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Possibilities of Analyzing Sources of Impurities in Hydraulic Oil

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The task of comprehensive machine care, monitoring of reliability and maintenance is currently a priority not to wait for a malfunction, but to prevent malfunctions before they occur. In the possibilities of modern diagnostic tools, this activity is the task of oil analysis, which is a highly effective tool for monitoring the condition of hydraulic oils during their long-term operation. It is possible to prevent the failure of the entire system by regular monitoring of the technical condition of oils based on the analysis of hydraulic oil pollution. Oil analysis can reveal the amount of additive elements, oil pollution, the amount of additives and changes in the physico-chemical parameters of the oil. In order to determine the current state of the oil filling, it is necessary to use a suitable diagnostic method. The submitted contribution describes an experiment aimed at evaluating the state of hydraulic oil by means of EDX (Energy-dispersive X-ray spectroscopy) analysis, DSC (Different Scanning Calorimetry) analysis and infrared spectroscopy.

Keywords: EDX analysis, Reliability, Oil pollution, Additives, Infrared spectroscopy

1 Introduction

Tribotechnical diagnostics is included in the non-dismantling method, which deals with determining the technical condition of friction nodes on the basis of a sample of the grease with which these nodes are lubricated. Its task is to analyze the current state in the laboratory, evaluate it and determine the presence of foreign substances in the lubricant, determine its change, from a quantitative and qualitative point of view. Tribotechnical diagnostics allows us to determine such an analysis of lubricants and to identify emerging malfunctions that may occur in the operation of machines and equipment well in advance.

In order for oil analysis to be effective, it is advisable to follow the sequence of the following steps:

- what the analysis will bring, setting measurement goals,
- from which technological units the engine oil samples will be taken,
- which are the sampling locations,
- when to take the sample,
- sampling interval,
- what tests to perform.

For trouble-free and reliable operation of the technological unit, it is necessary to ensure proper lubrication in individual friction nodes of machines and equipment. In the normal practice of using technology, the chemical and physical stability of lubricating oils during their application is necessary and important. This is about ensuring important qualitative indicators of lubricating oils, such as viscosity (lubricating film thickness), rheological properties (fluidity), demulsification ability (water separation) and many others. Such stability of lubricating oils can be disturbed for various reasons and influences, such as increased operating temperature, load, dusty and humid environment, mixing of oils, which leads to oxidation (oil aging), loss of additives (hydrolysis), oil foaming (impurities) and other undesirable conditions [1,2]. In order for the development of lubricants and lubrication technologies across a wide range of applications to achieve the necessary level of success, it is necessary to bring together lubrication theory with many areas of expertise outside of tribology. In most applications, from automotive fluids to industrial oils and process oils, liquid lubricants are consumed. Worldwide, more than 10 000 different types of lubricants are used in the lubrication industry [3,4,5]. In addition to energy transmission, hydraulic oil also ensures the lubrication of components in the machine's hydraulic system. Contaminated hydraulic oil can cause many malfunctions and accidents. Classic mechanical pollution of this oil is mainly caused by metal particles and dust. Contamination of hydraulic oil is also caused by a number of other substances, so it is important to determine them through analyses.

A proven method of industrial oil analysis is the determination of the number of particles in the sample. This method helps to determine the oil contamination ratio and avoid machine breakdowns and unexpected downtime. Visual inspection of oil samples is not sufficient and authoritative to obtain complete and verified data on the degree of oil contamination.

Particles that are not visible to the naked eye can cause serious damage to the hydraulic system. A sample of the collected hydraulic oil may look clean to the naked eye, but it can nevertheless cause abrasive wear of the components in the hydraulic system, resulting in a reduction in the performance of the equipment and the efficiency of the entire system. The standardized number of particles gives a clear overview of the degree of purity of the oil. By measuring the number of particles in a sample of used oil, it is possible to determine the contamination ratio of this oil and subsequently determine whether the oil is clean enough for further reliable operation of the equipment. By monitoring the number and size of these particles in an oil sample, it is possible to prevent a number of future problems quickly and easily in the hydraulic system [6,7].

2 Experimental material and experiment

2.1 EDX analysis

The Energy Dispersive X-ray (EDX) analysis is a technique of elemental analysis associated to electron microscopy based on the generation of characteristic Xrays that reveals the presence of elements present in the specimens [8]. The individual separated nanoparticles are deposited on a suitable substrate that does not interfere in the characterization of nanoparticles. This method has found some limitations with regard to accurate dimensional and elemental characterization [9,10].

The basic principle of EDX is a generation of X-rays from a specimen through the electron beam. The X-rays are generated according to the characteristics and nature of the elements present in the sample. Hence, this technique also can be used to measure the energy of emitted X-rays. This method gives accurate results for not only element detection, but also to determine their concentration after maintaining the ideal instrumental conditions. There are three principal components present in basic EDX systems. These components include the X-ray detector, pulse processor to measure the voltage with respect to X-ray energies, and a computer system. The X-ray detector is positioned to intercept X-rays emitted from the specimen [11,12].

The emitted X-rays from the sample are detected by an X-ray detector. Upon entering the detector, an X-ray generates a small current, which is then converted into a voltage pulse. The voltage pulse is dependent on the X-ray energy. Histograms of this data may then be plotted following measurement of the voltage pulse for a 60 s period. This histogram represents the X-ray energy spectrum through which elemental analysis can be done [13,14,15]. The measurement of the number of particles was carried out on a sample of 13 oils from cars that work in a polluted environment, on a Shimadzu EDX-7000 device. It is an energy-dispersive X-ray fluorescence spectrometer that measures the energy and intensity of the generated fluorescent X-ray radiation to determine the type and content of the elements that make up the sample. The device works on non-destructive elemental analysis of solid, powder and liquid samples [16].

When a sample is irradiated with X-rays from an X-ray tube, the atoms in the sample create unique X-rays that are emitted from the sample. These x-rays with a unique wavelength and energy are known as fluorescent x-rays. They are characteristic of each element that produces them, so a qualitative analysis can be made by examining the wavelengths of X-rays. Due to the fact that the intensity of X-ray fluorescence is a function of concentration, it is also possible to perform a quantitative analysis by measuring this amount of X-rays at a wavelength specific to each element [17]. The content of chemical elements in the samples is shown in Tab. 1.

Tab. 1 *The content of chemical elements in the samples*

Element resources	
Element	**Possible source**
Al	bearings, bearing pans, pistons, coolers, pumps, dust, washers
Cr	bearings, pistons, rockers, bushings, valves, piston rings, seals, shafts
Cu	bearings, bearing pans, washers, pumps, transmission parts
Fe	aggregate blocks, bearings, bearing pans, cylinders, pumps, gears, piston rings
Pb	bearings
Ni	bearings, valves
Sn	bearings, bearing pans, pistons
Ca	water, detergent/dispersant additive, road salt
Mg	water, detergent/dispersant additive,
Mn	detergent, steel wear
Mo	friction modifier, anti-wear additive
P	anti-wear additive, EP (high-pressure) additive
Si	seals, dust, anti-wear additive, anti-foam additive
Na	anti-wear additive, road salt, detergent
Zn	anti-wear/antioxidant additive, alloy wear (mostly bearings).

Additive elements used in hydraulic oil (Tab. 2):

- Calcium (Ca) – the content in the new oil ranges from 40-50 mg/kg (ppm).
- Phosphorus (P) - the content in the new oil ranges from 300-350 mg/kg (ppm).
- Zinc (Zn) - the content in the new oil is around 400 mg/kg (ppm).
- The content of additive elements should not fall below 20 % of the value of the new oil. Due to possible changes in the additive content, it is necessary to verify the content with each batch of new oil.

The determined values for individual elements are compared with reference sample no. 13, whose values are considered limit values. Graphical comparison of results from Tab. 2 is shown in Fig. 1.

Tab. 2 *Contents of measured additive elements on 13 oil samples*

	Sample no.												
Element (ppm)	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**
S	2676	3392	7788	1862	5295	4136	3971	2883	6056	1608	4973	4148	4982
Zn	302	541	148	702	295	239	211	298	305	703	246	199	334
P	241	502	56	654	157	126	174	237	128	621	155	82	167
Ca	89	825	45	316	63	48	46	62	70	180	39	41	63
Cu	26	31	0	30	25	28	29	21	26	32	27	32	0
Mo	0	25	0	0	0	0	0	0	0	0	0	0	0
Fe	0	17	14	22	10	16	10	13	39	14	12	22	0
Cr	0	0	0	17	0	0	0	0	0	0	0	0	0
Ag	0	0	0	0	0	0	0	0	37	0	0	0	0

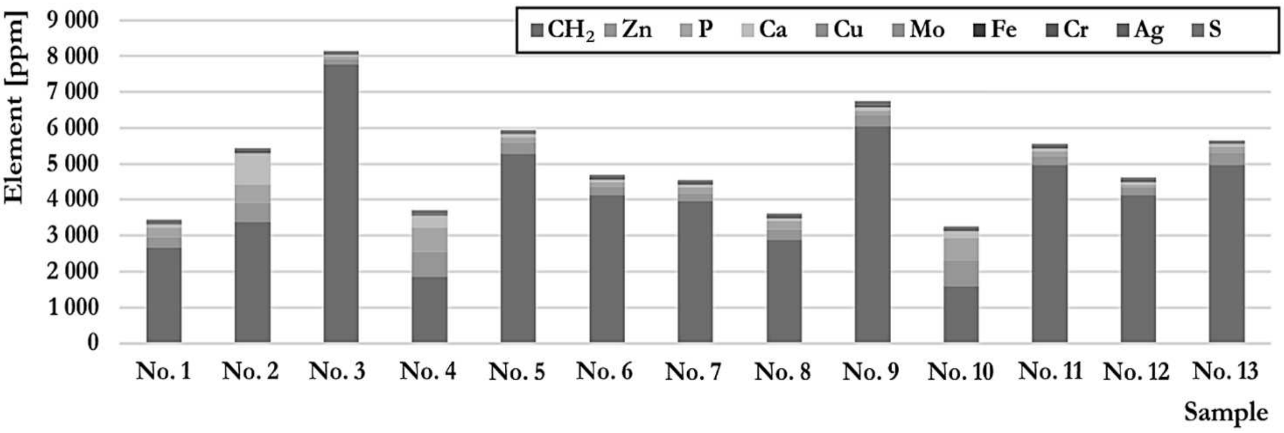


Fig. 1 *Representation of individual elements in the samples*

In Tab. 2, the permissible measured values of the given element are marked in green and the inadmissible over-limit values are marked in red. For sample no. 1, increased values of P and Cu were recorded, which can negatively affect the service life of the oil itself and indicate certain mechanical wear. Exceeding values of all additional elements except S were recorded for sample no. 2. An increased amount of elements Zn, P, Ca and Cu results in the formation of deposits that can clog valves and filters, Mo and Fe affect the viscosity of the oil and its abrasiveness, which leads to a shorter service life and further mechanical wear of parts. Sample no. 3 shows an over-limit amount of S and Fe, while excessive S limits can lead to the formation of acids and, consequently, corrosion of internal parts. Sample no. 4 shows, in addition to the already mentioned additional elements, as the only tested sample, the content of Cr. Its presence indicates wear of pistons and cylinders in hydraulic systems. Samples no. 5 and no. 9 show almost the same overlimit values of S, Cu and Fe. The content of these elements indicates a certain mechanical wear of the internal parts due to intruded corrosion and abrasion. Samples no. 6, no. 11 and no. 12 show a weighted mass of Cu and Fe, which may indicate the starting oxidation of the oil and mechanical wear of parts. Samples no. 7 and no. 8 show an increased amount of Cu and Fe, as in samples no. 6, no. 11 and no. 12. In addition, there is an over-limit amount of P.

Essential chemical elements such as copper, iron and chromium were found in the samples used. Copper (Cu) could get into the hydraulic oil from the pump and gears, iron from the distribution hydraulic pipe, from blocks or casings of individual aggregates, bearings, or transmission. Chromium (Cr) particles can come from bearings, hydraulic pistons, bushings, hardened surfaces, or some seals. The presence of calcium (Ca) can be caused by the operation of this machine in subsoil with different rocks or a high proportion of water in the oil. Iron (Fe) can come from packaging of aggregates, bearings or their pans, hydraulic cylinders and their piston rings, pumps, hydraulic lines, seals, etc. Given that these elements are significantly abrasive, the findings predict damage to the main hydraulic aggregates such as priming pumps, hydraulic motors, distributors, or hydraulic cylinders.

According to the data from Tab. 2, it is therefore possible to evaluate the samples of the collected oil through EDX analysis as follows (Tab. 3). Tab. 3 shows the numbers of the oil samples together with the name of the machine from which it was taken. Next, the engine hours of the given machine are listed with the engine hours worked with the given oil. In the last column, single oils are evaluated as pass or fail.

Tab. 3 *Evaluation of EDX analysis*

Sample no.	**Machine**	**Moto hour status at sample collection**	**Working moto hours of sample taken**	**Rating**
1	VÖGELE S 1800-3i	3 266	1 318	pass
2	CAT D6N XL	7 963	1 914	fail
3	VOLVO G 946	9 977	1 762	pass
4	CAT 352F XE	6 067	1 067	fail
5	VÖGELE S 1900-3i	5 947	2 298	pass
6	BOMAG BW 174AP-4/AM	4 400	2 389	pass
7	BOMAG BW 174AP-4F/AM	3 733	1 733	pass
8	BOMAG BW 120AD-4	6 015	487	pass
9	BOMAG BW 100AD-4	5 959	1 896	fail
10	CAT 323	2 575	2 575	fail
11	BOMAG BW 174AP-4/AM	5 955	1 842	pass
12	BOMAG BW 174AP/AM	11 925	3 896	fail
13	**New Oil -RENOLIN B46 HVI**	-	-	-

2.2 DSC analysis

Different Scanning Calorimetry (DSC) is an analytical technique that is used in the study of thermal properties of materials and their phase transformations such as melting and solidification temperature, heat capacity, thermal effects of material crystallization, polymorphism and decomposition, thermal stability, and enthalpy and also to determine the thermal behavior of polymers, metals, and ceramics. In DSC analysis, the examined sample and the reference oil sample are placed in two separate chambers, which are subsequently exposed to a linearly increasing temperature. During the measurement, the heat consumed, and the heat released from the sample in response to temperature changes are measured. This difference in heat fluxes between the investigated and the reference sample is converted into a signal that is visually displayed as a curve. It shows the thermal response of the investigated sample depending on the temperature [18,19].

DSC analysis was performed on a Mettler Toledo instrument. In the DSC analysis used, sample no. 2. compared with reference sample no. 13. The purpose of this experiment was to compare and possibly evaluate the difference between new uncontaminated (unused) and contaminated (used) hydraulic oil. A comparison of these samples of hydraulic oil (sample No. 2 is marked in red, sample No. 13 in black - Fig. 2) shows that the thermal stability of the new sample of hydraulic oil (sample No. 13) against oxidation is up to a temperature of 262.73 °C and thermal stability of sample no. 2 is up to a temperature of 256.81 °C. Based on this knowledge, it can be concluded that sample no. 2 is contaminated compared to new oil and shows worse thermal stability against oxidation.

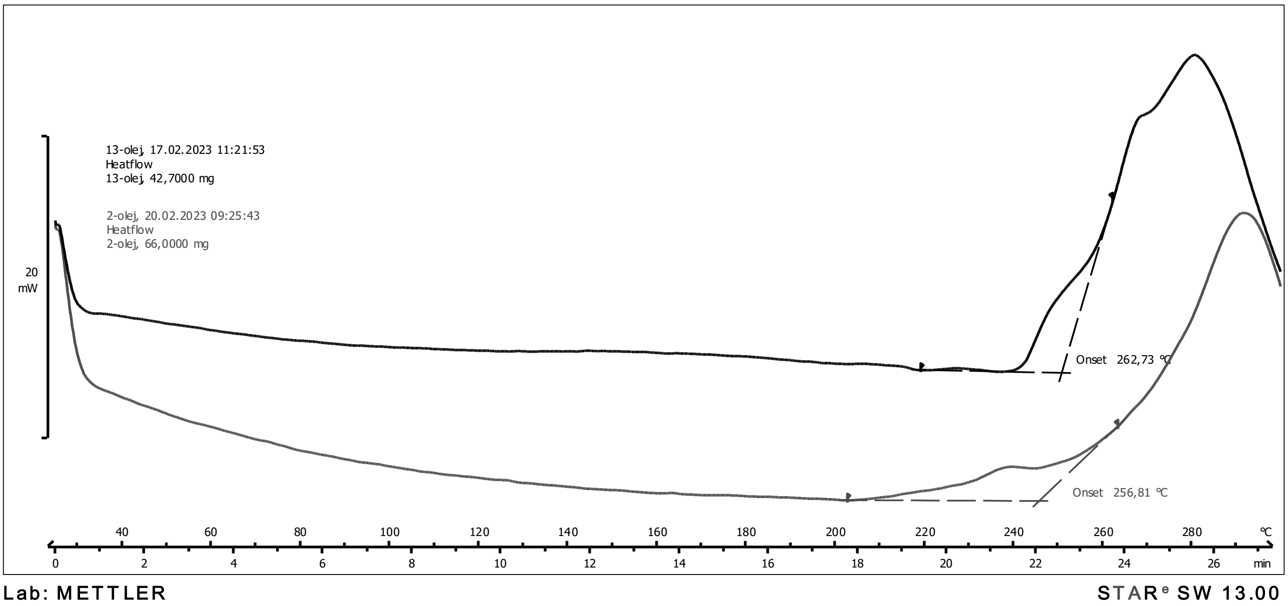


Fig. 2 *DSC analysis of sample no. 2 and sample no. 13*

2.3 Infrared Spectroscopy

Infrared spectroscopy (IR) is classified in the group of non-destructive analytical methods without damaging the examined sample. In this analysis, only a small sample of the investigated substance is used, but it provides full information about its composition. The values of vibrational energies obtained by this spectroscopy are related both to the strength of chemical bonds and to the molecular geometry and masses of nuclei, i.e. with molecular structure. These facts make infrared spectroscopy an excellent experimental technique. In addition to quantitative and qualitative analysis, infrared spectroscopy is an important part of research into molecular dynamics, chemical properties of molecules and other areas [20,21]. In the infrared spectrum, these fields shown in Fig. 3:

- low-temperature antioxidant (NTA) – 3650cm^{-1},
- high-temperature antioxidant (VTA) – 970 and 670 cm^{-1}, a decrease in antioxidants is observed, the content should not fall below 20% of the value of new oil,
- oxidation products (OP) – 1720 – 1690 cm^{-1},
- products of thermal oil degradation - aromatic hydrocarbons (ArU) - 1605 and 815 cm^{-1}, the object of monitoring here is the increase, there is no fixed limit [22,23].

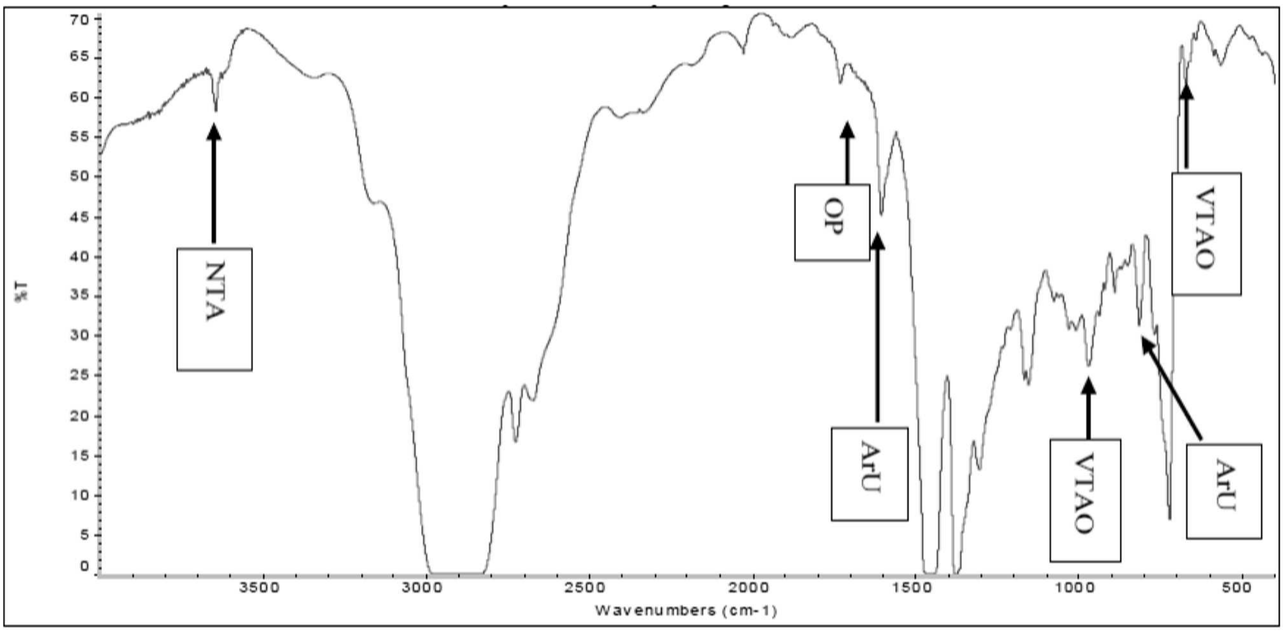
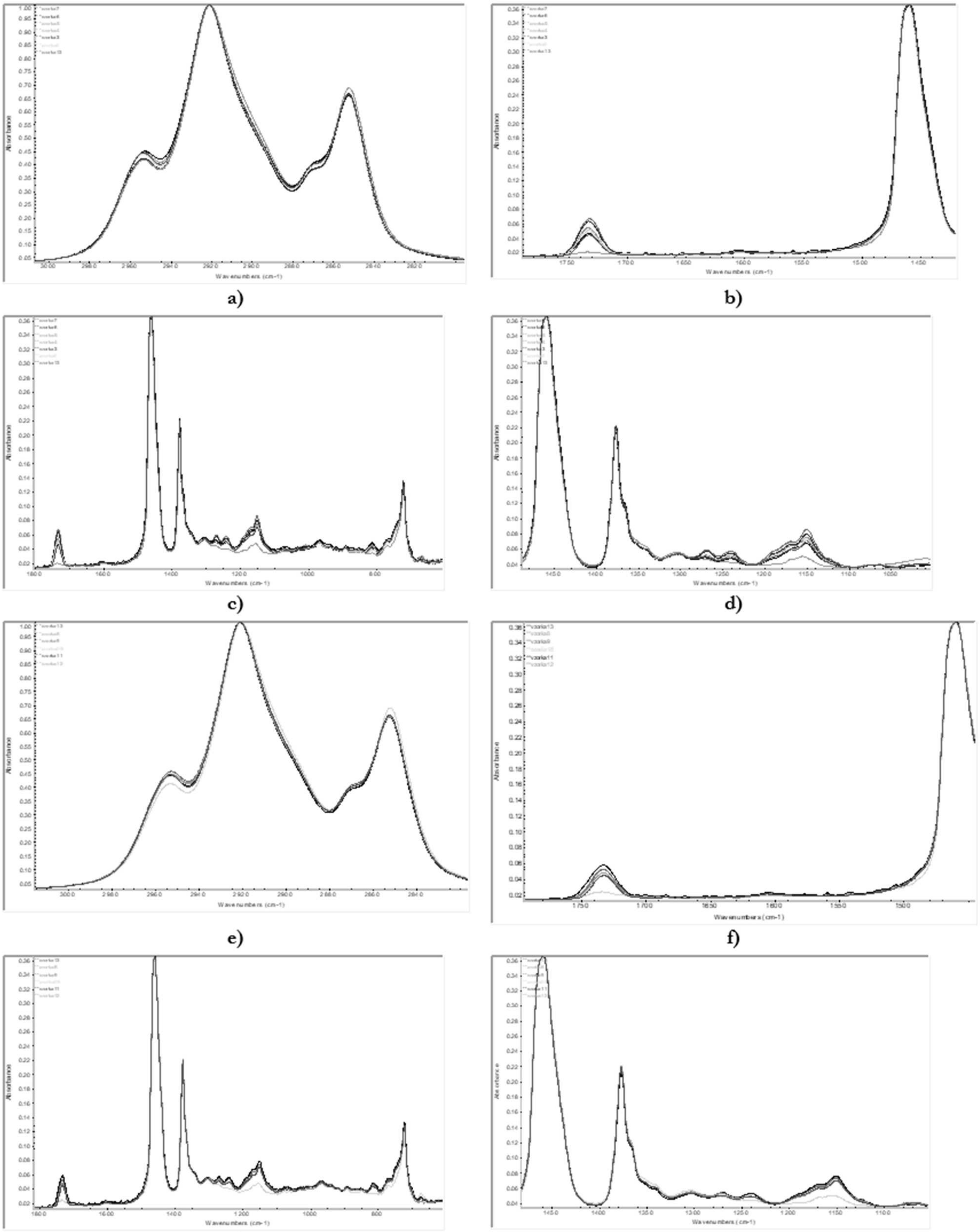
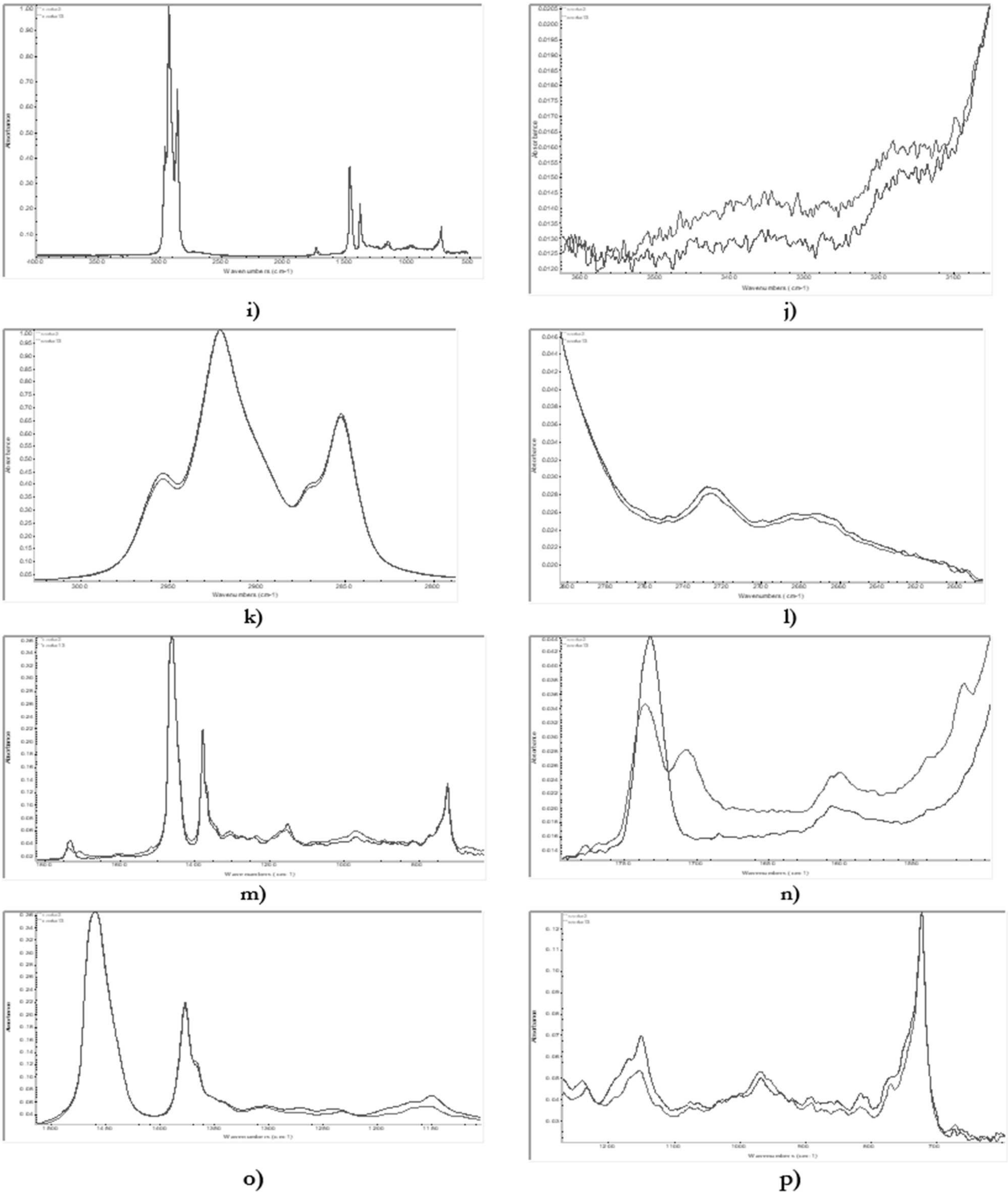


Fig. 3 *Fields of the IR spectrum*



a) b) c) d) e) f) g) h)

Fig. 4/1 *a) Comparison of samples no. 1, 3, 4, 5, 6 and 7 with reference sample no. 13 at 2 800 – 3 000 cm*$^{-1}$*, b) Comparison of samples no. 1, 3, 4, 5, 6 and 7 with reference sample no. 13 at 1 400 – 1 800 cm* $^{-1}$*, c) Comparison of samples no. 1, 3, 4, 5, 6 and 7 with reference sample no. 13 at 600 - 1800 cm*$^{-1}$*, d) Comparison of samples no. 1, 3, 4, 5, 6 and 7 with reference sample no. 13 at 1 000 – 1 500 cm*$^{-1}$*, e) comparison of samples no. 8 to no. 12 with reference sample no. 13 at 2 800-3 000 cm*$^{-1}$*, f) comparison of samples no. 8 to no. 12 with reference sample no. 13 at 1 400 – 1 800 cm*$^{-1}$*, g) comparison of samples no. 8 to no. 12 with reference sample no. 13 at 600 – 1 800 cm*$^{-1}$*, h) comparison of samples no. 8 to no. 12 with reference sample no. 13 at 1 100 – 1 450 cm*$^{-1}$



i) j) k) l) m) n) o) p)

Fig. 4/2 *i) comparison of sample no. 2 with reference sample no. 13 at 500 - 4 000 cm*$^{-1}$*, j) comparison of sample no. 2 with reference sample no. 13 at 3 000 – 3 600 cm*$^{-1}$*, k) comparison of sample no. 2 with a referral sample no. 13 at 2 800 and 3 000 cm*$^{-1}$*, l) comparison of sample no. 2 with reference sample no. 13 at 2 600 – 2 800 cm*$^{-1}$*, m) comparison of sample no. 2 with reference sample no. 13 at 600 – 1 800 cm*$^{-1}$*, n) comparison of sample no. 2 with reference sample no. 13 at 1 500 – 1 800 cm*$^{-1}$*, o) comparison of sample no. 2 with reference sample no. 13 at 1 100 – 1 500 cm*$^{-1}$*, p) comparison of sample no. 2 with reference sample no. 13 at 600 – 1 200 cm*$^{-1}$

Infrared spectroscopy analysis was performed on a Nicolet iS50 FTIR Spectrometer. This spectrometer contains an ATR module with a built-in diamond crystal capable of mid- and far-infrared radiation [24]. The obtained absorbance spectra for individual samples were in the range of 400-4000 cm^{-1}.

In Fig. 4a) and 4e) a comparison of the absorbance of samples no. 1 and no. 3 to no. 12 with reference sample no. 13 in the range of 2800 – 3000 cm^{-1}, an almost identical course can be observed. In the wavenumber range of 2800 – 3000 cm^{-1} in the FTIR spectrum of hydraulic oil, characteristic absorption bands are primarily associated with C-H bonds in hydrocarbons. This region is crucial for oil analysis because it provides valuable information about different types of C-H stretching vibrations, which are typical of the aliphatic hydrocarbons present in the oil. The peak observed between 2950 – 2960 cm^{-1} corresponds to the asymmetric stretching of C-H bonds in CH_3 (methyl) groups. This peak indicates the presence of asymmetric vibrations in methyl groups within aliphatic hydrocarbons. The prominent peak found in the range of 2920 – 2930 cm^{-1} is associated with the asymmetric stretching of C-H bonds in CH_2 (methylene) groups. It is commonly observed in long aliphatic chains that constitute the base structure of hydraulic oils. Additionally, the peak at 2870 – 2885 cm^{-1} represents the symmetric stretching of C-H bonds in CH_3 groups. This provides information about the symmetric stretching vibrations in methyl groups. Another peak in the range of 2850 – 2860 cm^{-1} is characteristic of the symmetric stretching of C-H bonds in CH_2 groups. This peak, like the asymmetric stretching, is indicative of symmetric stretching vibrations in methylene groups. The peaks in this region offer insights into the molecular composition of the hydraulic oil, specifically the ratio and structure of hydrocarbon chains, both saturated and unsaturated. If these peaks remain stable and consistent, it suggests that the oil is maintaining its original properties. However, changes in the intensity or shape of these peaks can indicate contamination, degradation, or alterations in the oil's composition, such as oxidation or the introduction of additives.

Fig. 4b) and 4f) shows a comparison of the absorbance of samples no. 1 and no. 3 to no. 12 with reference sample no. 13 in the range 1400 - 1800 cm^{-1}, where differences in the intensity of the peaks in the region 1750 - 1700 cm^{-1} can be observed. Fig. 4c) and 4g) shows a comparison of the absorbance of samples no. 1 and no. 3 to no. 12 with reference sample no. 13 in the range 600 - 1800 cm^{-1}, where differences in peak intensity can be observed. In this range of the FTIR spectrum of hydraulic oil, several important absorption peaks are observed, each corresponding to specific molecular vibrations and functional groups. The region from 600 – 800 cm^{-1} is typically characterized by fingerprint region vibrations, which are unique to the specific molecular structure of the oil. Peaks in this area can provide information about the presence of various functional groups and structural features within the oil. Between 1000 – 1200 cm^{-1}, the spectrum often shows sulfur-containing compounds such as sulfides and sulfoxides, which are commonly present in hydraulic oils due to the inclusion of anti-wear additives. The peaks in this region are indicative of C-S stretching vibrations. In the range of 1500 – 1600 cm^{-1}, aromatic ring vibrations are observed. These peaks correspond to C=C stretching in aromatic compounds, which can be part of the additives or contaminants in the oil. This region also includes peaks related to C-H bending in aromatic rings, which can help identify the presence of such compounds. A peak around 1725 cm^{-1} is often associated with carbonyl groups (C=O). This peak indicates the presence of oxidized components or degradation products within the hydraulic oil. The intensity of this peak can provide insights into the extent of oxidation. Overall, the 600 – 1800 cm^{-1} range in the FTIR spectrum of hydraulic oil is crucial for identifying various functional groups and potential contaminants. Analyzing these peaks helps in assessing the condition of the oil, determining the presence of additives, and detecting any signs of degradation or contamination.

In Fig. 4d) and 4h) shows a comparison of the absorbance of samples no. 1 and no. 3 to no. 12 with reference sample no. 13 in the range of 1000 – 1500 cm^{-1}, where differences in the range of 1000 – 1300 cm^{-1} can be observed. In this range, peaks typically correspond to C-H bending vibrations, which are indicative of various hydrocarbon structures within the oil. Specifically, CH_2 bending vibrations often appear between 1400 – 1450 cm^{-1}, while CH_3 bending vibrations can be seen around 1370 cm^{-1}. These peaks are characteristic of the aliphatic components present in the oil. Additionally, the spectrum in this region may show peaks related to sulfur-containing compounds. For example, C-S stretching vibrations can appear, often providing information about the presence of anti-wear additives that include sulfur compounds. A peak around 1150 cm^{-1} is commonly associated with C-O stretching in ester or ether groups, which may be present if the hydraulic oil contains such additives or if it has undergone oxidation.

By comparing the absorbance spectra in different ranges from 400-4000 cm^{-1} of sample no. 2 and the reference sample no. 13 (Fig. 4i) to 4p)) differences in the intensity of the peaks can be observed, where a higher intensity means that the given molecular group or functional group that causes this peak is present in a greater concentration in the sample. The intensity of the peak is directly proportional to the amount of the substance that absorbs infrared light at a particular wavelength.

3 Conclusions

With all of these suggested measures, it is necessary to carry out the actions associated with them on a washed and dust-free machine and at the same time in clean and dust-free areas of the repair workshop in order to avoid contamination of the new oil with dust from the surrounding environment in order to prevent its re-degradation.

Nowadays, more than ever, reducing repair costs is a priority for all engineering companies. The issue of reliability and extending the life of machines, machine units and equipment is also largely related to this effort. Cost savings are primarily conditioned by investments in the prevention process. This consists, for example, in diagnostics, which is nothing more than the monitoring of machines and equipment. Tribotechnical diagnostics, which is one of the areas of diagnostics, mainly consists in monitoring the operating loads of machines. In cars, these are primarily engine and transmission oils, therefore timely and ongoing diagnostics allow us not only to check the state of the lubricant during aging, but also the occurrence of foreign substances and other parameters. In addition, these methods are able to detect in time the causes of emerging malfunctions and wear of machine parts, and many times prevent considerable damage or even machine breakdown.

From the measurements of the EDX analysis, it is clear that to prevent or minimization of damage to any of the elements of the hydraulic system is necessary on the stands CAT D6N XL bulldozer (7 963 Mth), CAT 352F XE excavator (6 067 Mth), BOMAG BW 100AD-4 cylinder (5 959 Mth), CAT 323 excavator (2 575 Mth) and cylinder BOMAG BW 174AP/AM (11 925 Mth) first completely clean the entire hydraulic system (also using a suitable flush) and replace all filter elements. These filter elements must be prescribed or approved by the manufacturer of this machine. Subsequently, it is recommended to replace the entire filling of suitable and filtered hydraulic oil and analyze it after working for 250 and then 500 Mth. Based on the results of these analyses, it is necessary to decide on further measures.

In the case of Caterpillar CAT 325F XE excavator with 6 067 Mth worked (sample no. 4) was with a high chromium content, in addition to the above-mentioned activities, it is also necessary to check all hydraulic piston rings with a focus on their surface wear due to abrasion or other mechanical damage that could have occurred during direct contact of some rock with one of the piston rings.

Based on the results of this IR spectroscopy analysis, a check of the nitrogen accumulator is proposed as a corrective measure for the CATERPILLAR D6N XL bulldozer (sample no. 2). Its replacement in case of damage, as well as the replacement of the hydraulic tank ventilation filter. Subsequently, it is necessary to replace the entire oil filling with suitable and filtered hydraulic oil and also to analyze it after working for 250 and then 500 Mth. As with the conclusion of the EDX analysis, it is necessary to decide on further measures based on the results of these analyses.

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