# **APARATURA** BADAWCZA I DYDAKTYCZNA

A new method and equipment for the detection of presence and estimation of the content of the petroleum-based fraction in lubricating oils, especially those emitted to the atmosphere

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**Keywords:** lubricating oils emitted to the environment, the presence and estimated content of aromatic hydrocarbons, field test, method and equipment, fluorescence

#### ABSTRACT:

Lubricating oil, used in cutting equipment, works in the so-called open lubrication system and is entirely emitted to the environment. When such an oil contains even a small portion of the crude oil-derived fraction, which is still very common, the oil is a serious environmental pollution. In addition, the oil mist poses a serious threat to the health of employees. Current legal regulations require users of saws and harvesters to use only biodegradable oils. In the case of hydraulic oils used in machines and vehicles, there is always a risk of leakage into the environment e.g. in case of breakage of connecting pipes or other types of breakdowns. Therefore, it seems reasonable to postulate the use of biodegradable oils in power hydraulics systems of machines used in forests, fields, mines or in rail or road vehicles. Biodegradable oil, most often, with base vegetable oil, is more expensive than lubricating oil produced with the use of an oil-based oil base – i.e. a product of oil refining. Today, users of saws and harvesters still use mineral lubricating oils from crude oil, without any attention to the negative consequences for health and the environment. The known methods of the assessment of biodegradability of lubricating oils are expensive and time-consuming. The paper proposes a principle of method and equipment for performing a quick and cheap screening test for the presence of petroleum-derived fractions containing aromatic hydrocarbons in the analyzed lubricating oil samples. The test uses an oil fluorescence test under UV light of wavelength  $\lambda$  = 365 nm. The proposed method is simple and does not require specialist qualifications from the user. Easy-to-prepare equipment for repetitive testing in field conditions has been described.

# Nowa metoda i wyposażenie do badania obecności i orientacyjnej oceny zawartości frakcji naftowej w olejach smarowych, szczególnie emitowanych do środowiska podczas użytkowania

**Słowa kluczowe:** oleje smarowe emitowane do środowiska, badanie obecności / orientacyjna ocena zawartości węglowodorów aromatycznych, test terenowy, metodyka i wyposażenie, fluorescencja

#### STRESZCZENIE:

Olej smarowy, wykorzystywany w urządzeniach tnących, pracuje w tzw. otwartym układzie smarowania i w całości jest emitowany do środowiska. Wówczas, gdy taki olej zawiera nawet niewielką część frakcji pochodzącej z ropy naftowej, co ciągle jeszcze ma nagminny charakter, olej stanowi poważne zanieczyszczenie środowiska. Do tego mgła takiego oleju stanowi poważne zagrożenie dla zdrowia pracowników. Aktualne regulacje prawne wymagają od użytkowników pilarek oraz harwesterów stosowania wyłącznie olejów biodegradowalnych. W przypadku olejów hydraulicznych stosowanych w maszynach i pojazdach zawsze istnieje niebezpieczeństwo wycieku lub wylewu do otoczenia w przypadku pęknięcia przewodów łączących albo innego rodzaju awarii. Celowy wydaje się więc też postulat stosowania biodegradowalnych olejów w systemach hydrauliki siłowej maszyn użytkowanych w lasach, na polach, w kopalniach czy w pojazdach szynowych lub drogowych. Biodegradowalny olej, najczęściej z bazowym olejem roślinnym, jest droższy od oleju smarowego wyprodukowanego z zastosowaniem, nawet tylko w części, naftowej bazy olejowej – produktu rafinacji ropy naftowej. Dzisiaj użytkownicy pilarek oraz harwesterów ciągle jeszcze stosują mineralne oleje smarowe z ropy naftowej, nie licząc się z negatywnymi konsekwencjami dla zdrowia i środowiska. Znane metodyki badania biodegradowalności olejów smarowych są kosztowne i czasochłonne. W pracy zaproponowano zasadę wykonania oraz wyposażenie do wykonania szybkiego i taniego testu przesiewowego obecności frakcji pochodzenia naftowego, zawierającej weglowodory aromatyczne, w badanych próbkach oleju smarowego. Test wykorzystuje badanie fluorescencji oleju w świetle UV -  $\lambda$  = 365 nm. Proponowana metoda jest prosta w wykonaniu i nie wymaga specjalistycznych kwalifikacji od wykonawcy. Opisano łatwe w przygotowaniu wyposażenie do powtarzalnego wykonywania badań w warunkach terenowych.

## **1. INTRODUCTION**

Current legal regulations in Poland and in the European Union require forest entrepreneurs and operators of chainsaws and harvester to use only biodegradable oils to lubricate the cutting system i.e. the chain [1-3].

Polish General Directorate of the State Forests published the decision of the General Director of the State Forests No 243 of 14 September 2017 on making uniform templates for documents regarding forestry services in all organizational units of State Forests, informing that forest inspectorates obligatorily require the use of biodegradable oils in cutting systems. Appointed inspectors are approved to take samples [2]. Unfortunately, despite the law regulations, many operators still use inappropriate types of lubricating oils, mainly because of economic reasons. Lubricating oils consist of oil base and a package of additives. The quantity, type and mutual proportions of the components determine the class of the oil. In modern oils, the amount of additives ranges from a fraction to several and approximately a dozen percent, and the remaining part is the base oil. Thus, base oil material is the main component (80-100% by volume) of the lubricating oil [4]. Due to the origin of base oils, mineral oils (produced from crude oil), synthetic oils (i.e. PAO, PAG) or oils of natural origin (vegetable or animal) are distinguished [5]. The most commonly used base oil is petroleum originated oil. Its production is cheaper than for synthetic or vegetable oil base. In addition, mineral oil is characterized by good lubricating properties [6]. Therefore, many users of saws and harvesters, but also gardeners use mineral oils [6-9].

Oils used to lubricate the cutting system of a harvester cutter or pummel, work in an open lubricating system which results in the oil emission to the atmosphere.

The emission of petroleum-based lubricating oils negatively affects the human health, the ecosystem and the environment [7]. The strength and effects of the lubricating oil on the environment are closely related to the type of oil and additives [10-11]. Oil mist emitted during the operation of the cutting device may penetrate through the skin or through the respiratory system into the human body [12]. These results in allergic reactions, changes in internal organs (in the lungs, in the liver, kidneys, adrenal glands and heart). Moreover, long-term and regular contact with the mist can lead to diseases related to the nervous system or cancer, in particular skin cancer [10, 13-14]. Additionally, by creating a film of petroleum-based substance on the surface of a water reservoir, disturbances in the oxygen exchange between the water and the atmosphere occur, limiting the access to light and UV, causing changes in the functioning of aquatic organisms and lead to eutrophication [12, 15-16]. What is more, soil contaminated with petroleum-based lubricants becomes a hazardous material with disturbed functioning of the ecosystem. Mineral oil reduces the permeability of soils and the aeration of groundwater, leading to soil degradation due to oxygen deficit [11, 17-18].

Most of the crude oil components are slowly biodegraded and at the same time enter into unavoidable interactions with environmental components. As a consequence, the newly formed secondary chemicals are mostly much more toxic than the original ones, and have a possibly undefined harmful influence on health [9, 19].

Biodegradability is one of the most important requirement for oils used to lubricate open cutting systems. Biodegradability is defined as the distribution of complex organic compounds to simple inorganic compounds under the influence of microorganisms [19-20]. Within the European Union, lubricating oils that enter the environment are subjected to an assessment of the rate of biodegradation using 301 A-F tests developed by the Organization for Economic Cooperation and Development (abr. OECD). The tests determine the potential of the biodegradation and the rate of biodegradation [1, 21]. The assessment of the biodegradability of lubricating oils is complicated, as they are poorly water-soluble. In addition, the composition of the lubricant is diversified, depending on the components and the set of improvers. Therefore, it is necessary to select the appropriate test for the assessment of biodegradability and apply an appropriate sample preparation method [6, 22-23].

Oil meeting the requirements of the OECD 301B test should be biodegraded in more than 60% after 28 days of the test conditions. It is assumed that the total degradation of the remaining part of the analyte will take place. The test results indicate that this assumption is valid only for synthetic and natural lubricating oils. Most of the components derived from crude oil are slowly degraded, as various chemical reactions occur [1, 9].

Known and widely applied biodegradability tests for lubricating oils are expensive and time--consuming. This is why there is a need for development of quick and simple methods for preliminary evaluation of the composition of lubricating oils. Fast methods and simple protocols have been developed for similar applications, e.g. non-expensive identification and group separation of compounds enabling fast screening of oils, fuels and related samples, without extensive and complex samples separation or for the assessment of analytical material's quality [24-26]. In this paper we propose a fast and cheap screening test named as "a drop test". The test is simple to perform and does not require specialized service. The proposed preliminary method for the assessment of the presence of petroleum-based fractions in a sample of lubricating oil is based on the fluorescence phenomenon of petroleum fractions when irradiated with UV light. Petroleum-based oils are mixtures of aliphatic, aromatic, and high molecular weight organic compounds. Such oils contain polycyclic aromatic hydrocarbons (PAHs) that have inherent fluorescence, with emission spectra and optimal excitation and emission wavelengths dependent on the chemical composition of the oil [27-28]. This is why the fluorescence spectroscopy is widely exploited in the petroleum industry, due to e.g. high sensitivity, good diagnostic potential and relatively simple instrumentation [29]. The characteristic fluorescence of the petroleum-based fractions contrary to the lack of fluorescence of commercially available high-quality biodegradable lubricating oils is the basis of the method proposed in this paper. The proposed drop test method is intended for preliminary screening for the presence of a petroleum-based fraction in the oil sample. The presence of these fractions is indicated by blue or light blue fluorescence of the sample in the proposed test conditions. It is worth of note that this test may be performed on-site and it is the first step of a multi-step procedure for determination of a group-type composition of a lubricating oil sample [30-31]. In this paper, the equipment as well as the results of a drop test method for both accepted standard samples and commercially available lubricating oils are presented.

### 2. EXPERIMENTAL

#### 2.1 Materials

In order to find the differences between the samples of lubricating oils and bases of oils of various types based on the fluorescence phenomenon, fifteen samples of oils of different chemical composition were used for the drop tests. These samples are as follows:

- 1. Vegetable oil fresh rapeseed oil (non eruc)
- 2. Vegetable oil sunflower seed oil
- 3. Animal oil fish oil, liquid fraction
- 4. Food processing used oil frit

5. Used oil – oil from fat plants from the process of refining vegetable oil

- 6. Deodorised oil
- 7. PAO synthetic oil (poly-alpha-olefins)
- 8. Synthetic oil commercially available

9. High-quality vegetable oil with enriching additives – commercially available

10. Commercially available biodegradable oil of low quality (mixture of mineral oil and vegetable oil 1:1)

11. Oil of petroleum origin – commercially available

- 12. Technical FAME
- 13. Base oil SAE 30
- 14. Base oil SAE 10
- 15. Brightstock.

Previous investigations in our research team using HPLC method revealed that the oil sample depicted by no 10 is a mixture of petroleum-based and vegetable oil, in proportions about 1:1 by volume.

#### 2.2 Equipment

The authors proposed a simple equipment that allowing for the repeatable comparison of the fluorescence properties of the oil samples. The proposed equipment used to perform the test includes: • Photographic chamber – impermeable to visible light (made of cardboard), dimensions:  $245 \times 105 \times 210$  mm with two holes drilled in the upper base (for the camera with a diameter of 10 mm, for a flashlight – of 30 mm (Fig. 1 and 2),

• Camera (mobile phone: Samsung Galaxy S3, matrix: 8 Mpx, resolution: 3264 × 2448 pxs, sensitivity according to ISO 80-1600, F2.6 lens),

• LED lamp emitting UV light with - 365 nm wavelength – (Producer: MR Chemie),

• Black matt plate made of acrylonitrile – butadiene-styrene copolymer, dimensions: 90 × 50 × 10 mm with 8 holes (diameter of 10 mm and depth equal to 7 mm) (Fig. 3 and 4),

 $\bullet$  100  $\mu I$  automatic pipette, or syringe if necessary,

• Disposable tips with a capacity of 100 μl.

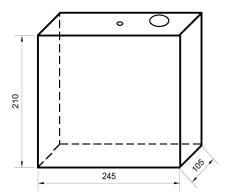


Figure 1 Photographic chamber for drop test performance – dimensions

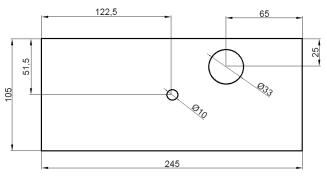


Figure 2 Top view of the photographic chamber to perform the drop test

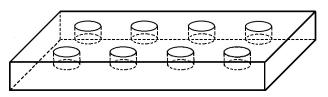
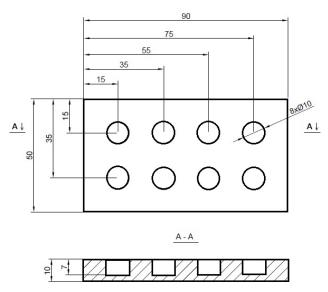


Figure 3 Proposed plate for the drop test performance



**Figure 4** Dimensions of the plate for the drop test performance – cross-section (A-A) and overall plan

#### 2.3 Methods

The investigated oil material is introduced into the hollowed cylinder in the black matte plate (Fig. 1 and 2) in a volume of 20  $\mu$ l. After filling all the reference and test samples onto the plate, the plate is placed in a photographic chamber (Fig. 3 and 4). Then, pictures are taken of the samples irradiated by the ultraviolet light with the excitation wavelength  $\lambda$  = 365 nm, emitted by the LED lamp. For excitation wavelength of 365 nm, fluorescence is observed mainly for petroleum-based oil, however vegetable oils may also reveal UV fluorescence but for different wavelengths [32]. The evaluation of the results is realized by the visual analysis based on the comparison of the intensity or lack of fluorescence of the oil samples in comparison with fixed standards i.e. oils of known composition. The use of a proposed photographic chamber supports repeatable conditions of the measurement i.e. photographic picture is always taken for the same mutual location of oil samples with respect to both the UV light source as well as the camera's lens, which strongly affects the results [33].

#### **3. RESULTS AND DISCUSSION**

The drop test is intended to determine the presence (or absence) of the petroleum-based oil fractions in the investigates oil. The detection limit of the method, (own research, not published yet) is approx. 3% by volume of the mineral-based oils. The test uses the characteristic fluorescence of aromatic hydrocarbons – unsubstituted, aliphatically substituted, alicyclic or monocyclic – which fluorescence is low-intensively blue, to polycyclic – showing intense fluorescence of light-blue color. In the case of the sample containing oil of petroleum origin or other components derived from crude oil in a concentration higher than the limit of detection (abr. LOD) for the described method (in the range of 0.5-9% v/v depending on the type of the oil – own research, not published yet) in the excitation wavelength  $\lambda$  = 365 nm (UV lamp) fluorescence occurs (as light blue or blue fluorescence).

The occurrence of the fluorescence described above informs about the presence of petroleum fraction. The occurrence of blue and dark blue fluorescence indicates a very high probability of the occurrence of a monocyclic structures, i.e. benzene, in the investigated material and/or its aliphatic and alicyclic derivatives. Such oil may contain a very high-refined base oil of petroleum origin, low-volatile alkylate or petroleum oil (i.e. turbine oil, other types of oils subjected to a catalytic hydrotreating process). In the absence of fluorescence during the drop test, it can be concluded that no petroleum-based oils and its derivatives are present or their concentration is lower than in the approx. 9% (v/v) (own research, not published yet). If the oil sample does not show fluorescence, it may preliminary be classified as biodegradable and be allowed for application in forestry operations. On the other hand, its oil base and enriching additives may include vegetable or animal oils as well as mixtures. Such oil may also contain a biodegradable synthetic polyester oil base and other components of this type (most often derivatives of succinic acid), an oil base and poly-alpha-olefin (abr. PAO) type components, or mixtures (currently used in high-quality engine oils).

The content of the hydrocracked or isomerized petroleum-based oil is also not excluded in the drop test. Such oil bases consisting of very stable, usually synthetic compounds of chemical polymers and are similar to poly-isoethylene or poly-isopropylene, but with lower molecular weights, or, to synthetic polyesters and their various mixtures. In this case, no UV light absorption in the excitation wavelength  $\lambda = 360$  nm occurs and no phenomenon of fluorescence occurs.

Synthetic polyesters belong to the group of completely biodegradable organic chemical compounds, although their biodegradability is rel-

atively slow. However, poly-alpha-olefins (abr. PAO) are biodegraded after a very long time. However, there are no objective reasons to discuss the toxic influence to the organisms in this case, as PAOs are completely inert to microorganisms. However, PAO residues in the soil, or on the surface waters, in the form of a film of water-insoluble organic liquid phase, can pose a significant ecological threat. The result of investigations regarding all mentioned samples are presented in the Figure 5. In the case of vegetable, animal and synthetic oils (samples 1, 2, 3, 4, 5, 7, 8, 9 and 12), fluorescence was not observed, which indicates that the given oil sample did not contain any petroleum fraction, or the fraction content was below the LOD of the proposed method. In the case of samples 10, 11, 13, 14 and 15 both, light--blue and blue fluorescence was detected, which indicates the presence of the petroleum oil fraction in the oil composition. In the case of sample 10, the fluorescence was dark blue as it is an oil composed of a mixture of petroleum oil bases, as depicted in point 2.1.

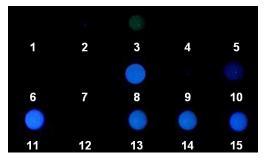


Figure 5 Results of the drop test of 15 oil samples: 1 – Vegetable oil – fresh rapeseed oil (non eruc);
2 – Vegetable oil – sunflower seed oil; 3 – Animal oil – fish oil, liquid fraction; 4 – Food processing used oil – frit;
5 – Used oil – oil from fat plants from the process of refining vegetable oil; 6 – Deodorised oil; 7 – PAO synthetic oil (poly-alpha-olefins); 8 – Synthetic oil – commercially available; 9 – High-quality vegetable oil with enriching additives – commercially available; 10 – A mixture of vegetable and petroleum oil – commercially available; 11 – Oil of petroleum origin – commercially available; 12 – Technical FAME; 13 – Base oil SAE 30; 14 – Base oil SAE 10; 15 – Brightstock

#### 4. CONCLUSIONS

The problem of emission of petroleum-based lubricating oils into the environment is gaining attention. All lubricating oils working in open cutting systems, emitted or hydraulic oils, potentially emitted to the environment should contain only readily biodegradable ingredients in order to eliminate the negative impact on the environment and possibly on human health. The proposed method for evaluating the petroleum oil fraction presence in an oil sample, using "a drop test", may be performed on-site as a preliminary and screening test. A multi-step laboratory investigations of the group-type composition of lubricating oils are however indispensable for precise quantitative analysis of an oil sample composition.

Proposed drop test method and proposed adequate equipment to perform the field test's does not require the need of sample preparation. Additionally, no additional chemicals are used and the test is fast to perform. The test is simple and does not require specialized equipment or installation and both investment and operational costs are low.

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**Conflicts of Interest:** The authors declare no conflict of interest. The founding source had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

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