

Capillary Sensor with Fluorescence Reading of Effects of Diesel and Biodiesel Fuel Degradation in Storage

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Abstract—There are many standards set by national organizations and fuel producers to test and guarantee the quality of diesel fuel and its stability under storage conditions. The diesel fuel stability is related with the fuel composition that evolved to the modern fuels from the historical ones. The stability of modern diesel fuel is mainly due to the reduction of the oxidation processes, the result of the presence of unsaturated components and components with oxygen as organic components and cetane index improvers. The simple characteristic of serious degradation of diesel fuel is the appearance of resins and sediments. Traditional techniques for measuring fuel stability, like the rancimat methods, are relatively complex. On the user side, fast and low-cost sensing of the degradation of diesel and biodiesel fuel is important. The present paper concentrates on the construction of the capillary sensor which enables the examination of the presence of resin and degradation of the most widely used cetane improver (2-ethyl hexyl nitrate) in one arrangement. Results of a development of a sensor working on the principle of fluorescence excited in a disposable capillary cell with high power light emitted diodes are presented. We discuss the principle of the sensor's operation, the construction of the sensor, and the experimental results of testing diesel fuels instability.

Keywords—biodiesel fuel stability; diesel fuel instability; cetane index improvers; capillary sensor; LED excited fluorescence.

I. INTRODUCTION

A. Modern diesel fuels

Classical petro-diesel fuels are made from stable components of straight run distillate products of crude oil (alkanes) with an addition of improvers [1]. Biodiesel fuel is a mixture of classical petro-diesel fuel and bio-components.

It was historically postulated that petro-diesel fuel may be kept in storage for prolonged periods. But modern petro-diesel fuels include stable components (alkanes) and cracked material which contains olefins (alkenes) characterized by a double bond chemical. Alkenes are more reactive than

alkanes. Bio-diesel components include significant amounts of fatty acids esters that are also characterized by the presence of double bonds. Oxidation of hydrocarbons with double bonds may lead to waxy solids or gums emerge [2].

One of the most important diesel fuel quality parameters is the ignition quality. The ignition quality depends on the molecular composition of the fuel and is characterized by the ignition delay time, which is the time between the start of injection and the start of combustion. The minimum cetane number of diesel fuel differs by region, for example in Europe the minimum value is 51, but in some regions of United States it is 40. In Europe, the major problem of the refineries is obtaining fuel with high cetane number.

There are two methods to increase the cetane number. The first method is to reduce the content of aromatic and/or double bonds of fuel by hydrogenation - this is usually not done because of a high cost. The second method is using cetane improving additives - this method is preferred due to its low cost [3]. The alkyl nitrates and peroxides cetane improvers have been in use since 1921. Cetane improvers decompose rapidly and form free radicals when exposed to temperatures above 100°C. As these radicals increase the rate of main fuel components decomposition, the ignition delay is negatively affected. The use of cetane improvers in diesel fuel of high quality increases the engine durability [4].

The 2-ethyl hexyl nitrate (2-EHN) is the most popular cetane index additive. Its production volume in 2014 was about 100,000 tons. The ASTM D 4046 standard test method is used for determining the amount of alkyl nitrate added to diesel fuel to judge compliance with specifications covering alkyl nitrates. Unimolecular decomposition of 2-EHN creates nitrogen dioxide (NO₂), which further reacts with available hydrogen atoms to produce hydroxyl radicals. This effect is greatest at temperature and density conditions, matching the start-up conditions in a diesel engine, and becomes negligible at the highest temperature-density conditions found in standard engine working conditions [7]. On the other hand, the addition of 2-EHN to fuel improves the spray formation in diesel engines [8]. Therefore, 2-EHN performs well physically and chemically during the combustion of

fuel, but can also decompose slowly even at temperatures below 100°C [9]. Thus, 2-EHN can be an additional reason of the fuel instability, introducing temperature instability and being the initiator and promotor of oxidation of fuel.

The results of diesel fuel instability are sediments and gum formation. The bio-diesel fuel has been found to be more prone to oxidation than the vegetable oils or petrodiesel fuel. The effects of bio-diesel fuel instability may be the presence in the fuel tank of a wide variety of alcohols, aldehydes, peroxides, polymers, as well as the previously mentioned insoluble gum and sediments. The results of polymer and soluble waxes formation is the increase in the diesel fuel viscosity [5]. The changes in molecular composition of fuel affect its ignition characteristics. Therefore, fuel instability is of concern when the modern diesel fuel is stored over an extended period of time. Diesel fuel stability may be affected by a large number of parameters which can be categorized by oxidation, thermal and storage conditions [2]. The storage conditions can be described as exposure to air and/or light at the environment temperature. Oxidative properties of biodiesel are commonly tested according to the EN 14112 standard as rancimat and Iodine values, but several other methods have also been used in the analysis of oxidation state of oil and biodiesel [6].

The visible effects of serious diesel fuel instability may be described as fuel darkening and sediments presence, as presented in Figure 1, on which the degradation is the effect of two years fuels samples storage in transparent glass vessels exposed to ambient light at room temperature. The sediments and darkening are present in cases of premium (Cetane Number = 59.6) and standard fuel (Cetane Number = 54.1) bought at a fuel station, while the own-mixed fuel does not seem affected by instability. All fuels above the formed sediment layer are transparent. The own-mixed fuel at the beginning of the examination was characterized by a worse fit for use parameters than the fuels bought at the fuel station. However, the mixed fuel was prepared with clear and fresh components with the maximum permissible concentration of antioxidants and the smallest concentration of 2-EHN (Cetane Number = 51.2). Some fuels prepared in such way show visual effects of instability as a conversion into pale and cloudy liquid. The visible effects of four weeks exposition to ambient light of premium fuel are presented in Figure 2. At first, the fuel instability process leads to fuel fading and cloudiness, next the dark phase forms at vessel's bottom and separates from the clear fuel. Hence simple a fuel color evaluation is not sufficient for fuel instability discrimination.

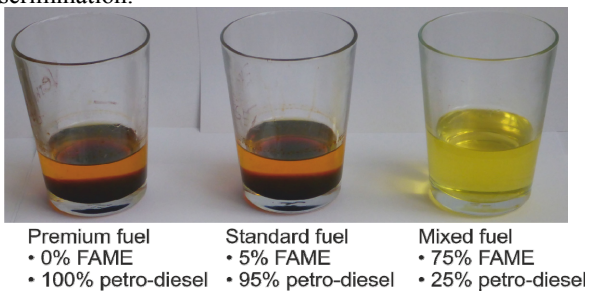


Figure 1. Visible effects of fuel instability.

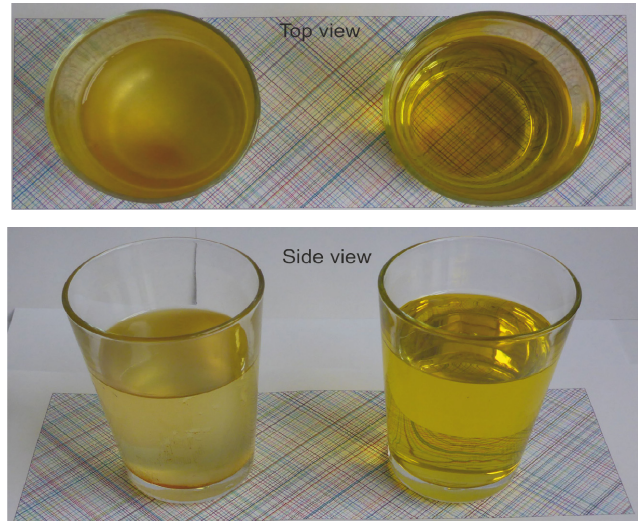


Figure 2. Visible effects of premium fuel cloudiness after exposition to ambient light for four weeks. Fuel sample in the left side container has been exposed to light. The sample in right side container has been kept in dark conditions.

B. Optical methods and sensors for diesel fuel testing

These are a set of spectrophotometric method used for diesel fuel testing. The examinations of light absorption, scattering, fluorescence, and chemiluminescence are performed. Some of them are executed in a combustion chamber as evidence of the combustion process [10][11]. Many of them are performed on processed diesel fuel sample and include a tedious liquid to liquid extraction with organic solvents. Biodiesel and diesel fuels show native fluorescence [12], but with very limited excitation efficiency. However, the diesel fuel may contain many fluorophores. The task of the determination of the fuel's condition is further complicated by the fact that the fluorophores have different characteristic excitation and emission ranges in different samples. Despite this, ultraviolet fluorescence spectroscopy methods were developed to identify oils by the differences of their aromatic compositions. For this purpose, advanced measurement technology as synchronous fluorescence spectroscopy and time-resolved fluorescence have been developed. They demonstrate an improved potential for classification of fuels than the classic fluorescence spectroscopy [13][14]. But presently available spectroscopic methods are still characterized with some disadvantages. Mathematical models proposed to convert spectroscopic examination results into diesel fuel set of parameters work only with known and examined previously components [15]. Also, plastic disposable cuvettes are not fit for examination as they are slowly dissolved by diesel fuel. Quartz cuvettes used in spectroscopic examination require precise maintenance between measurements. The light sources are costly when as laser or lamp excitation is used. Despite these drawbacks, dedicated components for spectroscopic sensors are under development [16][17].

Alternative methods to spectroscopy, which include an examination of diesel fuel with local heating in a closed

capillary vessel and the examination of the dynamical rise of fuel in a inclined capillary, show a potential of a fast fuel fit for use determination [18-22]. Also, for these methods dedicated components have been developed [23][24].

But in the routine daily work, the transport business still demands the development of new rapid and low-cost sensors for reliable determination of fuel fit for use or fuel degradation below usefulness.

The rest of this paper is organized as follows. Section II describes the idea of a multiparametric sensor for diesel fuel instability effects testing. Section III describes the sensor construction including sensor head with optrode as well as optoelectronics system set-up. Section IV addresses the experimental results of the fluorescent signal analysis. Section V goes into short conclusions.

II. IDEA OF A MULTIPARAMETRIC SENSOR FOR DIESEL FUEL INSTABILITY EFFECTS TESTING

The idea of the sensor was inspired by the need to come up with a low-cost measurement method of a set of diesel fuel instability effects in one system that would require the minimum of automated mechanical elements.

The aimed-at set of diesel fuel parameters includes the initial state of oxidation and the serious instability effects. Both effects are characterized by a transparent liquid state. As the fuel is transparent the both fraction may be mixed resulting in a visibly uniform color of the fuel. The cloudiness of fuel is considered as an intermediate state and can be easily determined by human observation or with white light scattering measurements.

The initial state of oxidation may be connected with 2-EHN degradation that should lead to a reduction of its concentration. The concentration of 2-EHN may be evaluated with the direct analysis of fluorescence signal excited with 366nm wavelength light, selected from the output of the xenon discharge light source of 150W power with a 3nm slit [15]. Due to the relatively low power of the signal, the detection is made with a photon multiplying tube. The exit signal with a characteristic shape of double-hump is in the range from 380nm to 450nm.

The serious effect of fuel instability is the presence in it of soluble and insoluble gums. Insoluble gums create sediments in tanks, while soluble gums can degrade the injection system. The laser induced fluorescence, with argon-ion laser operating at 488 nm with the constant power of 100mW used as the excitation source, is presently the proven measurement method [25]. Due to high laser power, the detection is performed with a photodiode array. The emitted signals range from 510nm to 750nm with a maximum at 570nm. For the real fuels, there was always observed some background fluorescence, but its intensity was usually low compared to that of the degraded fuel.

Both the fluorescent measurements were performed in our work in standard fluorescence cuvettes, 10mm x 10mm x 45mm. A cuvette was mounted in place with a shielded holder, which also minimized scattered light from the cuvette input and output faces. The aperture for the fluorescence measurement was 7mm in the central portion of the cuvette.

III. SENSOR CONSTRUCTION

A. Sensor head

The set of parameters of interest may be observed using the fluorescence excited with 488nm and 366nm radiation in the sample of liquid positioned in a capillary vessel using optical fibers as probes (Figure 3).

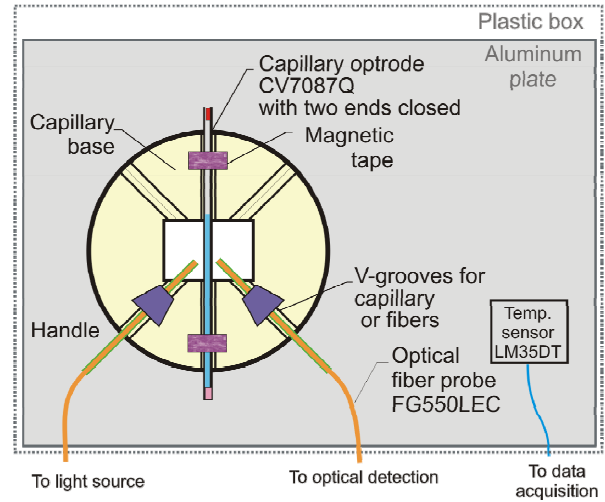


Figure 3. Scheme of the capillary sensor head.

The use of capillary reduces significantly costs of measurement and in two ways.

First, the measurement of degraded diesel fuel that is characterized by the presence of adhesive gums required the use of disposable cuvettes or a specific cleaning and washing process. Moreover, disposable plastic cuvettes are affected by diesel and biodiesel fuels. Therefore, the capillary optrode CV7087Q is used here as a replaceable and disposable vessel that is of considerably lower cost than a classical fluorescence cuvette.

When a capillary optrode CV7087Q is considered, the approximated fluorescent aperture is 0.7mm. This fluorescent aperture is 10 times lower than when a classical cuvette is used. Therefore, the optical power used for excitation of fluorescence in the proposed head may be significantly lower (100 times) than when a commercial spectrophotometer is used. In the case of the laser induced fluorescence and a spectroscopic cuvette, the excitation power is about 100mW. Therefore, in the presented case the high power light emitting diodes can be used as their minimal specified powers are 3mW from M365FP1 working at 365nm range, and 1.5mW from M490F2 working at 490nm.

The FG550LEC step-index multimode fiber was used as the light probe, as it enables positioning of the optical axis of the probes and the optrode in one plane. Moreover, the fiber is characterized by a 550µm diameter of the core, which allows the effective coupling with high power light emitting diodes, and a low numerical aperture, that reduces the fluorescent aperture of the head. This fiber is characterized by high attenuation below 400nm, but in the sections used of 0.5m long, the attenuation is acceptable.

The head was also equipped with a temperature sensor made with LM35DT circuits.

B. Optoelectronic system set-up.

The sensor’s head consists of two functional blocks: the head proper and the optoelectronic set-up, shown in Figure 4.

The optoelectronic set-up is divided into two parts, the light source and light detector, both connected to a personal computer equipped with spectra acquisition software and system script designed for measurement automation.

As the light source, two fiber-coupled LEDs were used. The high power LEDs selected were Thorlabs M490F2, with the 490nm dominant wavelength and M365F1 with the 365nm dominant wavelength. The diodes were connected to DC2100 controllers operating in the constant current mode.

The diodes were coupled into the optical fiber switch of our own design, presented in [26].

The head was connected to two spectrometers Maya 2000pro with an optical signal divider. This enables simultaneous monitoring of the signal of light emitted diode and of the excited fluorescence. The fluorescence signal is measured in a 6-fibers arm, while the excitation signal uses a 1-fiber arm.

The system element causing the highest light damping was the DMC1-02 monochromator, the attenuation of which is about 1:1000 at analyzed wavelengths.

IV. EXPERIMENTAL RESULTS

In this section are presented the experimental procedures and the results of examination of different diesel and biodiesel fuels.

The measurement procedure consists of a few steps. First, the background signal of for the empty optrode is measured, then the optrode is directly filled from the desired layer of the fuel in the tank and the outer optrode wall is

wiped. Next, the signal of fluorescence is measured. Last, the spectra are calculated as the difference between the fluorescence and the background signals.

A. Diesel and biodiesel fuels used for examination

The operation of the sensor was examined with fuels that are bought at a petrol station and with mixtures prepared from components. These fuels were not aged. Reference fuels were stored at room temperature in glass tanks in closed dark containers exposed to natural light. The fuel samples that are subject to degradation were stored in transparent glass tanks also exposed the same light. The exposition was made on a windowsill with direct sun view on from south direction in Warsaw. The short term aging of 4 weeks duration was performed in winter conditions of sunlight, for visible effects see Figure 2. Fuels aged for two years were exposed to full year cycles, visible effects can be observed in Figure 1.

The four different premium diesel fuels were selected for tests. One was a clear mixture of alkanes with no additives (CN=49.8), and the three others were mixtures of petrodiesel with additives including 2EHN – one commercial with no bio-component (CN=59.6) and two with a bio-component, one with 1% of fatty acids methyl esters (FAME) (CN=59.1) and second with 7% of hydrogenated vegetable oil (HVO) (CN=59.2). The standard commercial diesel fuel includes 7% of FAME (CN=54.1).

B. Boundary examinations

Boundary examinations included measurements of fluorescence of fresh fuels and two years aged fuels. In all mentioned in this section cases, the outer walls of the capillaries were cleaned after the test. Spectra of fresh fuels, scanned with a 1-second time frame of scanning, and excited with 366nm wavelength, were presented in Figure 5.

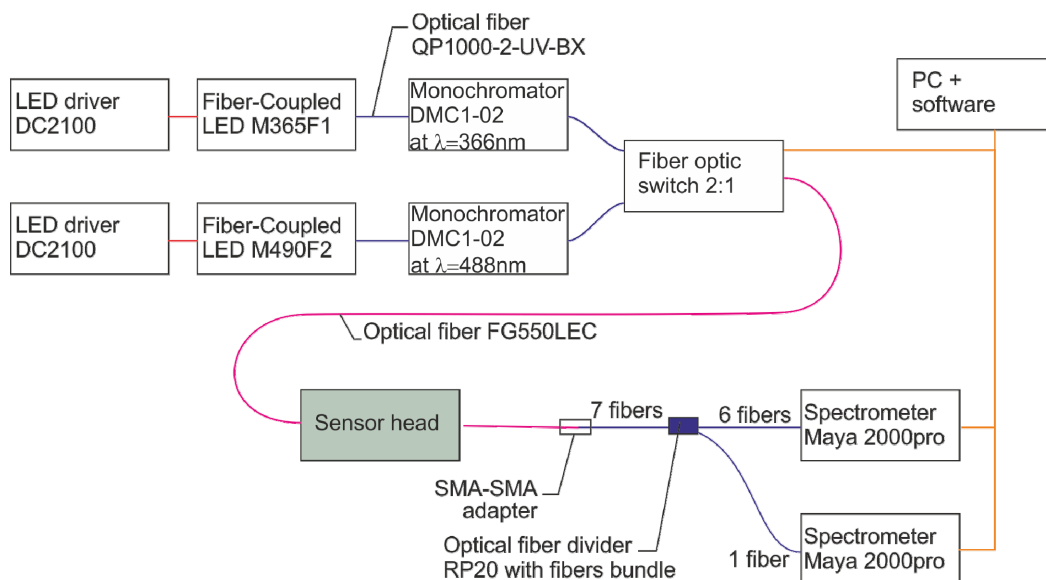


Figure 4. Sensor system scheme.

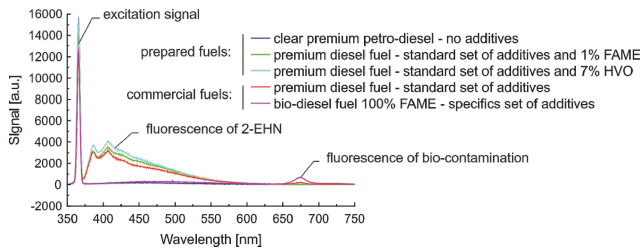


Figure 5. Spectra of fresh fuels excited with 366nm wavelength.

As stated previously, the excitation and fluorescence signals differ significantly, but the signal received from 2-EHN is relatively high. The 2-EHN signal is not present in the 100% FAME bio-diesel fuel and in the clear premium petro-diesel, which can be expected as EHN is not an additive for such fuels. Except for the 2-EHN, the fluorescence signal of bio-contaminants is observed for commercial fuels, while the bio-diesel fuel is characterized with a higher signal than the premium petrodiesel. The biodiesel shows a small fluorescence in the range of 450-700nm, due to aromatic components. Such fluorescence is not observed in the clear premium petro-diesel, which as previously stated is prepared from alkanes.

In the case of gum presence examination with excitation at 488nm, the power of excitation and fluorescence signals differ so much that their observation requires using both arms of photo-detection with different times of scanning. In the presented case, the fluorescence was measured with 10-second scanning and the excitation was measured with 5-second scanning.

Spectra of fuels stored for two years on windowsill measured with 488nm wavelength excitation are presented in Figure 6. The excitation signals are the same in both cases and do not affect the fluorescence characteristics. The presence of gums is evident, as a fluorescence signal in the range from 530nm to 700nm is present.

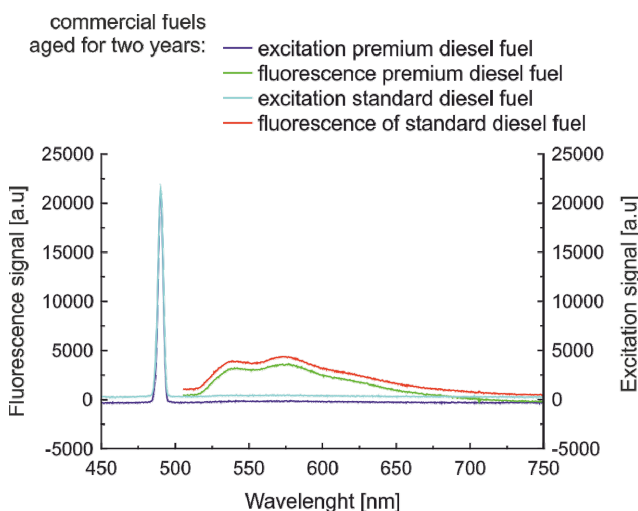


Figure 6. Spectra of commercial fuels stored two years on windowsill excited with 488nm wavelength.

In both samples, the signal is almost the same, which means that the concentration of gums in both cases is the same. That at first view may seem strange. The vertical shift of signals can be the result of a difference between radii of used capillaries, as a small variation of this parameter is normal. In such case, the examination of the excitation signal at the receiver arm enables a simple mathematical correction.

Examinations of fresh fuels do not show any fluorescence signals comparable to results presented in Figure 6.

C. Examination of premium commercial fuel exposed for light for few weeks

We examined premium commercial diesel fuel stored at windowsill for up to five weeks and compared the results to storage of the same fuel in dark conditions. The fuel stored in dark condition does not show any visible changes or any changes when measured using the method described above. The change of appearance of fuel exposed to light can be described as follow: after one week the sample color started fading, after two weeks the sample becomes cloudy, and after five weeks there is an emergence of sludge. The sludge color is dark brown, similar to that of buckwheat honey, and it is sticky to the touch. The cleaning of outer capillary walls after probing of fuel exposed for five weeks need some attention, as sticky areas are palpable. Thus one can conclude that some reactions occurred in the sample under storage. It is interesting, that the examination of fluorescence excited at 488nm did not show any fluorescence signals.

The examination results of fluorescence excited at 366nm when the fuel was stored at a windowsill are presented in Figure 7. The observed fluorescence signals show significant changes of shape and power. The signal of bio-contamination disappeared. The signal power has not monotonically reduced. The initial spectrum maxima (point 4 in Figure 7.) after consecutive weeks of exposition shifted toward lower wavelength which was stable for some time (wavelength point 3 in Figure 7.). Local signal minima wavelength (point 2 in Figure 7.) were stable for some time, and then minima disappeared. The same effect applied to the first local maxima (wavelength point 1 in Figure 7).

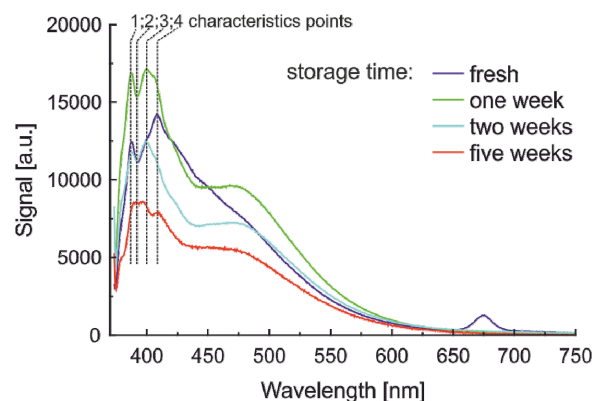


Figure 7. Measurement data of fluorescence excited at 366nm for premium commercial fuel stored at windowsill.

Therefore, in the described sensor device, the difference of signals S(4)-S(2) values measured in point 4 and in point 2 may be a good measure of fuel degradation. The wavelength at the point 4 was 408nm, and at the point 2 was 392nm. The results of examinations are presented in Table 1, including results for fresh fuels, fuels degraded to five weeks as well as seriously degraded fuels with gum presence the fluorescence of which was presented in Figure 6.

TABLE I. FUELS CLASSIFICATION

Fuel type	Exposition for light [weeks]	Visual report	Difference of signals; S(4)-S(2) [a.u.]
Premium - no additives	0	transparent, light yellow	0
Premium commercial	0	transparent, yellow	2585
Premium commercial	1	transparent, light yellow	847
Premium commercial	2	turbid, light yellow	-18
Premium commercial	5	turbid, light yellow, sludge stains	-523
Premium commercial	104	transparent, buckwheat honey, sediment	-563
Standard commercial	104	transparent, buckwheat honey, sediment	-346

The proposed measure is lower than zero for degraded fuels with are characterized by the presence of turbidity and first appearance of gum sludge. The difference is greater than zero for transparent fluids. It should be noted that premium fuels, that do not shows fluorescence, show zero difference the mentioned signals. The measured signal at the excitation wavelength (366nm) changes significantly, as can be expected for fuel transparency changes.

V. CONCLUSIONS

We proposed a sensor of diesel fuel degradation with fluorescence examination in a capillary optrode. The analysis of the measured signals of fresh and degraded diesel and biodiesel fuels showed the relationship of the degradation with measured and processed signals.

On the base of data collected in the experiments we can set the parameters determining the initial diesel fuel degradation level as the difference of signals registered at 408nm and 392nm for fuel sample excitation and 366nm. The seriously degraded diesel fuel is characterized by fluorescence signals in the range of 530nm to 700nm when the excitation signal is at 488nm.

We conclude that the proposed instrumentation may be in future a valuable added module to the capillary sensor system for diesel fuel fit-for- use examinations.

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