

COMPARATIVE ASSESSMENT OF SL/CF SAE 5W/30 LUBRICATING OIL SERVICE IN ENGINES FUELLED BY 95 HYDROCARBON GASOLINE AND GASOLINE WITH 5%(V/V) ETHANOL ADDITIVE

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Summary. The paper provides an assessment of physicochemical properties of SAE 5W/30 lubricating oil in service in vehicles fuelled by 95 hydrocarbon gasoline and gasoline with 5%(v/v) ethanol additive. It also presents the technical condition of investigated engines.

Key words: motor vehicle service, lubricating oil, hydrocarbon gasoline, gasoline with 5%(v/v) ethanol additive.

1. INTRODUCTION

A trend towards increasing reliability of motor vehicle engines and reducing the consumption of lubricants and fuels, thus lowering operating costs, sets stricter requirements for physicochemical parameters of engine oils. The development of engine oil technologies is also affected by advancements in the design and technology of piston combustion engines, including turbocharging, exhaust gas recirculation, increasingly more effective catalysers or exhaust gas filters, etc. [1]. Users of automotive vehicles expect to be offered an engine oil which guarantees the maximum performance and optimum engine durability throughout its whole service life. Manufacturers of both lubricants and engines try to meet these requirements by adopting an approach in which engine oil is treated as an engine's structural component. Thus, the problem of engine oil quality concerns engine manufacturers, producers of lubricants and additives used to enhance lubricant performance, and also an enormous number of motor vehicle users.

2. MONITORING CHANGES IN FUNCTIONAL PROPERTIES OF LUBRICATING OIL IN THE ENGINE FUELLED BY 95 HYDROCARBON GASOLINE WITH 5% (V/V) ETHANOL ADDITIVE

An increased interest in ethanol both as hydrocarbon gasoline additive and also as E85 engine fuel has been noted in Europe. The data on the fuelling in different car makes are presented in Table 1 [2].

Table 1. Selected makes of cars fuelled by either gasoline with ethanol additive or by E85 ethanol fuel

No.	Car make	Acceptable ethanol content	Year of manufacture
1.	Chevrolet S10 Pickup Flexifuel	85 %	200–
2.	Chevrolet, all models	10 %	1997–
3.	Citroen Berlingo	10 %	1998–
4.	Citroen Zx	10 %	1998–
5.	Ford Escort	10 %	1998–
6.	Ford Fiesta	10 %	1998–
7.	Ford Focus	10 %	1998–
8.	Ford Focus Flexifuel	85 %	2001–
9.	Ford Mondeo	10 %	1998–
10.	Ford Puma	10 %	1998–
11.	Ford Scorpio	10 %	1998–
12.	Ford Taurus	85 %	1994–
13.	Ford Transit	10 %	1998–
14.	Honda	10%	all
15.	Mazda	10 %	1987–
16.	Mitsubishi MPI motor	10÷15 %	1989–
17.	Opel	10 %	all
18.	Renault Clio 19	15 %	all
19.	Renault Espace	15 %	all
20.	Renault Laguna	15 %	all
21.	Renault Mégane	15 %	all
22.	Renault Twingo	15 %	all
23.	Saab 9-5 2,0	10 %	1998–
24.	Saab, all with catalyser	10 %	1987–

25.	Suzuki	10 %	all
26.	Toyota Avensis	10 %	1998–
27.	Toyota Camry	10 %	1998–
28.	Toyota Corolla	10 %	1998–
29.	Toyota HiAce	10 %	1998–
30.	Volvo S/V 40	10 %	1998–
31.	Volvo S/V 70	10 %	1998–
32.	Volvo S/V 70 2,5 GLT	10 %	1998–
33.	Volvo S80	10 %	1992–

Although a lot of research work on engine fuelling by gasoline with ethanol additive has been done, many issues still remain to be thoroughly investigated. Those include fuel interaction with lubricating oil or the compatibility of the latter with the engine structural material [3,4]. The internal combustion engine durability should be analysed taking into account the three components of the system: the engine, the fuel and the lubricating oil. The investigations reported in the paper involved SL/CF SAE 5W/30 engine oil, which was singled out to lubricate motor vehicle engines fuelled by 95 hydrocarbon gasoline and hydrocarbon gasoline with 5% (v/v) ethanol additive. Basic physical and chemical properties of fresh SAE 5W/30 engine oil selected to lubricate vehicles in service are presented in Table II.

Table 2. Basic physicochemical properties of the SL/CF 5W/30 engine oil selected to lubricate vehicle engines in service [3]

No.	Investigated properties	Units	Experimental results
1.	Kinematic viscosity: – at 40°C – at 100°C	mm ² /s	58.19 10.02
2.	Total acid number	mg KOH/g	2.2
3	Total base number	mg KOH/g	10.8
4	Content of elements from quality package: – calcium – magnesium – zinc – phosphorus	ppm	1108 925 942 898
5.	Spectrum		Appendix 1
6.	Resistance to oxidation in a thin layer	min.	54

Comparative investigations into changes in physicochemical properties of the SL/CF 5W/30 engine oil during the service period of vehicles fuelled by 95 hydrocarbon gasoline and gasoline with 5% (v/v) ethanol additive covered the following processes:

- reactions of neutralisation of acidic exhaust products by neutralising additives;
- hydrocarbon oxidation reactions due to the action of high-temperature oxygen present in the combustion chamber;
- nitration reactions, that is reactions occurring when components of lubricating oils, mainly hydrocarbons, come in contact with nitrogen oxides formed under the conditions of engine work (especially those that facilitate high temperatures and mean combustion pressures in chambers), which lead to the formation of organic nitrates; the concept was introduced to differentiate such processes from those known from practical organic chemistry of nitration processes;
- by analogy, sulphur oxides reactions in the presence of steam at elevated pressure, which lead to the formation of sulfonation products;
- reactions of degradation of non-hydrocarbon components (mainly improver additives), which lead to a partial or total loss of some properties of the utilised engine oil, e.g. lowering of the total base number, decompositions of anti-wear additives based on dithiophosphates, or mechanochemical processes (shearing processes) of additives modifying the oil's rheological properties;
- reactions of interaction between surface-active compounds, improver additives, found both in the lubricating oil and the fuel, which can alter detergent-dispergation properties of the engine oil, leading to the formation of suspension, sludge, lacquer and precipitations of diversified chemical composition;
- higher-boiling fuel components migration to the engine oil, which can affect oil viscosity characteristics;
- the building-up in the engine oil, in the course of service, of other impurities of different origin, e.g. combustion products, such as water (which can cause the hydrolysis of some additives) or black soot, which mainly refers to self-ignition engines, and also dusts and environmental mineral contaminants, which can result in the increased wear of the engine parts.
- leaks of ethylene glycol from the coolant to the engine-lubricating oil due to faults.

Physicochemical properties of the said engine oil were monitored in accordance with the CEC M-13-T-92 test procedure, following standardized investigation methods currently in force – Table 3 [3].

Table 3. Research methods for the determination of lubricating oil's physicochemical properties in accordance with API classification and CEC-13-T-9 procedure [3]

No.	Engine oil property under investigation	Investigation method in accordance with
1.	Kinematic viscosity	PN-EN ISO 3104
2.	Viscosity innex	ASTM D 2270
3.	Total acid number	ASTM D 664
4.	Total base number	ASTM D 2896
5.	Water content	ASTM D 95
6.	Content of elements from quality package	ASTM D 4951
7.	Content of elements from the wear of engine elements	ASTM D 5185

8.	Content of insoluble impurities	ASTM D 893
9.	Sulphate ash	ISO 3984
10.	Evaporation loss by NOACK volatility test	PN-C-04124
11.	Oxidation, nitration sulfonation levels	ASTM E 2412
12.	Resistance to oxidation, resistance to corrosive action	CEC L-40-A-78

While assessing the degree of engine oil degradation in service, one should also take into account a vast volume of works on standardization carried out by ASTM working groups. Those included the determination of nitration, oxidation, sulfonation by-products, black soot content, fuel content, ethylene glycol contamination, water contamination, and also monitoring changes in the content of anti-wear additives with Fourier transform infrared spectral analysis techniques. The assessment of SAE SL/CF 5W/30 engine oil anti-wear properties in the motor vehicle 3x20 000 km service in relation to the selected working elements of the engine was carried out on the basis of **Induced Coupled-Plasma Atomic Emission Spectrometry** ICP AES in accordance with ASTM D 5195 method [5] and micrometric measurements, Fig.1.

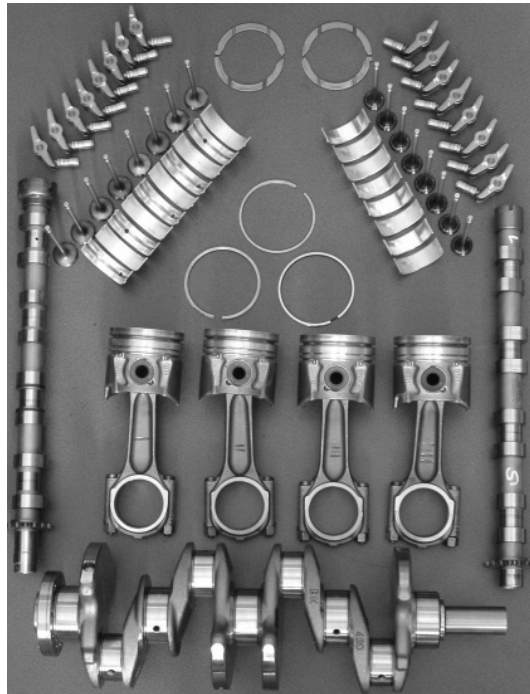


Fig. 1. Working elements of the engine lubricated with SL/CF SAE 5W/30 engine oil following 3x20 000 km service with their micrometric measurements

Results of laboratory tests on changes in physicochemical properties of the SL/CF SAE 5W/30 engine oil in in-service vehicles fuelled by engine gasolines are shown in Tables 4 - 5.

Table 4. The monitoring of physicochemical properties of SL/CF SAE 5W/30 engine oil in in-service (30 000 km mileage) vehicle fuelled by hydrocarbon gasoline

No.	Property under investigation	Mileage					
		5 000 km	10 000 km	15 000 km	20 000 km	25 000 km	30 000 km
1.	Kinematic viscosity; mm ² /s:						
	– at 40°C	57.45	62.47	69.91	72.20	76.70	79.95
	– at 100°C	10.16	10.41	11.15	11.29	12.30	12.61
2.	Total acid number; mg KOH/g	3.91	4.83	6.12	6.63	7.94	9.10
3.	Total base number; mg KOH/g	10.04	9.65	8.88	8.25	7.06	6.10
4.	Elements; ppm:						
	– phosphorus	790	821	839	938	933	913
	– zinc	937	1008	1013	1016	1023	1032
	– magnesium	943	959	976	951	981	976
	– calcium	1224	1266	1269	1234	1283	1285
	– lead	<0.5	2.6	4.7	5.5	7.2	15.6
	– iron	14	23.3	31	43	50.7	57
	– chromium	0.6	1.2	2.4	2.6	2.8	3.0
	– tin	2.1	2.1	4.7	5.7	7.0	7.3
	– silicon	14	18	21	21	23	27
	– aluminium	2.2	7.5	9.6	9.6	9.8	13
	– copper	3.8	7.0	8.1	11	12.9	15
5.	Insoluble sediments; % <i>(m/m)</i>						
	– in n-pentane	–	–	–	–	–	0.113
	– total	–	–	–	–	–	0.92
6.	Dispersion index	–	–	–	–	–	0.94
7.	Resistance to oxidation, min.	–	–	–	–	–	22

Table 5. The monitoring of physicochemical properties of SL/CF SAE 5W/30 engine oil in in-service (30 000 km mileage) vehicle fuelled by hydrocarbon gasoline with the 5%(v/v) ethanol additive

No.	Property under investigation	Mileage					
		5 000 km	10 000 km	15 000 km	20 000 km	25 000 km	30 000 km
1.	Kinematic viscosity; mm ² /s:						
	– at 40 ⁰ C	53.39	59.64	63.30	66.53	68.84	75.49
	– at 100 ⁰ C	9.68	10.36	10.68	11.04	11.21	11.73
2.	Total acid number; mg KOH/g	3.79	4.84	6.42	6.87	7.66	8.72
3.	Total base number; mg KOH/g	9.83	8.03	7.20	6.64	6.00	5.44
4.	Elements; ppm:						
	– phosphorus	847	907	886	861	820	830
	– zinc	985	998	996	985	999	969
	– magnesium	956	949	986	976	963	992
	– calcium	1289	1274	1256	1280	1258	1270
	– lead	4.7	5.7	6.5	8.3	9.7	9.8
	– iron	17	24.8	29.9	36	38	40
	– chromium	2	2.7	2.7	2.9	3	3.4
	– tin	5.2	5.9	7.3	8.3	9.7	10.6
	– silicon	8.9	10.2	11.3	13	16.7	17
	– aluminium	6.5	8.3	8.9	9.0	10.5	10.9
	– copper	3.7	5.9	8.6	10.8	10.9	12
5.	Insoluble sediments; %(m/m)						
	– in n-pentane	–	–	–	–	–	0.095
	– total	–	–	–	–	–	2.03
6.	Dispersion index	–	–	–	–	–	0.92
7.	Resistance to oxidation, min.	–	–	–	–	–	17

The range of investigations into SL/CF SAE 5W/30 engine oil covered the assessment of the technical state of the working parts of the engine fuelled by hydrocarbon gasoline and gasoline with 5%(v/v) ethanol additive before and after 60 000 km vehicle running. Exemplary results of the piston head and skirt measurements are presented in Tables 6 - 7, whereas the piston measurement base is shown in Fig. 2.

Table 6. The results of micrometric measurements of the piston skirt of the engine lubricated with SL/CF SAE 5W/30 lubricating oil for the vehicle fuelled by hydrocarbon gasoline after 60 000 km service

Meas- urement plane	The piston successive number	1		2		3		4	
	Measurement direction	A – A	B – B	A – A	B – B	A – A	B – B	A – A	B – B
I – I	Before exami- nation	82.331	82.332	82.330	82.331	82.342	82.321	82.350	82,319
	After examina- tion	82.386	82.311	82.393	82.311	82.383	82.318	82.388	82,317
	Wear [mm]	-0.055	0.021	-0.063	0.020	-0.041	0.003	-0.038	0,002
II – II	Before exami- nation	81.978	81.978	81.971	81.970	81.980	81.979	81.979	81,978
	After examina- tion	81.970	81.968	81.965	81.961	81.970	81.971	81.968	91,970
	Wear [mm]	0.008	0.010	0.006	0.009	0.010	0.008	0.011	0,008
III – III	Before exami- nation	82.978	–	82.977	–	82.975	–	82.976	–
	After examina- tion	82.964	–	82.954	–	82.961	–	82.963	–
	Wear [mm]	0.014	–	0.023	–	0.014	–	0.013	–

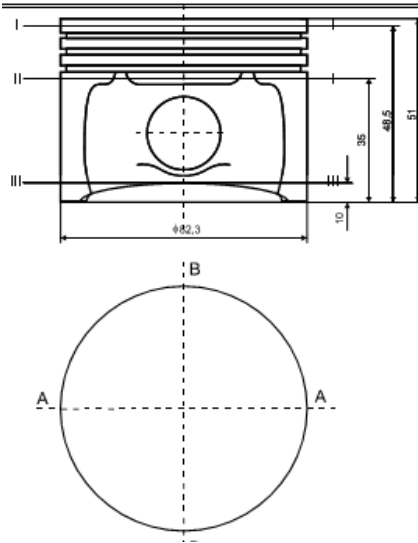


Fig. 2. The base of micrometric measurements of the engine piston after 60 000 km service

Table 7. The results of micrometric measurements of the piston skirt of the engine lubricated with SL/CF SAE 5W/30 lubricating oil for the vehicle fuelled by hydrocarbon gasoline with 5%(v/v) ethanol additive after 60 000 km service

Measurement plane	The piston successive number	1		2		3		4	
	Measurement direction	A – A	B – B	A – A	B – B	A – A	B – B	A – A	B – B
I – I	Before examination	82.340	82.340	82.390	82.330	82.340	82.330	82.330	82.340
	After examination	82.407	82.331	82.405	82.291	82.399	82.318	82.404	82.325
	Wear [mm]	-0.067	0.009	-0.015	0.039	-0.059	0.012	-0.074	0.015
II – II	Before examination	82.600	82.590	82.580	82.570	82.580	82.570	82.580	82.570
	After examination	82.592	82.587	82.568	82.561	82.569	82.561	82.567	82.562
	Wear [mm]	0.008	0.003	0.012	0.009	0.011	0.009	0.013	0.008
III – III	Before examination	82.977	–	82.975	–	82.979	–	82.978	–
	After examination	82.964	–	82.950	–	82.945	–	82.941	–
	Wear [mm]	0.013	–	0.025	–	0.034	–	0.037	–

3. PROCESS OF DEGRADATION OF SAE 5W/30 ENGINE OIL IN SERVICE

While performing its function in the piston internal combustion engine, the engine oil is, at the same time, affected by processes that impair its operational properties. The process, which is termed in-service aging by many researchers [6, 7, 8], is caused by the following phenomena:

- oxidation of the oil by air oxygen;
- contamination of the oil base stock by oxidation products and external impurities from the environment;
- destruction of the package of improver additives;

The loss of physicochemical properties results in an increase in contamination with coke, lacquer or resin deposits on the engine working elements: the head, the head cover, external and internal surfaces of piston rings and skirts. Table 8 presents results of the assessment of the degree of immobilisation of piston rings in the engine fuelled by gasoline with 5%(v/v) ethanol additive after 60 000 km service. The assessment was made in accordance with PN-73/C-04158 standard, the visual evaluation of the elements is shown in Fig.3.

Table 8. Score assessment of the degree of immobilization of piston rings in the engine fuelled by hydrocarbon gasoline with 5%(v/v) ethanol additive

Automobile make	The piston successive number	Type of piston rings	Unrestricted	Stub-born	Seized on circumference / foot	Stuck on circumference / foot	Score
Ford Mondeo 1.8	1	– compression –compression/scraper – scraper	yes	no	no	no	10
	2	– compression –compression/scraper – scraper	yes	no	no	no	10
	3	– compression –compression/scraper – scraper	yes	no	no	no	10
	4	– compression –compression/scraper – scraper	yes	no	no	no	10
	Average score of the degree of immobilisation of piston rings						10
	Average score of the degree of immobilisation of scraper rings						10



Fig. 3. Visual evaluation of the technical condition of piston rings and the piston ring area in the engine fuelled by hydrocarbon gasoline with 5%(v/v) ethanol additive

Rings of the piston of the engine fuelled by 95 hydrocarbon gasoline and by gasoline with 5%(v/v) ethanol additive after 60 000 km service retained their high (maximum) score, i.e. 10 points in accordance with PN-73/C-04158 standard. It should be emphasised that the degradation of lubricating medium is largely affected by the engine service conditions:

- short driving distances of a vehicle (multiple engine heating and cooling);
- driving in traffic jams (driving conditions of “stop and go” traffic);
- **engine running** for a **prolonged** period of time at **idle** speed (stop and go driving conditions of traffic-jammed vehicles);
- frequent engine starts at low temperatures,
- vehicle service in mountainous terrain (alternate, rapid engine overheating and cooling),
- water condensation on the engine internal parts and formation of lubricating oil-in-water emulsion.

*** kinematic viscosity**

Viscosity is a fundamental feature of the engine oil. If it is too high, substantial energy losses occur in the working system. Too low viscosity can cause oil leak from between lubricated elements, which, in turn, leads to an increase in wear, and even the system seizure. The engine oil viscosity is influenced by many factors. Increase in viscosity can result from the presence of solid contaminants, incomplete fuel combustion, the oil superheating due to degradation processes accompanied by oxidation and polymerisation. At the initial stage of service, kinematic viscosity of the SAE 5W/30 engine oil in the engine fuelled by gasoline with 5% (v/v) ethanol additive changed only slightly when compared with the fresh oil. After further 30 000 km service, increase in viscosity was more noticeable and amounted to 39 % under winter driving conditions.

*** total acid number**

When the engine oil works, it comes in contact with products of acidic nature that originate both in fuel combustion and in the oxidation of oil components with air at high temperature, which results in a gradual fall in the total base number. First, it is connected with the neutralisation of hyperacid products, and finally, with insoluble substance precipitation in the oil. In 30 000 km service, the total acid number of the oil under consideration in the engine fuelled by gasoline with 5% (v/v) ethanol additive tended to increase more than in the engine fuelled by 95 hydrocarbon gasoline. Under winter driving conditions, the total acid number increase was approx. 320%, and for 95 hydrocarbon gasoline – approx. 290% when compared with the value for the fresh oil.

*** total base number**

For gasoline with 5% (v/v) ethanol additive a more significant fall in TBN was noted in the engine oil, where under winter service conditions it amounted to approx. 52%, whereas for 95 hydrocarbon gasoline to approx. 41%, when compared with the fresh oil.

*** content of insoluble sediments and dispersion index**

Service conditions of automobile engines did not significantly affect either the level the total amount of impurities (dispergated ones or sediments which tend to precipitate on the engine elements), or the dispersion index, that is oil ability to keep contaminants in the state of dispersion.

*** oil resistance to oxidation in a thin layer**

The 30 000 km service of the engine oil under investigation resulted in substantial deterioration in resistance to oxidation in a thin layer (assessed in the rotating bomb oxidation test). At the end of 30 000 km service, in the engine fuelled by gasoline with 5% (v/v) ethanol additive, the resistance was 31% (winter conditions) of the resistance of the fresh oil, whereas for 95 hydrocarbon gasoline the resistance value was approx. 37%.

4. ANALYSIS OF OIL SEDIMENTS FROM THE ENGINE LUBRICATING SYSTEM IN SERVICE WITH RESPECT TO SAE 5W/30 LUBRICATING OIL COMPATIBILITY WITH FUEL IMPROVER ADDITIVE PACKAGE

SL/CF SAE 5W/30 engine oil (fresh) was examined with X-ray fluorescence method – Fig. 4., using XKR ED 2000 spectrometer from Oxford Instruments company.

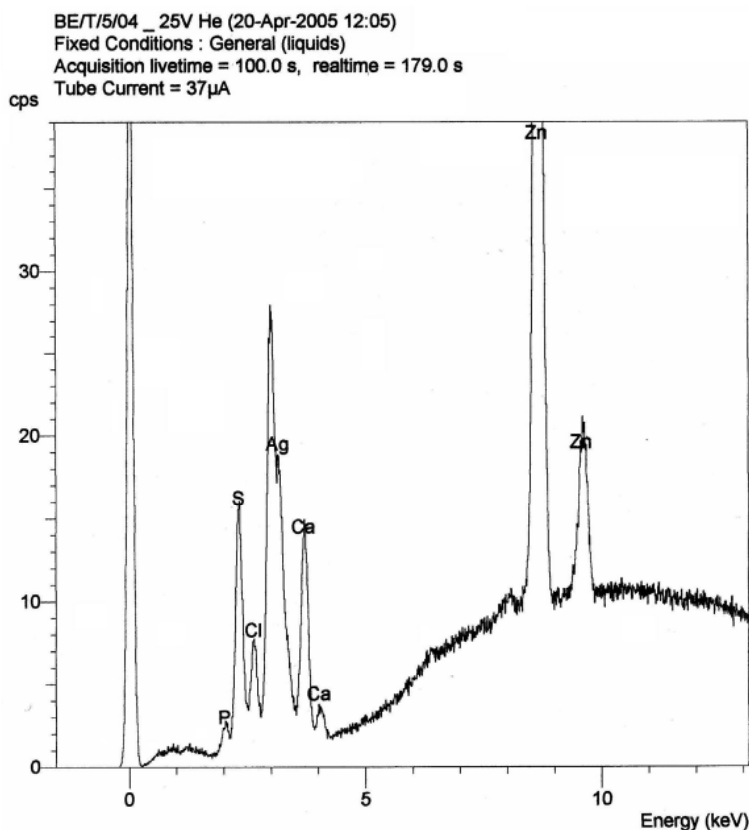


Fig. 4. XRF spectrum of fresh SL/CF SAE 5W/30 engine oil

The following elements, originating in improver additive package, were found to be present: calcium, magnesium, zinc, sulphur, chlorine and phosphorus. In the lubricating system of the engine fuelled by gasoline with 5% (v/v) ethanol additive, the oil spectrum underwent very similar but more pronounced changes in service than in the engine fuelled by 95 hydrocarbon gasoline. Fig. 5. shows the spectrum of fresh SL/CF SAE 5W/30 engine oil. The IR spectrum in the range of $1800\text{--}1470\text{ cm}^{-1}$ of SL/CF SAE 5W/30 engine oil from the lubricating system of the engine fuelled by 95 hydrocarbon gasoline with 5%(v/v) ethanol additive, for 5,10,15,20,25,30 000 km mileage (summer conditions) is presented in Fig. 6.

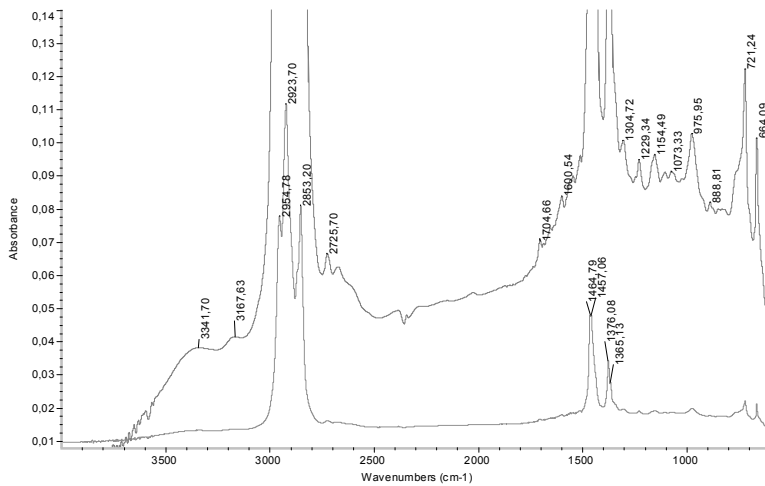


Fig.5. IR spectrum of fresh SL/CF SAE 5W/30 engine oil

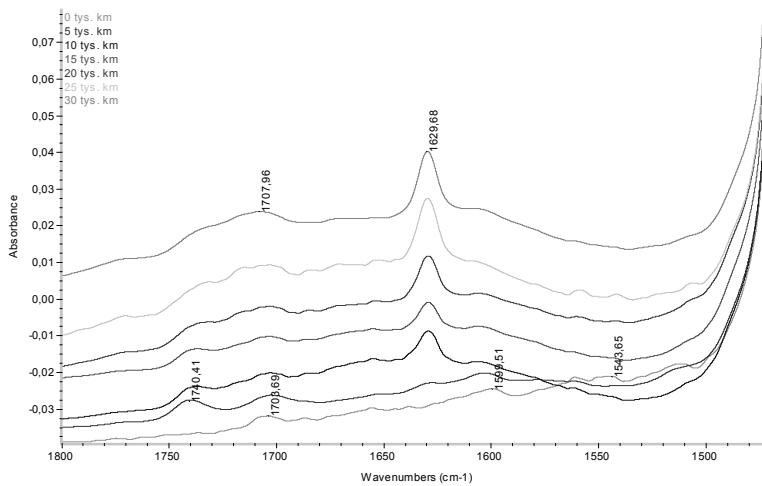


Fig.6. IR spectrum in the range of $1800-1470\text{ cm}^{-1}$ of SL/CF SAE 5W/30 engine oil of the sample from the lubricating system of the engine fuelled by 95 hydrocarbon gasoline with 5%(v/v) ethanol additive, for 5,10,15,20,25,30 000 km mileage (summer conditions)

5. CONCLUSIONS

The results of investigations of SL/CF SAE 5W/30 engine oil indicated that the type of fuel used to run the engine had a significant effect on the engine oil viscosity increase and seasonal conditions of vehicle service.

The engine oil under consideration contained viscosity modifier, which in the course of service was subjected to slight mechanical shear, due to which kinematic viscosity did not fall rapidly at the beginning of the service. It should be emphasised that viscosity modifier destruction processes were of key importance for the oil kinematic viscosity, prior to aging and nitration processes. Although the viscosity of the examined oil approached the viscosity lower limit designated for SAE 30 grade, on no occasion viscosity reduction due to viscosity modifier shear was high enough to downgrade the oil to SAE 20. Resistance to oxidation, evaluated on the basis of an increase in the total acid number to the level acceptable to top manufacturers of gasoline (petrol) engines, and on the cleanness of working elements was assessed as good for the 25,000 km mileage.

Similar conclusions can be drawn on the basis of investigations into TAN increment in SL/CF SAE 5W/30 engine oil in service, into total contaminant content and determined oil resistance to oxidation in a thin layer.

Results concerning the total content of oil contaminants indicate that the amount of oil degradation products resulting from complex processes differ, to a great extent, depending on the fuel used to run the engine. The assessment of said engine oil contamination with metal particles from engine element wear was made **using Induced Coupled-Plasma Atomic Emission Spectrometry** ICP AES in accordance with ASTM D 5185 method.

Detergent-dispersion properties, acceptable for 30 000 km service, were determined on the basis of the amount and the character of contaminants in oil and those precipitated on elements of the engine fuelled by hydrocarbon gasoline with 5%(v/v) ethanol additive.

Good anti-wear properties of the oil under consideration for 30 000 km service under summer and winter driving conditions were confirmed by the determination of the content of metal elements (Pb, Fe, Cu Cr) from engine part wear, as the content of metals did not exceed boundary values, and by the results of the engine micrometric measurements following 60 000 km service.

X-ray and infrared investigations of SL/CF SAE 5W/30 engine oil in service did not clearly demonstrate improver package components migration from the fuel to the engine oil. One of the probable reasons why intensity increase in polyether band at 1106 cm^{-1} is observed under winter conditions, is the dissolving in oil of polyether component of gasoline fuel additives.

Summing up, it can be stated that SL/CF SAE 5W/30 engine oil demonstrates reliable service usability for 25 000 km mileage in the whole range of functional properties of SI engines fuelled by both types of engine gasoline that are PN – EN 228:2003 standard compliant.

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OCENA EKSPLOATACYJNA PORÓWNAWCZA OLEJU SMAROWEGO SL/CF SAE 5W/30 W SILNIKACH ZASILANYCH BENZYNĄ WĘGLOWODOROWĄ 95 I BENZYNĄ Z DODATKIEM 5% (V/V) ETANOLU

Streszczenie. W referacie zamieszczono informacje dotyczące oceny właściwości fizykochemicznych oleju silnikowego SAE 5W/30 w okresie eksploatacji samochodów napędzanych benzyną węglowodorową 95 i benzyną z dodatkiem 5% (V/V) etanolu oraz ocenę stanu technicznego silnika.

Słowa kluczowe: eksploatacja, olej silnikowy, benzyna węglowodorowa, benzyna z dodatkiem 5% (V/V) etanolu.