

# Application of Atomic Emission Spectrometry in Aviation Piston Engine Abnormal Wear Prevention

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The measurement of wear metals concentration in used lubricating oil can be used to determine whether an engine is approaching failure, allowing further damage to be avoided. In this paper the application of inductively coupled plasma-atomic emission spectrometry (ICP-OES) technique in prevention of abnormal wear of aviation piston engines is explained. Used oil samples taken from two different engines are analyzed in respect with the wear metals concentration using ICP-OES. Concentration level for each metal is plotted against time of flight and its changes are observed. It is shown that low metals levels and small concentrations changes are connected with the well lubricated surfaces, while sudden concentration increase is an indication of wear beginning. Factors affecting the wear quantification are also summarized.

*Key words:* aviation engine, piston engine, wear, engine oil, atomic emission spectrometry.

## Introduction

THE main function of lubricating oil in the engine is friction reduction between metal parts mating together. Therefore the wear rate of metal components is reduced. However, since continual wear occurs anyhow during the operation, wear debris remain in lubricating oil and circulate with it through the system. Lubricating oil analysis is a fairly rapid and easy way to determine the engine contamination and wear [1]. Periodic oil sampling and analyzing during the engine exploitation allow data base formation for a normal wear rate monitoring, as well as abnormal wear diagnosis. Such analyses provide information of metal engine parts condition [2, 3].

The first worldwide spread application of this type of monitoring dates from 1940 when it was introduced by the American railway. By the middle 80s, spectroscopic engine oil analysis became a part of the engine condition monitoring program throughout USA [4, 5]. Regarding this success, the American Air Forces implemented Spectrometric Oil Analysis Program (SOAP) in their regular turbine engine condition maintenance system.

In the Serbian Air Forces this way of monitoring started in a modest manner a decade ago, reaching its full expansion with inductively coupled plasma spectrometer implementation few years ago.

Fossil lubricating oils are petrol derivatives composed of paraffinic, naphthenic and aromatic hydrocarbons. In order to fulfill numerous functions in the engine, their performance is enhanced with the addition of different types of additives. Metal to metal contact can be suppressed by adding a film forming anti-wear additives, abrasive wear with air filtration and corrosion wear with alkaline detergents. Diverse processes, both physical and chemical, are a cause for engine wear. Physical wear occurs because of the friction between metallic parts, high working pressures and temperatures,

abrasion caused by different contaminants from air. The main reason for chemical wear is corrosion caused by the acidic products formed during the exploitation. However, both wear types produce metal particles. They are both a consequence of wear as well as new damaging debris. Particle size distribution is wide [1,6]. Oil filter retains particles bigger than its porosity, while those smaller circulate through the system. Recording the key metals concentrations trend in used oil during the engine operation allow determination of normal wear rate concentrations and appropriate indications of abnormal wear of lubricated parts. For example, Ni, Sn or Cr concentration increase indicate bearings, pistons or valves corrosion, Al indicates piston pin plugs wear, Cu bearings wear, Fe indicates corrosion of different parts, etc. [1].

Metal concentrations in used lubricants are mostly on the low levels connected with the well lubricated surfaces while normal wear rate, but during the exploitation they increase slowly [7]. Sudden sharp rise of some metal content or ever increasing concentration indicate an increased wear and, eventually, inappropriate engine work.

Conventional analytic techniques used for the oil monitoring are electroanalytical [8, 9], XRF (X-Ray Fluorescence Spectrometry) [10], those based on atomic spectrometry like FAAS (Flame Atomic Absorption Spectrometry) [11-16], ETAAS (Electro Thermal Atomic Absorption Spectrometry) [17], ICP OES [15, 16, 18-20] and ICP MS (Inductively Coupled Plasma Mass Spectrometry) [16, 21, 22].

The main goal of this paper is to present the application of inductively coupled plasma atomic emission spectrometry in determination of the wear metals concentrations in used lubricating oil samples taken from two aviation piston engines in specified periods of flight and to present the way of using the obtained data in the engines wear monitoring. It will be shown how this kind of analysis can be useful in taking corrective

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actions at the beginning of the wear process and prevent further wear increase through the preventive maintenance.

### Factors affecting wear quantification

There are numerous factors that make quantitative engine wear monitoring very demanding. It must be pointed out that this kind of monitoring should not exclude other diagnostic techniques, like filter content inspection, differential cylinder pressure checks, boroscopic examination, etc.

Macian has already reviewed the majority of the factors influencing the wear quantification [23]. According to his findings, they can be divided in the following classes: the limits of technique used for wear quantification, the effects of exploitation conditions on the measured results and, finally, the performance of the particular engine (manufacturer, age, environment conditions, etc.).

#### Analyzing technique limits

Different wear types produce different particle size distribution. However, each spectrometry determination involves the particle size limitations. Inductively coupled plasma spectrometer has a 100% recovery for the particle size of maximum 5  $\mu\text{m}$ . Therefore this limit should be taken into account when results analysis is done.

Apart from the aforementioned, the majority of advances of this technique make it by far the best wear analysis technique at the moment and the one most used. For more complicated cases, the results of these analyses are combined with the additional analysis, like particle counting, ferrografi, etc.

#### Exploitation conditions

##### Oil consumption

Quantification of the wear might be expressed like a concentration of a metal in an oil sample ( $\mu\text{g/g}$ ). However, the concentration itself is affected by other factors, apart from the wear. Lubricant consumption during the exploitation is inevitable; therefore the lubricant has to be refilled periodically. The consequence of this consumption is wear metals concentrations increase, even though the wear rate remained constant. On the other hand, an addition of fresh lubricant causes decrease in the measured concentration. In a sufficient time period certain constant metals concentrations are reached [23].

##### Filtering effect

The important factor that influences the metals concentrations in lubricating oil is its filtration in the system. The contaminants quantity retained by the filters depends on the filter condition, as well as the particle size distribution and filter retention efficiency. Since spectrometry detects particles smaller than 5  $\mu\text{m}$ , and filters retain more than 90% of particles bigger than 10  $\mu\text{m}$ , therefore this effect can be neglected [23].

#### Oil chemical composition

Metallo-organic compounds can be added as additives in lubricants formulation. Therefore, it can be found that the same wear metal is present as additive in fresh lubricant. So, the fresh oil analysis is required in order to compensate current concentration.

### Method description

Even though inductively coupled plasma is used for a long time for metal analysis in the water solutions, significant effort

has been made in last decades to apply it to the organic solutions [22, 24-29]. American Society for Testing and Materials has developed the ASTM D5185 Method for Wear metals analysis in used lubricating oils using ICP-OES. The following analyses are done in accordance with ASTM D5185 [30].

Optical emission spectrometry with inductively coupled plasma source uses argon plasma energy for the sample atomization and excitation. Oil sample diluted in an organic solvent (mixed xylenes, kerosene, etc.) is nebulized into plasma. Sample evaporation, atomization, ionization, and excitation take place. The intensity of emitted light is measured. Each metal emits light in special wavelengths and those lines are used for a qualitative sample analysis. The emitted light intensity is proportional to the element concentration in lubricant and quantitative information is obtained. This technique has a low detection limit, wide dynamic concentration range and simultaneous and rapid determination of more than 20 elements.

### Experimental part

In this paper the aviation piston engines wear monitoring is done, using the spectroscopic analysis of used engine oil sampled from aircraft each 10 hours of flight, after the run-in period preceding the overhaul, when the system is settled in. Commercial piston engine oil AeroShell W80, SAE grade 40 is used. Obtained concentration values are plotted against time of flight, and concentration trend is observed. Slight concentration changes indicate normal wear rate, while sudden rise alarms for increased wear beginnings.

The main engine parts are made of the aluminum pistons, steel or chrome cylinders, chrome plated or steel piston rings, aluminum or aluminum bronze piston pin plugs, etc. These parts are prone to wear during the operation, and their metal particles are deposited in lubricant.

Sampling of the used lubricant samples is made in accordance to the engine manufacturer service letter recommendation. The oil sampling is also done in accordance with the engine manufacturer service letter recommendation. Fresh oil analysis of the wear metals is done, as well.

#### Apparatus

All analyses are performed on Spectro Genesis Inductively Coupled Plasma Optical Emission Spectrometer, with a radial viewing. With 15 linearly positioned CCD detectors, wavelengths range from 175 to 777 nm can be measured. Burgener nebulizer and cyclonic spray chamber are used. Working parameters are listed in Table 1.

Table 1. Operating parameters

Plasma Power	1450 W
Coolant flow	14 L/min
Auxiliary flow	1.8 L/min
Nebulizer flow	0.7 L/min
Spray Chamber	Cyclonic Type
Nebulizer	Burgener type
Pump tubing	Orange/orange for sample Grey/grey for drain

### Wear metals and wavelengths selection

Wear metals are chosen in accordance with the information about alloys composition used for the engine parts. Silicon is usually an indication of air dust contamination, so its concentration is also measured. The following elements are measured: Al, Cr, Cu, Fe, Sn, Si. Wavelengths are chosen taking into account the spectral interferences and are listed in Table 2.

**Table 2.** Selected wavelengths

Element	Wavelength, nm
Al	396.15
Cr	267.71
Cu	327.40
Fe	238.20
Sn	189.99
Si	251.61

### Calibration standards and calibration

For calibration purposes, certified reference materials, organic standards, are used:

1. Multi-element metallo-organic oil standard with 23 elements, V23 Wear Metals, LGC Standards, VH G Labs, 10 µg/g, 50 µg/g, 100 µg/g;
2. Base oil Blank 75mm<sup>2</sup>/s, LGC Standards, VH G Labs;
3. Internal oil standard, Co, 5000 µg/g, LGC Standards, VH G Labs;
4. Dilution solvent, V-SOLV™ ICP Solvent, LGC Standards, VH G Labs;
5. Multi-element metallo-organic standard of aviation oil with 19 elements, D19 Standard 10 µg/g; LGC Standards, VH G Labs;

Calibration standards of different concentrations are prepared by diluting the aforementioned metallo-organic standards in ICP solvent, 10% m/m. Cobalt solution is used as an internal standard. Internal standard is added in order to compensate for differences in viscosity and volatility of the samples and calibration standards. For a blank sample, base oil is dissolved in ICP solvent.

Calibration standards concentrations, correlation coefficients and detection limits are given in Table 3 and Table 4, respectively.

Multi-element aviation oil solution is used as a check standard during the analysis. The precision and accuracy of the measurements are given in Table 5.

**Table 3.** Calibration standards concentration

Element	CS 1, µg/g	CS 5, µg/g	CS 10, µg/g	CS 20, µg/g
Al	1.01	4.99	9.99	20.06
Cr				
Cu				
Fe				
Sn				
Si				

**Table 4:** Regression correlation coefficients and detection limits

Element	Correlation coefficient	Detection limit (LOD), µg/g
Al	0.99988	0.0085
Cr	0.99996	0.0072
Cu	0.99998	0.0107
Fe	0.99999	0.0063
Sn	1.00000	0.0371
Si	0.99999	0.0298

**Table 5.** Precision (RSD) and accuracy (Bias) of the check standard measurements

Element	RSD, %	Bias, %
Al	1.9	1.2
Cr	2.2	1.3
Cu	1.5	0.9
Fe	2.9	1.0
Sn	2.5	1.1
Si	3.0	2.2

### Samples preparation

Used oil samples are taken following the engine manufacturer instructions every 10 hours of flight. Appropriate homogenization of used oil sample is an important step in preparation for the analysis. This is done using the automatic homogenizer for half an hour (labs personnel experience with this type of homogenizer and the sample material). A sample portion of 2 g is then diluted with dilution solvent to 20 g. Two probes of each sample are prepared. The result is the mean value of three instrument readings. The final result is the mean value of two probes samples. Before and after the samples measurements, the control standards are measured.

### Results and discussion

The results for the piston engine 1 (APE-1) and 2 (APE-2) are presented in Table 6 and Table 7, respectively. Concentrations levels vs. time of flight for the piston engine 1 and 2 are presented in Figures 1 and 2, respectively. Used oil analysis from the aviation piston engine APE-1 shows that the concentration level of wear metals can be divided into two levels: element which concentration is above 10 µg/g constantly, and those elements present in concentrations below 10 µg/g.

Iron belongs to the first mentioned group. Such a result is expected, since the wear sources for iron are numerous. Their cumulative value is measured concentration, and therefore the most difficult element to locate in case of the abnormal wear. The limit value considered as a "normal wear" is 100 µg/g (the value given by the engine manufacturer, table 8). The average value measured in the samples from APE-1 for 130 hours of flight is around 10 µg/g. Since these measurements are done long enough after the overhaul, when the run-in period is elapsed and the engine is settled in, very low wear rate is observed.

The second element group involves the following metals: copper, chrome and aluminum. Their concentrations do not increase above 5 µg/g while normal wear. They have some slight fluctuations from 0.20 to 2.20 µg/g, without obvious rising trend. The limit values for normal wear rate for these metals are given in Table 8.

**Table 8.** Normal wear limit values given by the engine manufacturer

Element	Limit values, µg/g
Fe	100
Cu	10
Cr	50
Al	20
Si	10

Fig.1 indicates the following: sudden Si concentration increase in the sample number 2, from 3.7 to 23.8 µg/g is probably triggered by the contaminants (dust and sand) ingested with air during the engine operation. Consequently, slight rise of Cr and Fe concentration is noticed in the same sample, most likely caused by the abrasive contaminants wear. These results show eventual beginning of rings and cylinders increased wear, the engine parts made of these metals alloys. In order to prevent further wear propagation, corrective filter replacement, and therefore better contaminants retainment, quickly resulted in decreased silicon content below the limit value in the next sample, number 3, and prevention of further iron and chrome content increase. The following samples (number 4, 5 and 6) analyses show constant concentration level of copper, chrome and aluminum, with values less than 1.5 µg/g. At the same time, iron content

is at 10 µg/g. These values are inside the normal wear rate limits, and such steady concentrations levels in this operation period is a sign of no serious wear. In the samples number 6 and 7 iron content is still rising, along with the increase of silicon, copper and aluminum concentrations in the sample 7. This is an indication of the beginnings of aluminum alloys engine parts wear (pistons and piston pin plugs), caused again with dirt ingestion (Si rise). Subsequent filter change resulted in silicon decrease in the next sample, better dust and sand retainment, and sudden concentration decrease of all other four measured metals. This is evident from the analysis of the sample number 8. The following results show normal wear rate trend, with copper, chrome, iron and aluminum concentrations inside the acceptable limits for used oil. Silicon content increase is again evident starting from the sample number 8 until the sample number 12, followed by a sudden iron rise in the sample number 12. Corrective action taken immediately, the third filter replacement, prevented further increase of the engine wear.

Table 5. Engine APE-1: used engine oil analyses every 10 hours of flight

Sample number	Hours of flight	Fe µg/g	Cu µg/g	Cr µg/g	Al µg/g	Si µg/g
0	Fresh oil	< LOD	< LOD	< LOD	< LOD	< LOD
1	10	6.91	1.30	0.60	0.32	3.70
2	20	9.62	1.90	1.41	0.21	23.81
Filter replacement						
3	30	7.52	1.01	1.21	1.02	5.11
4	40	5.21	0.81	0.52	0.11	3.22
5	50	9.83	0.52	1.02	0.90	3.21
6	60	12.41	0.61	1.33	1.32	3.43
7	70	13.61	1.32	1.43	2.21	5.62
Filter replacement						
8	80	8.00	0.54	0.81	0.73	1.51
9	90	7.60	0.71	0.82	1.01	2.31
10	100	7.41	0.31	0.64	0.87	4.22
11	110	7.51	0.42	0.80	1.21	5.21
12	120	28.81	0.72	1.02	1.52	4.32
Filter replacement						
13	130	8.03	0.52	0.61	0.52	2.61

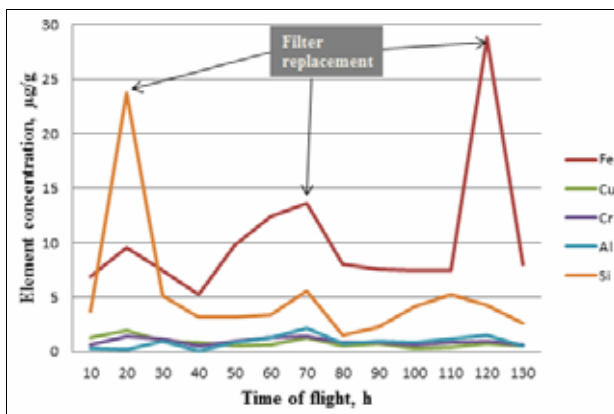


Figure 1. Engine APE-1: Elements concentration every 10 hours of flight

Used oil analysis from the aviation piston engine APE-2 reveal constant “normal wear” rate, showing slight fluctuation of the concentration levels in oil samples during time. All four metals concentrations (without Si) are inside the normal wear limits for used oil. Also, in this engine, the iron content is around 10 µg/g in samples. Except in the case of sample number 8, copper, chrome and aluminum do not rise above 5 µg/g, as well. This steady trend is associated with the satisfactory lubrication of metal parts. However, the analysis of the sample number 8 shows sharp rise of aluminum content, from 1.9 to 7.5 µg/g, simultaneously with iron and silicon rise. This abrupt escalation indicates that the wear

process takes place. That was the alarm for the subsequent engine inspection, necessary for increased wear confirmation and failure prevention. This shows how sudden wear can occur in one stable system, and how it results in concentration levels changes.

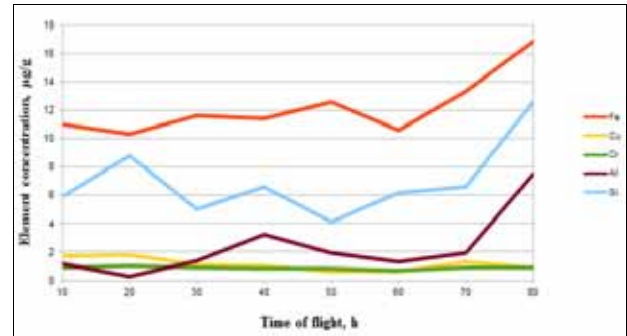


Figure 2. Engine APE-2: Elements concentration every 10 hours of flight

Table 6: Engine APE-2: used engine oil analyses every 10 hours of flight

Sample number	Hours of flight	Fe µg/g	Cu µg/g	Cr µg/g	Al µg/g	Si µg/g
0	Fresh oil	< LOD	< LOD	< LOD	< LOD	< LOD
1	10	11.01	1.70	0.92	1.20	5.92
2	20	10.32	1.81	1.20	0.32	8.81
3	30	11.61	1.12	0.91	1.41	5.12
4	40	11.44	1.11	0.81	3.22	6.61
5	50	12.63	0.63	0.82	1.92	4.12
6	60	10.62	0.61	0.73	1.31	6.22
7	70	13.32	1.31	0.91	1.91	6.61
8	80	16.81	0.92	0.92	7.53	12.63

### Concluding remarks

In this paper the application of inductively coupled plasma atomic emission spectroscopy as a useful tool for the aviation piston engines condition monitoring is described. Also, spectrometry technique limits and advantages are summarized. Discussing results of used engine oils taken in specified time of flight intervals from two different engines, it is explained how the wear metals and contaminants concentrations trends and changes can reveal engine contamination and wear beginning. It is also shown that taking corrective actions at the very beginning of the wear process further wear propagation can be suppressed. Therefore, this is a useful tool in predictive maintenance, especially for aviation engines, where any failure can be fatal.

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## Primena atomske emisije spektrometrije u prevenciji habanja avionskih klipnih motora

Merenje koncentracije metala habanja u korišćenom ulju za podmazivanje motora može biti dobar pokazatelj da li je blizu otkaz rada motora, omogućavajući da se pravovremenom reakcijom izbegnu krupnija oštećenja vitalnih delova motora. U ovom radu je prikazana primena tehnike indukovane kuplovane plazme-atomske emisije spektrometrije (ICP-OES) u prevenciji pojačanog habanja avionskih klipnih motora. Analizirani su uzorci korišćenog ulja za podmazivanje na sadržaj metala habanja iz dva avionska motora, korišćenjem ICP-OES. Grafički je predstavljena koncentracija svakog pojedinačnog metala habanja zavisno od vremena leta aviona i praćene su promene koncentracije. Pokazano je da je nagli skok koncentracije metala indikacija početka habanja, dok je niska vrednost iste pokazatelj dobro podmazanih površina. Predstavljani su faktori koji utiču na kvantifikaciju habanja.

*Ključne reči:* avionski motor, klipni motor, habanje, motorno ulje, atomska emisija spektrometrija.

## Применение атомно-эмиссионной спектроскопии в предотвращении износа авиационных поршневых двигателей

Измерение концентрации износа металлов в отработанном масле для смазки двигателя может быть хорошим индикатором того, является ли вблизи отказ и отмена двигателя, что позволяет своевременно реагировать, чтобы избежать большего повреждения жизненно важных деталей двигателя. В данной работе показан метод получения индуцированной связанной плазмой атомно-эмиссионной спектроскопии (ICP-OES) для предотвращения чрезмерного износа поршневых авиационных двигателей. Анализированы образцы использованного смазочного масла на содержание износа металлов из двух авиационных двигателей, с использованием ICP-OES. Графически представлена концентрация износа каждого металла в зависимости от времени работы – полёта самолёта, и здесь сопровождаются изменения в концентрации. Здесь показано, что внезапное увеличение концентрации металла – это индикация зачатков износа, в то время как низкое значение концентрации – показатель хорошо смазанной поверхности. Также представлены рекомендуемые факторы, влияющие на количественную оценку износа.

*Ключевые слова:* авиационный двигатель, поршневой двигатель, износ, моторное масло, атомно-эмиссионная спектроскопия.

## Application de la spectrométrie émissive dans la prévention de l'usure des moteurs d'avion à piston

Le mesurage de l'usure de métal dans l'huile de graissage usée des moteurs peut être un bon indicateur si la défaillance du moteur est proche et cela permet de réagir à temps pour éviter les endommagements considérables chez les parties vitales de moteur. Dans ce travail on présente l'application de la technique de plasma induit couplé – spectrométrie atomique émissive (ICP-OES) dans la prévention de l'usure rapide des moteurs d'avions à piston. On a analysé les échantillons de l'huile utilisée de graissage à l'égard du contenu du métal d'usure chez deux moteurs d'avion en appliquant la technique ICP-OES. On a présenté graphiquement la concentration de chaque métal particulier par rapport au temps de vol d'avion et on a observé les changements des concentration. On a démontré que l'augmentation rapide de la concentration de métal indique le début de l'usure alors que la basse valeur de concentration indique les surfaces bien huilées. On a présenté aussi les facteurs influents sur la quantification de l'usure.

*Mots clés:* moteur d'avion, moteur à piston, usure, huile de graissage, spectrométrie atomique émissive.