

## Soot contamination of engine oil – the case of a small turbocharged spark-ignition engine

The paper presents the results of thermogravimetric tests of engine oil used in a small turbocharged spark-ignition engine. The main observation from the research was a significant soot contamination of engine oil, that appears even at its low mileage. This indicates that also in the case of port fuel injection spark-ignition engine, high particulate matter emissions may occur. A rapid soot contamination of the oil in this engine indicates that the oil change interval should be shortened.

Key words: engine oil, thermogravimetric analysis, soot, particulate matter, spark-ignition engine

### 1. Introduction

Internal combustion engines will be primary drive source for many years, probably decades. Thus, it is clearly important to perform research and development to provide a better understanding of the fundamental processes affecting engine efficiency and the production of undesirable emissions [9].

The combustion of real fuels in a real engine is always imperfect. This results in the presence of incomplete combustion products in the exhaust gas. One of these components is particulate matter (PM), consisting of inter alia soot. Particulates belong to the most harmful components of exhaust gases [11].

Particulate matter emissions are primarily considered from the perspective of diesel engines. The process of reducing particulate emissions in engine exhaust has been going on for many years. For many years as well, PM emissions from passenger cars with spark-ignition engines were not limited at all. Currently, the PM limit applies only to direct injection gasoline engines (GDI) and is the same as for diesel engines (Table 1).

Table 1. European emission standards on PM emissions for passenger cars (category M), g/km [2]

	Effective from (first registration)	Diesel	Spark-ignition
Euro 1	January 1993	0.14	–
Euro 2	January 1997	0.08	–
Euro 3	January 2001	0.05	–
Euro 4	January 2006	0.025	–
Euro 5a	January 2011	0.005	0.005 <sup>*)</sup>
Euro 5b	January 2013	0.0045	0.0045 <sup>*)</sup>
Euro 6b	January 2015	0.0045	0.0045 <sup>*)</sup>
Euro 6c	January 2018	0.0045	0.0045 <sup>*)</sup>
Euro 6d-Temp	January 2019	0.0045	0.0045 <sup>*)</sup>
Euro 6d	January 2021	0.0045	0.0045 <sup>*)</sup>

<sup>\*)</sup> Applies only to vehicles with direct injection engines

Port fuel injection (PFI) gasoline engines are considered to emit little particulate matter. Presented by Liang et al. [7] tests of two Euro 4 cars (the same model) with GDI and PFI engines showed that over the NEDC cycle the PM emission for a car with an MPI engine was 3 mg/km, and for this with a GDI engine as much as 14 mg/km.

Most of the particulate matter (soot) formed in the engine's combustion chamber is removed with the exhaust gases, but some of it is also absorbed in the engine oil. It is easy to notice that as engine oil is used, it changes its color to darker, which is caused inter alia by increase in soot content. There is no doubt that the amount of soot in oil depends both on the intensity of soot formation in the engine's combustion chamber, as well as on the mileage of the oil. Thus, the soot content of engine oil at a given mileage is to some extent an indicator of the tendency of a given engine to smoke. The soot content increases with increasing oil mileage and can reach level of several percent. At this concentration, soot significantly changes (worsens) the lubricating properties of engine oil.

Increasing the soot content in the oil increases engine wear. Results described in [4] show that soot interacts with oil additives reducing the oil's anti-wear properties possibly by abrasive wear mechanism. Ball-on-flat-disk type tests also revealed the increased wear due to the presence of soot. Scanning electron microscope studies of wear scar diameters suggest that soot is abrasive.

Similar research results are presented in [5]. Cumulative wear was more for samples with soot contamination. A negative impact of soot on wear was already observed at 2% soot in engine oil. Oil samples with low dispersant and high zinc dithiophosphate (ZDP) content performed better than samples having high dispersant and low ZDP concentrations at high soot levels. The reason behind this could be the fact that high dispersant levels together with soot contamination results in lubricant thickening resulting in increased wear.

By worsening the lubrication conditions, soot contained in the oil also increases fuel consumption. Although it is not a very significant impact, it cannot be ignored at present when seeking maximum fuel savings and striving to minimize CO<sub>2</sub> emissions. According to [8] low friction lubricants provide fuel savings of around 0.5%.

The aim of the tests described in this article was to check the soot content in the engine oil of a small turbocharged spark ignition engine used in a mini city car. The soot content in this oil will allow concluding on the tendency of this engine to emit particulate matter. It will also indicate if the original oil change interval of 15,000 km is appropriate taking into account oil contamination.

## 2. Test apparatus and procedures

The tests were carried out on oil used in a small turbocharged spark-ignition engine driving mini city car Smart. Some technical data and key features of the Smart's engine are shown in Table 2. The tested oil was grade: 5W-30 ACEA A3/B4 API SL. It was oil designed for engines operating in start-stop mode. Fresh oil and used oils collected after 5000 km and 8000 km after oil change were used in the tests. Table 3 shows some key parameters of the fresh oil.

Table 2. Technical data/key features of SMART's engine

Engine code	M160 E07 LA
Engine type	Spark ignition, in-line three
Displacement	0.698 dm <sup>3</sup>
Bore/stroke	66.5 mm/67 mm
Compression ratio	9.5
Maximum power	46 kW@5250 rpm
Maximum torque	95 Nm@2000–4000 rpm
Valvetrain	Single overhead camshaft, 2 valves per cylinder
Injection system	Multipoint sequential injection, max injection pressure 4.8 bar
Ignition	Dual ignition – 2 spark plugs per cylinder
Forced induction	Turbocharging with intercooling
Charge air control	Map-guided
Exhaust aftertreatment	Three-way catalytic converter
Emissions standard	Euro 4
Engine oil capacity	3 dm <sup>3</sup>

Table 3. Characteristics of the fresh engine oil [1]

Name	Method	Units	Value
Density @ 15°C	ASTM D4052	g/ml	0.855
Kinematic viscosity @100°C	ASTM D445	mm <sup>2</sup> /s	12.0
Viscosity, CCS –30°C (5W)	ASTM D5293	mPa·s	6400
Kinematic viscosity @40°C	ASTM D445	mm <sup>2</sup> /s	73
Viscosity index	ASTM D2270	–	161
Pour point	ASTM D97	°C	–43
Flash point, PMCC	ASTM D93	°C	201
Ash, sulphated	ASTM D874	% (m/m)	1.3
Grade	5W-30, ACEA A3/B4, API SL		

Thermogravimetric characteristics of oils were determined using a PerkinElmer thermogravimetric analyzer TGA 8000. Thermogravimetric analyzer (abbreviation: TGA) measures the change in mass of the sample as it is heated, cooled, or held at a constant temperature in a controlled atmosphere. Table 4 shows the characteristics of TGA 8000 and Fig. 1 shows the picture of TGA 8000. Figure 2 presents a sample of the used engine oil just before the start of the test, placed on the autosampler of the TGA 8000 analyzer (visible black oil color).

Thermogravimetric characteristics of the tested oils were determined according to the temperature program based on the temperature program described in the ASTM D5967 standard. ASTM D5967 is a test method which covers engine test procedures for evaluating diesel engine performance characteristics including viscosity and soot concentrations. Annex A4 is a recommendation on how to measure soot in engine oils. Details of the thermogravimetric analyzer working conditions during the tests are presented in Table 5. Pyris software version 13.3.1 was used to analyze the obtained results.

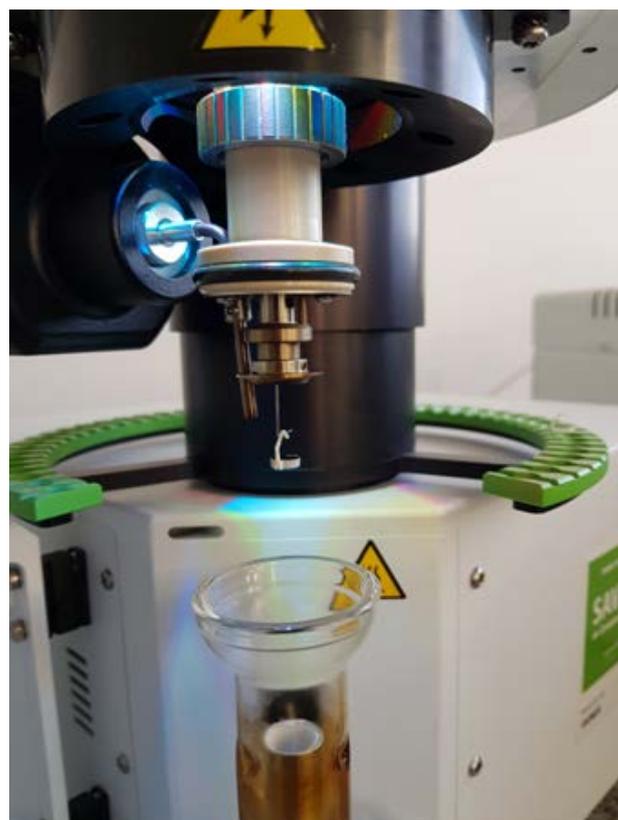


Fig. 1. PerkinElmer thermogravimetric analyzer TGA 8000



Fig. 2. Sample of used engine oil placed on the autosampler of TGA 8000 analyzer

Table 4. Specifications of TGA 8000 thermogravimetric analyzer used in the research

Design		A vertical design with a high sensitivity balance and quick response furnace. The balance is located above the furnace and is thermally isolated from it. A precision hang-down wire is suspended from the balance down into the furnace. At the end of the hang-down wire is the sample pan. The sample pan's position is reproducible.
Balance	Sensitivity	0.1 µg
	Capacity	1300 mg
	Accuracy	better than 0.02%
	Precision	0.01%
Temperature	Furnace	Low mass (< 10 g); Platinum heating element with ceramic protective coating; resistant to inert and oxidative gas over the full temperature range.
	Range	-20°C to 1200°C
	Scan rates	0.1°C/min to 500°C/min
	Precision	±1°C
Cooling	Method	Forced air cooled with an external fan
	Cycle time	1100°C to 50°C < 13 min
Sample Pans		Platinum or ceramic (60 µL)
Atmosphere	Sample environment	Static or dynamic, including nitrogen, argon, helium, carbon dioxide, air, oxygen, or other inert or reactive gases. Analyses done at normal or reduced pressures.
	Gas control	Balance purge (mass-flow controlled); Sample purge (switch between 2 gases; mass-flow controlled); Reactive purge.
	Gas mixing	Up to 3 gases
	Vacuum	10 <sup>-5</sup> Torr
Autosampler		48-position

Table 5. TGA experimental conditions

Instrument conditions	ASTM D5967 Annex A4 [10]	Conditions applied in this research
Temperature program	1. Hold for 1 min at 50°C 2. Heat from 50°C to 550°C at 100°C/min 3. Hold for 1 min at 550°C 4. Heat from 550°C to 650°C at 100°C/min 5. Heat from 650°C to 750°C at 100°C/min 6. Hold for 5 min at 750°C	1. Hold for 4 min at 50°C 2. Heat from 50°C to 550°C at 50°C/min 3. Hold for 2 min at 550°C 4. Heat from 550°C to 650°C at 10 °C/min 5. Heat from 650°C to 750°C at 10°C/min 6. Hold for 15 min at 750°C
Pan type	Platinum	Ceramic
Balance purge	40 ml/min	60 ml/min
Sample purge	N <sub>2</sub> , 30 ml/min for step 1 to 4 O <sub>2</sub> , 30 ml/min for step 5 and 6	N <sub>2</sub> , 40 ml/min for step 1 to 4 air, 40 ml/min for step 5 and 6
Sample quantity	Around 10 mg	Around 20 mg

### 3. Test results and discussion

The thermogram for fresh oil is shown in Fig. 3. Fig. 4, in turn, shows comparative thermograms of fresh oil and used oils – after 5 and 8 thousand kilometers. As can be seen from this comparison, the thermogram curves are similar in all cases, however, the distillation of fresh oil takes place in the narrowest temperature range (the curve is steeper). This confirms the correctness of the measurements carried out. For used oils, it can be seen that the visible weight loss during the thermogravimetric test starts at a lower temperature. This indicates that as oil mileage increases, its fuel contamination increases. At the same time, it is noted that the used oils contain more substances with high boiling points. These substances are the result of oil decomposition at high temperatures in the engine. Fresh oil contains practically no components with a boiling point higher than 450°C.

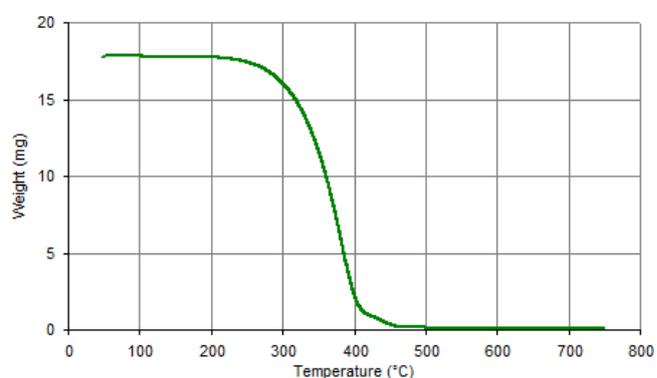


Fig. 3. Thermogram of fresh engine oil obtained during the temperature program presented in the Table 5

The main aim of thermogravimetric tests was to determine the soot content of fresh and used engine oils. Looking at the engine oil thermogram – the first weight loss that occurs as the sample is heated is the evaporative loss of the lubricating oil and any other volatile materials present. All that remains will be soot and other solid residues present in the sample. Switching the purge gas from nitrogen to oxygen or air will remove any soot present in the sample. The calculation of soot content was performed for the weight loss that commences after switching over the purge gas from nitrogen to oxygen or air at 650°C up to the temperature where constant residue was obtained around 750°C [3].

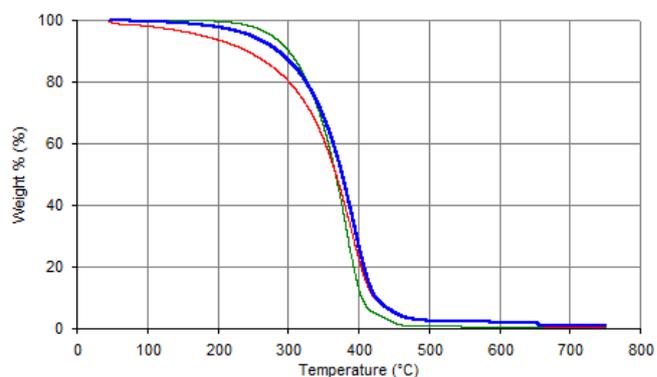


Fig. 4. Comparison of thermograms of fresh (green curve) and used oils (blue curve – mileage of 5000 km and red – 8000 km)

Figures 5–7 show enlarged parts of the thermograms for fresh and used oils, as well as the decrease in sample mass under conditions of oxidizing atmosphere. This weight loss corresponds to the soot content in engine oil. As can be seen in Fig. 4, fresh oil does not contain any soot at all. Engine oil after 5000 km already contained about 1% (m/m) soot, and after 8000 km about 1.5% (m/m).

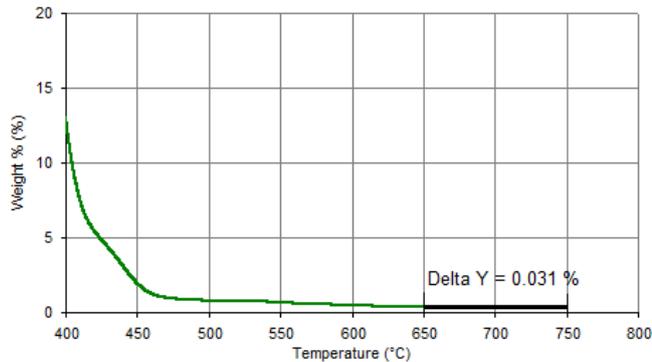


Fig. 5. Thermogram showing percentage soot content in the fresh engine oil sample

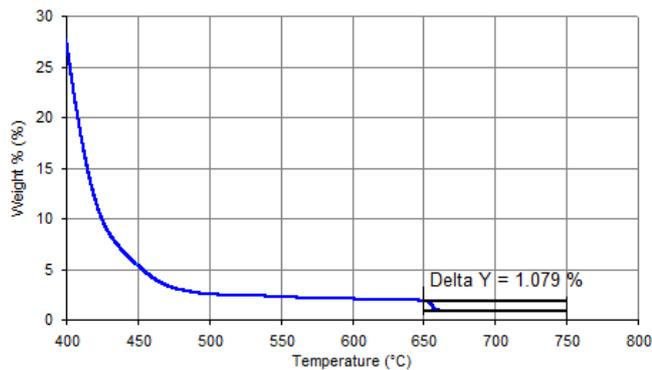


Fig. 6. Thermogram showing percentage soot content in the used engine oil sample (oil mileage: 5000 km)

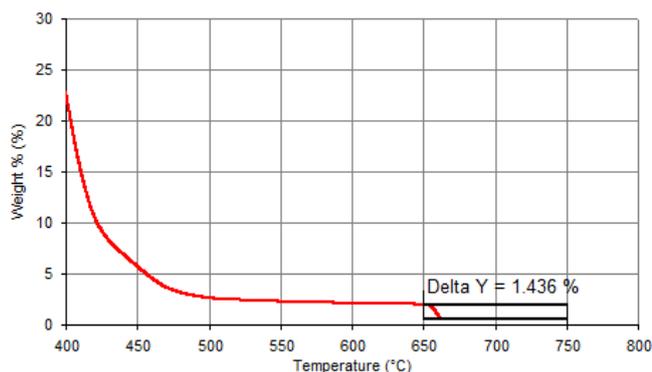


Fig. 7. Thermogram showing percentage soot content in the used engine oil sample (oil mileage: 8000 km)

It should be pointed out that the soot content found is, for a spark-ignition engine, powered in a PFI system, surprisingly large. The more so that the oil has not yet reached the qualifying mileage for its change (15,000 km). These values even exceed the soot content of used diesel engine oils. For example, in the tests of oils from four passenger cars with diesel engines presented by Skoczylas [12] it was found that in the case of good technical condition of the engine (low oil consumption), the content of soot in used oil does not exceed 1% (m/m). Similar results were obtained by author for an oil with a mileage of 16,000 km from a turbocharged diesel engine with a capacity of 3.0 dm<sup>3</sup> [6].

It seems that the main reason for this situation (high soot contamination) was a turbocharger working in the forced induction system of the engine and very dynamic (unfavorable) conditions of engine operation – only urban traffic conditions. It is very important that as it turns out also a spark-ignition engine in good technical condition can be a source of high particulate matter emissions.

Assuming a linear increase in soot content, at 15,000 km mileage (oil change period) it will be around 3% (m/m). This value is definitely unfavorable when it comes to the oil's lubricating role.

#### 4. Conclusions

The results described in this article relate to the determination of thermogravimetric characteristics and soot content in engine oil from a small turbocharged spark-ignition engine. It should be noted that the tests described in the article are tests of the oil from one car only. However, the authors do not know any reason that could diminish the credibility of the obtained test results. There was no indication that the engine was in poor condition. The engine did not consume oil. The tests conducted first of all lead to interesting conclusions regarding the content of soot in oil.

Firstly, the spark-ignition engine powered in the PFI system can generate large amounts of soot, and thus emit a large mass of particle matter. It seems that in the present case this is due to the design of the engine and its operating conditions. The results should be verified by measuring PM emissions from this engine, which is a part of the planned further tests. It may therefore turn out that the small city car can be more harmful to the environment than much larger vehicles.

Secondly, another important observation is that the recommended oil change interval – 15,000 km – for this engine (vehicle) is too long. Rapidly increasing soot oil contamination causes deterioration of oil lubricating properties and contributes to increased abrasive wear of the engine. Given the soot content of oil, this period should be reduced by up to half.

#### Nomenclature

ACEA European Automobile Manufacturers' Association  
 API American Petroleum Institute  
 ASTM American Society for Testing and Materials  
 GDI gasoline direct injection  
 NEDC New European Driving Cycle

PFI port fuel injection  
 PM particulate matter  
 TGA thermogravimetric analyzer  
 ZDP zinc dithiophosphate

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