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The assessment of surface acoustic wave sensing for testing fuel dilution of lubricating oils

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A B S T R A C T

This article presents an assessment of the accuracy and repeatability of oil dilution measuring apparatus. The device uses surface acoustic wave sensing (SAW), and the measurement method meets the ASTM D8004 standard. Since the described in the ASTM standard definitions of accuracy and repeatability are not easy to compare with definitions in the ISO standards, a comparative experiment was settled. Mixtures of the SAE 30 and SAE 40 grades lubricating oil with diesel oil in a high-precision concentration of 0, 1, 2, 5, and 10 % m/m were tested. The calculated root mean square error (RMSE) of measurement results against the expected value, was $RMSE_{SAE\ 30} = 0.57$ % m/m and $RMSE_{SAE\ 40} = 1.33$ % m/m. Additionally, the method based on the flash point was tested to compare estimated dilutions (SAW vs. flashpoint). The results show that the flash point gives a better estimation than SAW. The device can be used for industrial tests.

1. Introduction

When operating combustion engines, there is a risk of contamination of the circulating lubricating oil with fuel. This threat especially applies to crosshead engines, where the crankcase is separated from the combustion chambers only by pistons and piston rings [1]. The sources of contamination may include, in particular, excessive wear [2] or damage to piston rings and blow-offs of unburnt fuel from the combustion chambers into the crankcase, as well as leaks and improperly operating fuel equipment and/or installations for draining fuel leaks from injection pumps and injectors [3]. Wear debris may lead to further damages of the engine parts [4,5].

Fuel contaminating the lubricating oil leads to the deterioration of lubrication conditions and, thus, increased wear [6] of the oil-lubricated elements and reduced thermal and antioxidant stability of the oil (i.e., accelerated oil aging) [7]. Deterioration of the lubrication conditions results in accelerated wear of the bearings, cylinder liners, pistons, and piston rings [8] and the risk of premature ignition, which applies to

Abbreviations: A, accuracy of the apparatus; $A(\overline{C_M})$, accuracy of the concentration measurement; API, American Petroleum Institute; ASTM, American Society for Testing and Materials; C, exact (expected) mass concentration of diesel oil in the lubricating oil; CD, CF, API quality classes of lubricating oils for self-ignition engines; C_i, i-th measured value of the mass concentration of diesel oil in the lubricating oil; $\overline{C_M}$, mean value of measured concentration; d, measurement scale resolution; DMX, marine distillate fuels category according to the ISO 8217:2017 standard; ESR, electron spin resonance; FDM, fuel dilution meter; FTIR, Fourier-transform infrared spectroscopy; GC, gas chromatography; GC–MS, gas chromatography and mass spectrometry; GUM, guide to the expression of uncertainty in measurement; i, measurement number; ISO, International Organization for Standardization; JPI, Japan Petroleum Institute; K, coveragefactor; m_f, mass of diesel oil; m_o, mass of lubricating oil; *MSE*, mean square error; n, number of measurements; NMR, nuclear magnetic resonance; p, confidence level; R, repeatability of apparatus; $R(\overline{C_M})$, repeatability of the concentration measurement; *RMSE*, root mean square error; *RSD*, relative standard deviation; s, experimental standard deviation of the mean; $s(\overline{C_M})$, standard deviation of the measured concentration; SAE, Society of Automotive Engineers; SAE 30, SAE 40, viscosity grades of lubricating oils according to the SAE J300-2021 standard; SAW, surface acoustic wave; t, Student's distribution coefficient; t_{FP}, flash point temperature; $u(\delta)$, uncertainty of the balance indication error from the calibration certificate; $u(r)$, composite uncertainty of repeatability of scale indications; u_A, type A standard uncertainty of measurement; u_B, type B standard uncertainty of measurement; $u_B(C)$, type B standard uncertainty of the mass fraction C measurement; u_C, combined standard measurement uncertainty; u_m, uncertainty of the determined mass of fuel or lubricating oil; u_s, type B standard uncertainty of mass indication with a laboratory scale; UV, ultraviolet spectrometry; u_w, type B standard uncertainty for determining the diesel oil content in the mixture; X, average of the two test results; δ, error of scale indications from the calibration certificate; δ_D, measurement resolution; σ, standard deviation of measurement.

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spark ignition engines [9]. According to the literature [10], the fuel content in oil exceeding 2–5 % m/m (mass percentage concentration) requires immediate action due to a significant risk of engine damage. According to other sources, when the concentration of diesel oil in lubricating oil exceeds 8 % m/m during engine operation, there is an additional risk of explosion of oil vapors in the crankcase [11].

Accounting for the above, in the case of marine, aircraft, traction, and industrial combustion piston engines, periodic and routine monitoring of the physicochemical properties of the lubricating oils is carried out aimed at, among others, assessing the amount and type of impurities contained in the lubricating oil [12]. Fig. 1 presents the most important methods for evaluation of lubricating oil dilution with fuel. The SAW method is marked in grey. All standards mentioned in Fig. 1 are listed in the references [13–19].

The most used methods to evaluate contamination of lubricating oils with fuels are periodically used oil viscosity analyses and measurement of the oil's ignition temperature. These methods are relatively cheap and generally available in most industrial laboratories [21–23]. Moreover, there are oil analysis systems that measure viscosity and density simultaneously, and can distinguish between contamination and dilution to a certain degree [24–26]. These tests can be supported by using particular models [20,27] and wear simulations [28].

Contamination of the lubricating oil with fuel influences lubricating oil viscosity. It may decrease when the lubricating oil is diluted with distillation fuel or increase due to the dilution of the lubricating oil with residual energy. To draw conclusions regarding the dilution of oil with power, it is necessary to know the type of fuel used by a given engine, from which the lubricating oil is subject to assessment.

This method does not provide unambiguous results because the change in viscosity values may be related to many, sometimes very different, factors, including [29,30]:

- diluting the oil with other oil, grease, or coolant,
- oxidation and thermal decomposition processes of lubricating oil components,
- depletion of additives that modify the properties of the oil,
- accumulation of wear products of interacting machine elements, combustion products, and air pollutants in the oil,
- infection of the oil with protozoa, bacteria, fungi, and viruses.

Bearing the above in mind, the use of oil viscosity as a measure of the degree of dilution of the lubricating oil with fuel can only be used as a supplementary indicator determined alongside the oil's ignition temperature or, alternatively, determining the distillation range of a given oil.

The drop in the ignition temperature of the lubricating oil in the engine during its use shows that there is a very high probability that the lubricating oil has been contaminated with fuel. The method can, therefore, be used to assess the dilution of lubricating oil with various types of fuels. Despite the advantages of this method, it must be considered that the phenomenon of lowering the ignition temperature is also possible in situations other than diluting the lubricating oil with fuel, e.g., because of the mixing of different types of oils or as a result of oil aging. Moreover, there are cases where, despite diluting the lubricating oil with fuel, no significant drop in the ignition temperature was recorded [30]. This fact results from the lack of a guarantee that the oil-fuel mixture remains stable and from the possibility of evaporation of light hydrocarbon fractions (the most volatile fuel components) from the lubricating oil-fuel mixture, the presence of which normally contributes to lowering the ignition temperature of the liquids containing them [31].

Standardized methods based on measuring the oil evaporation range (i.e., the distillation range) include a method using lubricating oil

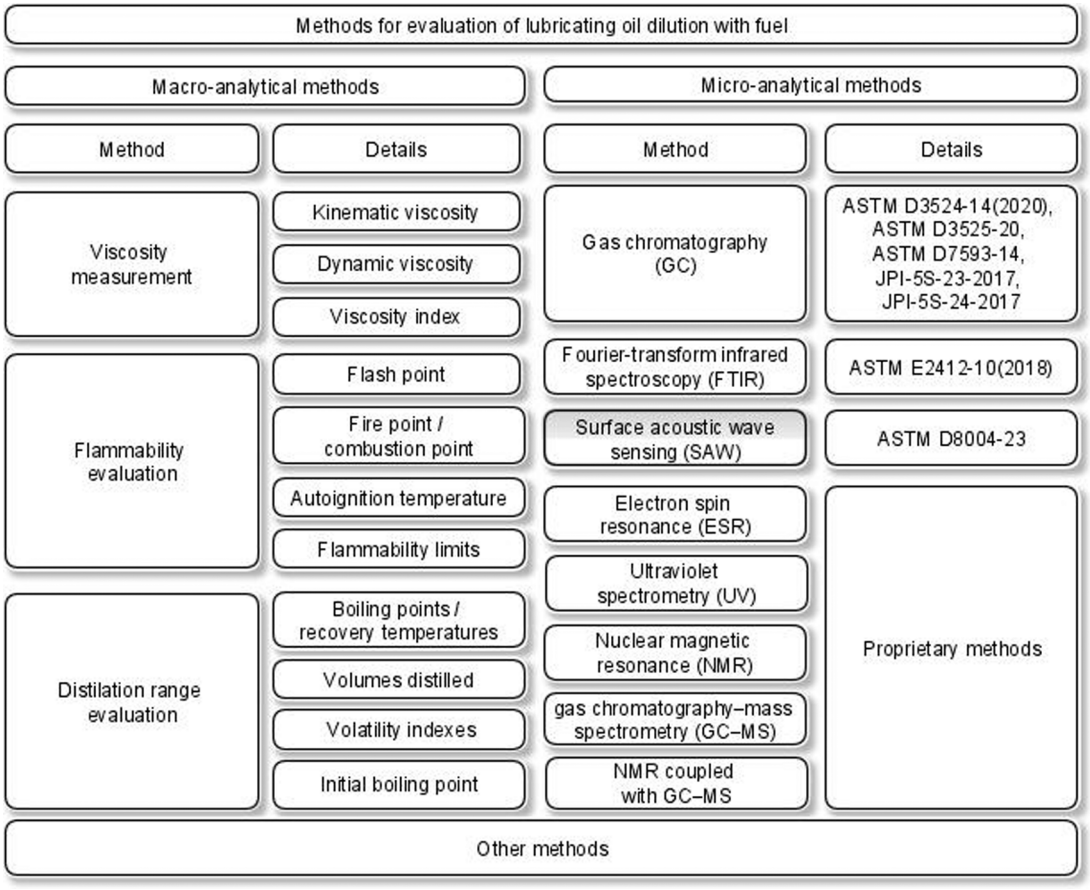

Fig. 1. The most important methods for evaluation of lubricating oil with fuel (modified from [20]).

distillation to assess its dilution with gasoline, described in the ASTM D322-97 (2016) standard [32]. These methods, like the measurement of the ignition temperature, do not provide clear results.

Accounting for the limitations of the above-mentioned macro-analytical methods, specialized methods have been developed [31], including gas chromatography (GC) [33] described in ASTM D3524-14 (2020) [13] and JPI-5S-23–2017 [16] (lubricating oil contaminated with diesel oil), ASTM D3525-20 [14] and JPI-5S-24–2017 [17] (lubricating oil contaminated with gasoline), and D7593-14 [15] (lubricating oil contaminated with diesel oil, biodiesel fuel, or gasoline). A concise description of GC-based methods is provided in the literature on the subject [34–37].

FTIR spectrometry can also be used to detect fuel contamination of lubricating oil [38], characterized by relatively user-friendly equipment and the ability to quickly measure the degree of dilution of lubricating oil with fuel. However, this method does not accurately differentiate the base material from which specific hydrocarbons originate [39]. There are devices of this type on the market which are dedicated for lubricating oil analyses [40,41].

Both GC and FTIR require the use of very expensive equipment, and in practice, their use is only possible in laboratory conditions (i.e., large stationary devices). An alternative to these methods is a procedure for determining the level of dilution of lubricating oil with distillation fuel, developed and popularized in the last decade [42] using a surface acoustic wave (SAW) sensor [43,44]. This method is presented in ASTM D8004 (previously ASTM D8004-15 [45], currently ASTM D8004-23 [19]). The advantages of this method include the availability of portable measuring devices, which allow its use in operational conditions, the speed of measurement, and the fact that tiny samples of the tested oil (0.5 ml) are sufficient to perform the measurements.

Since devices of this type are relatively expensive (approximately 25 thousand USD + VAT [46]), they are not widely used. Bearing in mind the advantages of this measurement method, the authors undertake a study to determine its suitability for scientific and industrial applications by carrying out an experiment based on testing a blind sample of lubricating oils diluted with diesel oil at given concentrations, which are not known to the person performing the test during the measurement. The ASTM D8004 standard refers to the measurement range from 0.1 % m/m to 10 % m/m of diesel oil content in the lubricating oil, and the measurements are also carried out in this range [47,48].

As the authors did not find any scientific papers tackling the applicability of the SAW method and the ASTM D8004 standard does not reveal the construction of the apparatus and its operation details, the experiment presented in this paper was done. This article presents the results of an experiment that assesses the accuracy and repeatability of measurements obtained by the SAW method implemented in the Spectro FDM Q6001 device.

2. Materials and methods

2.1. Reference samples

To verify the indications of the tested device, a set of reference samples was prepared using two popular grades of the SAE 30 and SAE 40 grades engine lubricating oil in accordance with the SAE J300-2021 standard [49], mixed with distillation diesel oil meeting the requirements of the DMX category (ISO-F-DMX) in accordance with the ISO 8217:2017 standard [50]. Both lubricating oils meet the requirements of the API CD/CF quality class (Series III) [51]. Reference samples were mixed at nominal fuel-in-oil concentrations of 0, 1, 2, 5, 10 % m/m for each type of lubricating oil. Each time, samples with a nominal weight of 200 g were prepared. A RADWAG WPs 510/C/2 precision laboratory scale (from Radom, Poland) was used to prepare the samples. The scale has a valid calibration certificate, and before each measurement, the repeatability of the measurement is checked using a standard weight. The basic balance data necessary to determine the standard uncertainty of the type B mass measurement are summarized in Table 1.

The mixture preparation procedure was as follows:

- A clean glass vessel was placed on the scale, and the scale was tared.
- The mass of the pure lubricating oil m_o was measured using a precise laboratory pipette.
- The scale was tared.
- The fuel mass m_f was measured using a precise laboratory pipette.
- The resulting mixture was stirred using a magnetic stirrer for 15 min.

The single preparation of the standard samples makes it impossible to determine the type A uncertainty of the mixture composition. To determine the type B standard uncertainty of the mass share of fuel in the mixture, the uncertainties of mass indication by a laboratory scale were found. The four most important uncertainty components were considered (Table 1). An analysis of the uncertainty of the mass indication by the scale was carried out according to the recommendations of the Guide to the Expression of Uncertainty in Measurement [52], i.e., by using the expression:

$$u_s = \sqrt{\left(\frac{d}{2\sqrt{3}}\right)^2 + \left(\frac{\delta}{\sqrt{3}}\right)^2 + \left(\frac{u(\delta)}{2}\right)^2 + u^2(r)} \tag{1}$$

where u_s is the B-type standard uncertainty of mass indications for a laboratory scale d is the scale unit, δ is the error of the balance indications from the calibration certificate $u(\delta)$ is the uncertainty of the balance indication error from the calibration certificate $u(r)$ is the composite uncertainty of the repeatability scale indications determined as the standard deviation from ten measurements of the standard mass (200 g) corrected with the Student's t-coefficient for 9 degrees of freedom and a confidence level $p = 68$ %. This accounts for the uncertainty of the standard mass.

Since the measured mass of fuel and oil was (each time) the difference between the mass of the liquid and the tare of the scale, it was decided that the uncertainty budget for mass measurement should also include the tare uncertainty of the scale. As a result, the liquid mass uncertainty was determined by the following:

$$u_m = \sqrt{(u_s)^2 + (u_s)^2} \tag{2}$$

where u_m denotes the uncertainty of the determined mass of fuel or lubricating oil.

The mass fraction C (% m/m) of the fuel in the lubricating oil of the reference sample is determined according to:

$$C = \frac{m_f}{m_f + m_o} \bullet 100\% \tag{3}$$

where m_o represents the mass of the lubricating oil (in units of g) and m_f is the mass of the diesel oil (g).

The partial differential equation (4), is derived from equation (3) to determine the type B standard uncertainty of the mixture, namely:

$$u_B(C) = \sqrt{\left(\frac{\partial C}{\partial m_f} \bullet u_m\right)^2 + \left(\frac{\partial C}{\partial m_o} \bullet u_m\right)^2} \bullet 100\% \tag{4}$$

Table 1
Basic data of the RADWAG WPs 510/C/2 laboratory scale for determining the measurement uncertainty.

Parameter	Value
Elementary scale d	1 mg
Scale indication error δ from the calibration certificate for a mass of 400 g	2 mg
Expanded uncertainty ($k = 2$) of the error of scale indications from the calibration certificate $u(\delta)$	1 mg
Combined uncertainty of repeatability of balance readings $u(r)$	0.8 mg

Where $u_B(C)$ (% m/m) is the type B standard uncertainty of the mass fraction C.

The solving for Eq. (4) is:

$$u_B(C) = \sqrt{\left(\frac{1}{m_f^2 + 2m_f + 1} \bullet u_m\right)^2 + \left(\frac{-1}{m_o^2 + 2m_o + 1} \bullet u_m\right)^2} \bullet 100\% \quad (5)$$

The most important values that describe the reference samples of the oil-fuel mixture, enabling verification of the results, are summarized in Table 2.

The values and their uncertainties are the same for fuel mixtures with each of the two types of tested oil: SAE 30 and SAE 40.

2.2. The SAW device

Despite a long-term query among the source materials, the authors did not find devices other than those indicated in the standard and, therefore, no other manufacturers use the SAW method in assessing the degree of dilution of lubricating oil with diesel oil. The mentioned ASTM D8004 standard does not provide details of the operation of the device, or the algorithms implemented in it. The only research report known to the public which presents the precision of the ASTM method was published by the ASTM itself as document RR:D02-2047 (Approved: May 01, 2023) [53] and it is restricted by copyright. Therefore, this method can be treated as proprietary and can be assumed that it is used only in devices offered by only one manufacturer. Currently, there are two FDM 6000 Series devices available on the market, marked as Spectro FDM 6000 and Spectro FDM 6001, differing in the number of stored calibrations, which in the case of the former is 1 and is 3 for the latter [54,55].

In this experiment, measurements were made using the FDM 6001 device. The basic technical data of this device, declared by the manufacturer, are presented in Table 3. During the measurements, the ambient temperature was 21.5 °C, the relative humidity was in the range of 55–60 %, and the barometric pressure was 1016 hPa.

To obtain the most reliable results possible during the experiment, the measurements were carried out in a blind trial, i.e., the operator of the measuring device did not know the exact content of diesel oil in individual samples, except for samples containing 5 % m/m diesel oil in lubricating oil, which was used to calibrate the device in accordance with the standard procedure intended to perform tests for a specific type of lubricating oil and diesel oil. All other samples were marked with code names. For each tested mixture of diesel oil and lubricating oils, a total of 10 measurements were made, which enabled a statistical analysis of the obtained measurement results.

Table 2
Parameters for the mixture of lubricating oils and diesel fuel with the uncertainties.

Parameter	A mixture of fuel and oil				
Sample no. i	1	2	3	4	5
Mass fraction of fuel in lubricating oil C (% m/m)	0.000	1.000	2.000	5.000	10.000
Oil mass, m_o (g)	200.000	198.000	196.000	190.000	180.000
Fuel mass, m_f (g)	0.000	2.000	4.000	10.000	20.000
Type B standard uncertainty of the determined mass, u_m (g)	0.0023				
Type B standard uncertainty of the mass fraction of fuel in the mixture $u_B(C)$ (% m/m)	ON	0.026	0.010	0.002	0.001

Table 3
Declared technical data of the Spectro FDM 6001 device (based on ref. [54]).

Parameter	Description
Application	Mineral and synthetic lubricants used in liquid-fueled engines
Accuracy	≤± 0.2 % fuel dilution in range 0.2–2 % ≤± 10 % of measurement in range 2–15 %
Repeatability	≤± 5 % *RSD* of measurement + 0.2 % fuel dilution
Measurement resolution, δ_D	0.1 % fuel dilution
Sample volume	0.5 ml
Ambient operating temperature	5 °C is 35 °C
Relative humidity	0 to 90 %, non-condensing
Ambient altitude	up to 2,000 m (barometric pressure ≥ ~800 hPa)

3. Results and discussion

The arithmetic mean, $\overline{C_M}$, is determined and its A-type standard uncertainty for each mixture is based on the 10 independent measurements with an FDM device. Then, the experimental standard deviation of the mean is determined from:

$$s(\overline{C_M}) = \frac{s(C_M)}{\sqrt{n}} \quad (6)$$

where $s(C_M)$ is the experimental standard deviation of the set of samples and n is the number of samples.

Due to the small number of samples n, it was decided to correct the determined experimental standard deviation of the mean $s\overline{C_M}$, t-coefficient of the Student distribution for 9 degrees of freedom and a confidence level of $p = 68$ %. Finally, to determine the value of the standard uncertainty of type A, the influence of the standard uncertainty of type B of the mass fraction of fuel in the mixture was also considered, i.e., $u_B(C)$ from Table 2. Calculations were made using the following expression:

$$u_A(\overline{C_M}) = \sqrt{(t \bullet s(\overline{C_M}))^2 + u_B{}^2(C)} \quad (7)$$

where $u_A(\overline{C_M})$ is the type A standard uncertainty of the tested sample, t is the Student's distribution coefficient in which $t = 1.06$, $s(\overline{C_M})$ is the experimental standard deviation of the mean calculated for the tested mixture, and $u_B(C)$ is the type B standard uncertainty determined for reference mixture C (Table 2).

Based on the data from the manufacturer's specifications (Table 3), the standard uncertainty type B is also determined for each average value of measurement of mixture samples using the FDM device. For this purpose, the accuracy values are determined in accordance with the specifications of the device. The variable $A(\overline{C_M})$ for a medium-sized $\overline{C_M}$ is found from the relationships:

$$A(\overline{C_M}) = \begin{cases} 0.2 for \overline{C_M} \le 2 \\ 0.05 \bullet \overline{k} for \overline{C_M} > 2 \end{cases} \quad (8)$$

And the repeatability $R(\overline{C_M})$ from the following dependency:

$$R(\overline{C_M}) = 0.1 \bullet s(\overline{C_M}) + 0.2. \quad (9)$$

Since there is no information about the type of the accuracy and repeatability statistical distribution, a uniform rectangular distribution is assumed in both cases. The resolution of the results, displayed via a FDM device, is also considered. The standard uncertainty type B is found as:

$$u_B(\overline{C_M}) = \sqrt{\frac{A^2(\overline{C_M})}{3} + \frac{R^2(\overline{C_M})}{3} + \frac{\delta_D^2}{12}} \quad (10)$$

where $A(\overline{C_M})$ is the accuracy of the average result $\overline{C_M}$ of the tested samples from the apparatus specification, $R(\overline{C_M})$ is the repeatability of

the average result $\overline{C_M}$ of the tested samples from the apparatus specification, and δ_D is the apparatus measurement resolution.

Ultimately, the combined standard uncertainty $u_C(\overline{C_M})$ is found from the following:

$$u_C(\overline{C_M}) = \sqrt{u_A{}^2(\overline{C_M}) + u_B{}^2(\overline{C_M})} \tag{11}$$

The test results are summarized in Tables 4 and 5 for each type of lubricating oil separately in order to maintain the accuracy of the calculations and, in accordance with the recommendations of the GUM guide [52] for further calculations, it was decided to retain four significant digits in the presentation of the partial results.

The measurement results of the mass concentration of the diesel oil in lubricating oil are graphically presented in Fig. 2. The implementation of the individual measurements and average values are shown in the form of scatter charts. The designated combined standard uncertainty limits are also presented, i.e., $u_C(\overline{C_M})$.

Measurements for the mixtures of SAE 30 oil show a slightly improved convergence with the characteristic line of the reference mixtures than for a mixture with SAE 40 oil. However, in the case of both mixtures, it can be seen that the averaged results of the FDM measurement (and even the combined standard uncertainty limits in more than 50 % of the measurement range) lie significantly outside the line of expected results (black line). To quantify the measurement error, the root mean square error (RMSE) values are determined by comparing the averaged measurement results using the FDM device with the standard values according to the following:

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(C_i - \overline{C_{Mi}})^2} \tag{12}$$

where C_i is the mass fraction of the fuel in the mixture of the i-th reference sample, $\overline{C_{Mi}}$ is the average measurement result of the i-th reference sample using the FDM device, and n is the number of samples tested.

Table 4
Results of the measurement of the mixtures with SAE30 lubricating oil using an FDM apparatus.

	Measured percentage *of* C_M fuels in the mixture (% m/m)				
Nominal concentration of the sample	0	1	2	5	10
Pensky-Martens flash point (°C)	180	166	160	134	110
1	0.20	1.70	1.60	3.70	8.40
2	0.10	1.40	2.00	4.20	9.00
3	0.00	1.30	1.90	4.30	8.90
4	0.00	1.50	2.10	4.60	9.50
5	0.10	1.70	1.90	3.50	9.60
6	0.00	1.50	2.10	4.50	9.70
7	0.00	1.40	2.80	4.30	9.40
8	0.00	1.40	1.90	4.30	8.90
9	0.10	1.30	1.50	4.20	8.90
10	0.00	1.20	2.10	3.90	9.40
Mean, $\overline{C_M}$	0.05	1.44	1.99	4.15	9.17
Experimental standard deviation of the $s(\overline{C_M})$ mean	0.0224	0.0521	0.1111	0.1098	0.1300
Student's t-coefficient for a small sample	$t = 1.06$				
Sample standard deviation corrected, $t \bullet s(\overline{C_M})$	0.0237	0.0552	0.1178	0.1164	0.1378
Type A standard uncertainty, $u_A(\overline{C_M})$	0.03	0.07	0.12	0.12	0.14
Type B standard uncertainty, $u_B(\overline{C_M})$	0.17	0.17	0.18	0.28	0.55
Combined standard uncertainty, $u_C(\overline{C_M})$	0.18	0.19	0.22	0.31	0.57

Table 5
Results of the measurement of the mixtures with SAE40 lunricating oil using an FDM apparatus.

	Percentage of fuel C_M in the mixture (% m/m)				
Nominal concentration of the sample	0	1	2	5	10
Pensky-Martens flash point (°C)	178	160	150	132	100
1	0.3	1.2	2.3	5.7	13
2	0	1	2.1	5.4	13.6
3	0.6	0.7	2.5	5.9	12.4
4	0.2	0.6	2.1	5.6	12.7
5	0	0.9	2.1	5.8	13
6	0	0.7	2.5	5.5	12.8
7	0	0.5	2.4	5.8	12.7
8	0.1	0.7	3	5.8	13.30
9	0.2	0.9	2.8	6	11.90
10	0.4	0.2	2.9	5.4	12.90
Mean, $\overline{C_M}$	0.18	0.74	2.47	5.69	12.83
Experimental standard deviation of the mean, $s(\overline{C_M})$	0.0647	0.0885	0.1066	0.0658	0.1477
Student's t-coefficient for a small sample	$t = 1.6$				
Sample standard deviation corrected, $t \bullet s(\overline{C_M})$	0.0686	0.0938	0.113	0.0697	0.1566
Type A standard uncertainty, $u_A(\overline{C_M})$	0.07	0.10	0.12	0.07	0.16
Type B standard uncertainty, $u_B(\overline{C_M})$	0.17	0.18	0.20	0.36	0.76
Combined standard uncertainty, $u_C(\overline{C_M})$	0.19	0.21	0.24	0.37	0.78

For the mixture of fuel with SAE 30 oil, the result is $RMSE_{SAE30} = 0.57$, while for SAE 40 oil, the result is much higher and amounts to $RMSE_{SAE40} = 1.33$. This may mean that the average error of the measurement result in the measuring range of the FDM device is 0.57 % of the fuel dilution for a mixture with SAE 30 oil and 1.33 % of the fuel dilution for SAE 40 oil. The device's readings vary significantly depending on the base oil. The difference is 0.76 % of the fuel dilution, which means that the relative difference is over 5 % of the measurement range.

Table 6 presents a comparison between the ISO standard uncertainties calculated according to equation (11) with the declared manufacturer accuracy (8) and repeatability (9). For SAE 30 samples the average result of 10 measurements $\overline{C_M}$ is off the expected result C both according to the ISO standard uncertainty $u_C(\overline{C_M})$ and the declared accuracy in case of nominal 1 % m/m and 5 % m/m samples. Very high offset, more than $u_C(\overline{C_M})$ limit, is observed also for 10 % m/m sample, however, it is within the declared accuracy limit. In case of SAE 40 samples the average $\overline{C_M}$ is off the expected result C both according to the ISO standard uncertainty $u_C(\overline{C_M})$ and the declared accuracy in case of all 0 % m/m concentration samples.

For lower measurement range of 0–2 % m/m dilution, the ISO uncertainties and the declared accuracy are nearly the same. However, the limits for higher measurement range (2–10 % m/m dilution) differ significantly because of a different calculation methodology. Generally, in that range the declared accuracy limits are from 35 % to 64 % wider than the standard uncertainties (Table 6). Nevertheless, the results obtained by the FDM apparatus differ by more than both limits (ISO and declared) from the reference values in all cases but one of SAE 40 base oil mixtures. The results are more accurate for samples based on the SAE 30 oil.

The actual repeatability of results does not meet the declared values. The repeatability calculated according to the manufacturer's declaration is from 0.20 % for higher concentration samples up to 0.27 % for clean oil sample. The range of the ten samples results for each concentration (Tables 4 and 5) is from 0.2 % to 1.3 % for SAE 30 samples and from 0.6 to 1.7 % for SAE 40 samples.

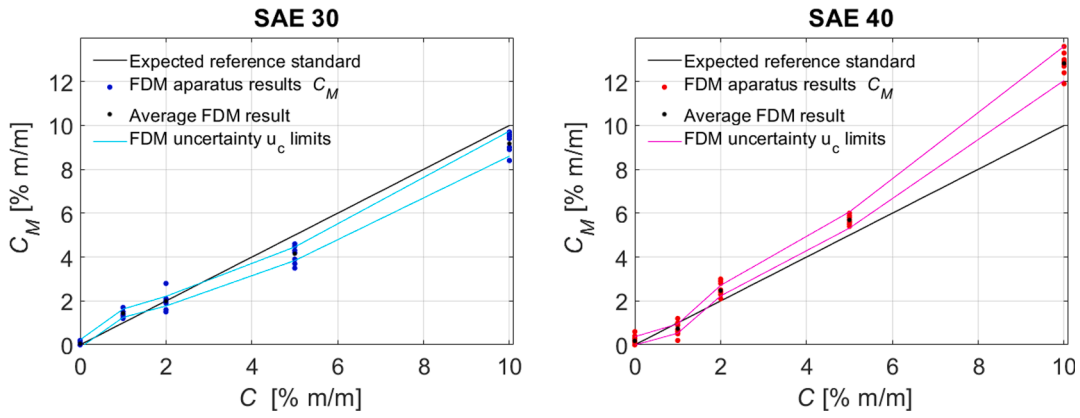

Fig. 2. Comparison of the measurement results using an FDM apparatus with the reference characteristics of standard samples.

Table 6
A comparison of the manufacturer's declared accuracies with ISO uncertainties.

Parameter	A mixture of fuel and oil				
Sample no., i	1	2	3	4	5
Mass fraction of fuel in oil reference sample C (% m/m)	0.000	1.000	2.000	5.000	10.000
Measured SAE 30 samples arithmetic mean, $\overline{C_M}$ (% m/m)	0.05	1.44	1.99	4.15	9.17
Measured SAE 30 samples combined standard uncertainty, $u_C(\overline{C_M})$ (% m/m)	0.21	0.19	0.22	0.31	0.57
Measured SAE 30 samples accuracy (declared) (% m/m)	0.20	0.20	0.21	0.42	0.92
Measured SAE 30 samples repeatability (declared) (% m/m)	0.27	0.21	0.21	0.21	0.20
Measured SAE 40 samples Arithmetic mean, $\overline{C_M}$ (% m/m)	0.18	0.74	2.47	5.69	12.83
Measured SAE 40 samples combined standard uncertainty, $u_C(\overline{C_M})$ (% m/m)	0.22	0.21	0.23	0.36	0.78
Measured SAE 40 samples accuracy (declared) (% m/m)	0.20	0.20	0.25	0.57	1.28
Measured SAE 40 samples repeatability (declared) (% m/m)	0.26	0.22	0.21	0.21	0.21

Even though the 5 % m/m samples were used for calibration, the average results for that concentration are outside the standard uncertainty and declared accuracy values in both cases.

Significant differences are observed for the results obtained for mixtures based on two lubricating oil grades. During this experiment we did not study the reason of those differences. We assumed that the SAW device was sensitive to chemical content appearing locally in lubricating oil. This hypothesis may be further investigated.

Supplementary, a flash point analysis of the mixture was performed, which is a parameter commonly used in industrial quality analysis of oils. Due to the availability, the closed-cup Pensky-Martens method was chosen. The measurement results are summarized in Tables 4 and 5 and presented in Fig. 3.

Fig. 3 presents second-degree curves that approximate the obtained results. The determined RMSE values in relation to the approximating curves are significantly lower for the flash point parameter compared to the analogous results obtained with the FDM apparatus. This value is 0.29 for the SAE 30 lubricating oil and 0.42 for the SAE 40 lubricating oil.

4. Conclusions

The test results presented in this article concern samples prepared using pure oil. Due to the variable and complex chemical composition of engine oils during operation, in the case of testing used oils, the results may differ from those presented in this article. This is due to the natural processes of oil aging, depletion of enriching additives, and oil contamination.

The values of the standard measurement uncertainties determined using the FDM apparatus are relatively high. As the diesel oil content in the tested mixture increases, the discrepancy between the measurements made on the same oil sample also increases. Moreover, it can be stated that the measurement result is strongly dependent on the type of lubricating oil. That makes the FDM apparatus measurement and conclusion much more difficult if the type of lubricating oil in the tested

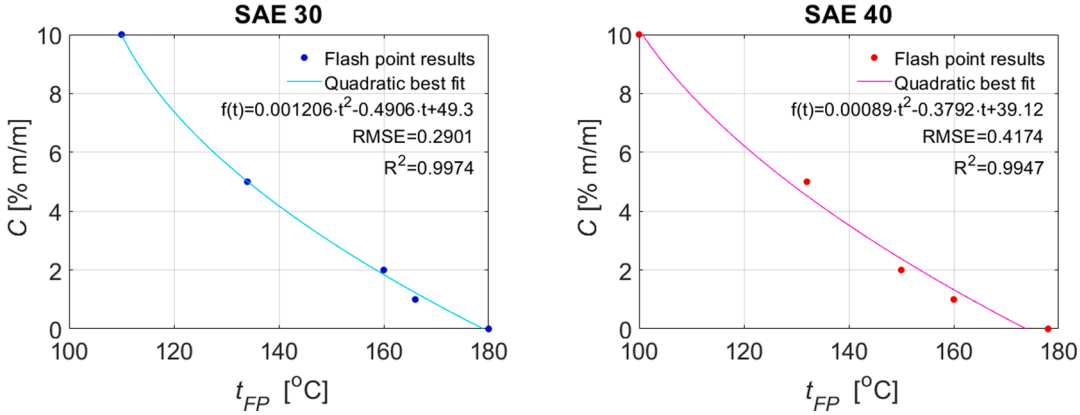

Fig. 3. Comparison of the relationship between the flash point and the composition of the oil-fuel mixture for the SAE 30 and SAE 40 base oils.

mixture is not known or if it is not possible to prepare a calibration sample based on the same base lubricating oil. This situation is practically common when testing used oils because the properties of such oils are usually unknown. The highest repeatability of measurements is achieved in the range of low concentrations of diesel oil in lubricating oil, i.e., 0–2 % m/m. However, in this concentration range, the measurement uncertainty is still high.

Taking this into account, a conclusion can be drawn that the device may not be sufficient for precise analyses but can be suitable for a quick, preliminary and rather qualitative than quantitative assessment of contamination. However, similar or better results can be obtained by flash point temperature testing. An indication for the use of an FDM device may be the cost of the test, including the purchase of the device, and its availability. An alternative is to use the flash point temperature, which is a widely used parameter, and the devices used to determine it are much cheaper than FDM.

Data Availability Statement: All the data is available in the paper and the dataset Chybowski. L. *Lube oil - diesel oil mixes - dataset*; 2022. Ver. 3. https://doi.org/10.17632/scbx3h2bmf.3. The dataset is available at https://data.mendeley.com/datasets/scbx3h2bmf/3 [56].

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CRediT authorship contribution statement

Leszek Chybowski: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Przemysław Kowalak:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis. **Marcin Szczepanek:** Writing – review & editing, Writing – original draft, Validation, Investigation, Formal analysis. **Przemysław Jóźwiak:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis. **Paweł Danisiewicz:** Writing – review & editing, Writing – original draft, Validation, Investigation, Formal analysis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

I have shared the link to my data https://doi.org/10.17632/scbx3h2bmf.3

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