a vertical groove on the side for pressure equalization. The test tube with the thermometer is fixed in a holder and placed in a water bath or, for lubricants with a dropping point higher than 90 °C, in an oil or glycerine bath. Up to a temperature of about 15 ° C lower than the expected softening point of the lubricant, heating is carried out fairly quickly. Above this temperature, the temperature rise should not be more than 1 °C per minute. While heating water or glycerine in the glass, stir the liquid with a mechanical agitator. The dropping point of the tested lubricant is taken as the temperature at which the first drop falls, or at which the column of lubricant protruding from the cup touches the bottom of the test tube.

Elaboration of the results

The final result will be the arithmetic mean of at least two measurements, not differing by more than 20ºC.

3.3. Identification of plastic lubricants
Testing the resistance of lubricants to water

The type of lubricant is determined by the type of thickening agent present in it. Thus, the determination of the type of lubricant comes down to the type of thickeners present in it, which are most often soaps, solid hydrocarbons or inorganic substances.

Note!

Before starting the identification of a lubricant sample, determine its penetration and dropping point.

3.3.1. Testing the resistance of lubricants to water

Rub a small amount of the tested lubricant between the fingers under the stream of running water. If the lubricant is resistant to water (not saponified), it can be considered to be one of the following lubricants, e.g., lithium, silver, lead, barium, strontium, zinc, magnesium, aluminium (they are resistant to water). When a lubricant is saponified and washed out, it is considered a sodium, potassium or sodium-potassium lubricant.

On the other hand, when the lubricant is easily emulsified during the above test (changes its colour to a brighter colour), but does not wash out, it can be considered as calcium lubricant (commonly referred to as towot).

Elaboration of the results

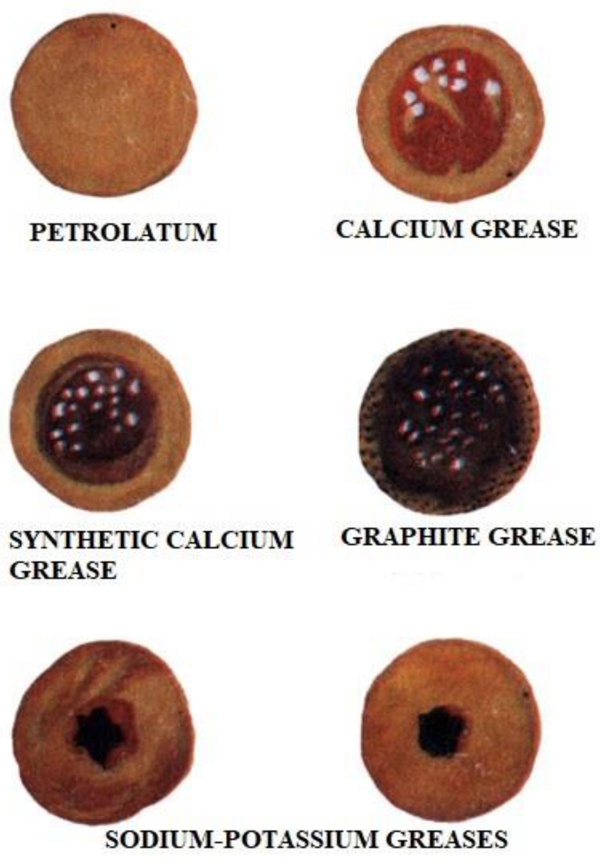
Determine the resistance of the tested lubricant to water and on this basis determine the type of soap used in the production of the tested lubricant.

3.3.2. Examination of a lubricant stain on a filter paper

The crushed balls of the test lubricant, about 1 – 2 mm in diameter, are placed on a filter paper and carefully heated over an electric razor. During heating, the fusible components of the lubricant will soak into the filter paper, and the remaining components will form a coloured stain on it. Perform identification knowing that:

* technical petroleum jelly completely melts and soaks up, leaving a uniform, light stain;
* calcium lubricants create a light spot in the centre of which a soft unmelted residue with water droplets is visible;
* sodium and sodium-potassium lubricants remain almost unchanged with a light border coming from the oil;
* scattered graphite particles are visible in the stain from graphite lubricant.

PATTERN STAINS OF SOME LUBRICANTS ON THE FILTER PAPER



4. DEVELOPMENT OF THE EXERCISE

1. Determine the lubricant consistency class according to NLGI (*National Lubricating Greases Institute*) by measuring the penetration of the lubricant after kneading. Compare the obtained result with the catalogue size for a given lubricant.
2. Compare the determined dropping point with the value given in the table under 3.1. of this manual and establish the safe operating temperature of the lubricant.
3. Present in the form of a table all obtained results (penetration, dropping point, consistency class, resistance to water, type of lubricant identified on the basis of its stains on the blotting paper in comparison with the reference stains and on the basis of the water resistance test).
4. Describe the type and nature of the stain obtained.
5. The appendices to the exercise include a scientific article at the end of the manual on the physicochemical and functional properties of modern plastic lubricants.

5. THE FORM AND CONDITIONS FOR PASSING THE LABORATORY EXERCISE

1. passing the so-called "entry" before starting the exercise.
2. submission of a correct written report on the completed exercise, which should contain:

   - short theoretical introduction,
   - operational significance of the measured parameters,
   - processing of the obtained results according to the position manual.

3. final credit for the test at the end of the semester.

Tasks and questions to be completed by the student

Tasks

1. The drop point of calcium lubricants is between 80 and 120°C. On this basis, determine the highest safe temperature of their operation.
2. Knowing that the dropping point of sodium and potassium lubricants is within the limits of 130 – 200°C, give the highest temperature of their application. Under what conditions can these lubricants not be used?
3. The drop point of lithium lubricants is in the range of 170 – 220°C. Provide safe working temperature and application.
4. The penetration of ŁT43 lubricant after kneading at 25°C was on average 235. Determine its consistency class according to NLGI and determine whether it is the correct consistency class for this lubricant.

Questions

1. Define the concept of plastic lubricant.
2. Explain the general chemical composition and give the classification of plastic lubricants.
3. What is the aging process of plastic lubricants?
4. What is the purpose of measuring the penetration of lubricants and what are they determined?
5. What is the drop point of a lubricant, how is it determined and for what purpose?
6. What is the influence of water and temperature changes on the performance of plastic lubricants? Provide water-resistant and water-sensitive lubricants.
7. How is the test for the detection of mechanical impurities in the lubricant and the resistance of the lubricant to water carried out?
8. How is the type of lubricant identified on the filter paper?
9. What are dry lubricants, specify their types, properties and application.
10. What is the classification of plastic lubricants according to NLGI?
11. What are EP, AW and anti-corrosive additives for lubricants?
12. What are the MoS_2 molybdenum disulphide additives in lubricating products used for?