

## Thermal Oxidation Stability of Lubricating Greases

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### ABSTRACT

Four base oils of different chemical nature were subjected to the oxidation processes using the ASTM D 8206–18 method. On the basis of these oils, three different types of lubricating greases were manufactured: lithium, silica and polyurea lubricating greases. The samples of the lubricating greases were also oxidized in accordance with ASTM D 8206–18. The obtained values of thermal oxidation stability were compared. The FTIR spectra were recorded for all the samples after the oxidation process. On the basis of the analysis of the results from the oxidation stability tests and the FTiR spectra (including differential ones), the groups of base oils and lubricating greases produced with their participation that demonstrate the best and the worst resistance to degradation processes as a result of the oxidation reaction, were determined.

**Keywords:** thermal oxidation stability, lithium and silica and polyurea lubricating greases

### INTRODUCTION

Under the operating conditions, lubricating greases are subject to a number of factors that cause their destruction. These factors are shear stresses, pressure, loads, and varying operating conditions, especially temperature changes with simultaneous contact with air. Physical degradation includes all the physical changes in the lubricating grease during use. It is an irreversible process associated with permanent changes in the structure of the lubricating grease. This includes the mechanical processes that cause damage to the structure of the thickener, increase in the base oil separation and evaporation, as well as contamination of the lubricating grease. Chemical degradation includes all chemical reactions within the lubricating grease, such as base oil oxidation, thickener oxidation, and additive depletion. An important issue related to the processes of chemical degradation is the resistance of the lubricating grease to oxidation [1].

Oxidation is the predominant ageing process that directly affects the service life of a lubricating grease. The oxidation process can be presented with a four-stage diagram of radical-chain

reactions [2–4]. This mechanism is referred to as the process of self-oxidation, because the main changes in the structure of oil compounds are the result of the reactions taking place, which are catalyzed by the products of subsequent transformations (Table 1).

Initiation of the oxidation reaction is the result of supplying energy to a hydrocarbon molecule in the form of heat, shear stress, or ultraviolet (UV) radiation. The initiation stage can be catalyzed by metal ions. At room temperature, the reaction rate is very slow, but increases significantly above 100°C. The ease with which radicals can be formed depends on the strength of the C-H and C-C bonds and the stability of the resulting radicals. During the propagation phase, the alkyl radicals react irreversibly with the oxygen dissolved in oil and form the peroxide alkyl radicals. Another reaction that results in the formation of hydroperoxides and other alkyl radicals is removal of the hydrogen atom from the hydrocarbon molecule. The consequence of this is the branching of the chain. Hydroperoxides split into the alkoxy and hydroxyl radicals. The activation energy of these reactions is high and, therefore, they occur at temperatures above 150°C. Radicals

**Table 1.** Oxidation reaction diagram

| No. | Stage       |                                     | Reaction  |
|-----|-------------|-------------------------------------|---|
| 1   | Initiation  | initiation of a chain of reactions  | $R - R \rightarrow R \cdot + R \cdot$<br>$RH \rightarrow R \cdot + H \cdot$<br>$RH + M^{(n+1)+} \rightarrow H^+ + R \cdot + M^{n+}$<br>$M^{n+} + O_2 \rightarrow M^{(n+1)+} + O_2 \cdot^-$  |
| 2   | Propagation | expansion of the chain of reactions | $R \cdot + O_2 \rightarrow ROO \cdot$<br>$ROO \cdot + RH \rightarrow ROOH + R \cdot$<br>$HO_2 \cdot + RH \rightarrow H_2O_2 + R \cdot$<br>$HO_2 \cdot + RH \rightarrow H_2O + RO \cdot$<br>$ROOH + M^{(n+1)+} \rightarrow ROO \cdot + M^{n+} + H^+$<br>$ROOH + M^{n+} \rightarrow RO \cdot + HO \cdot + M^{(n+1)+}$ |
| 3   | –           | branching of the chain of reactions | $ROOH \rightarrow RO \cdot + HO \cdot$<br>$RO \cdot + RH + O_2 \rightarrow ROH + ROO \cdot$<br>$HO \cdot + RH + O_2 \rightarrow H_2O + ROO \cdot$   |
| 4   | Termination | end of the chain of reactions       | $R \cdot + R \cdot \rightarrow R - R$<br>$R \cdot + ROO \cdot \rightarrow ROOR$<br>$ROO \cdot + ROO \cdot \rightarrow ROOR + O_2$   |

react with hydrocarbons to form water, alcohols, as well as more alkoxy radicals. The secondary and tertiary alkoxy radicals form aldehydes and ketones, which combine in an acid-catalyzed aldolic condensation reaction. The condensation products can polymerize, which leads to further degradation as well as formation of slurries and lacquer. The combination of radicals terminates these reactions [5].

Two alkyl radicals can form a hydrocarbon molecule, an alkyl radical can bind to the alkyl peroxide radical, thus producing peroxide, and two alkyl peroxide radicals produce peroxide and oxygen. If metals, such as iron and copper, are present, the reaction can be initiated at a much lower temperature. The metal ions can also catalyze the branching reaction [6] (Table 1).

There are many factors that accelerate the oxidation processes. These include water, metals (such as iron and copper), pollutants, and increased aeration. Probably the most critical factor, however, is temperature. Like in many other chemical reactions, the rate of oxidation also increases with temperature. According to the van't Hoff rule, an increase in temperature by 10 K results in a two- to four-fold increase in the reaction rate. This rule is fulfilled in the case of homogeneous reactions at temperatures up to 500 °C [4, 5, 7].

The methods used in the assessment of resistance of oils and lubricating greases to oxidation enable an analysis of the changes in the chemical structure of oil compounds before and after oxidation. The spectral analysis of infrared (IR) and ultraviolet (UV), gel chromatography, gas chromatography combined with mass spectrometry (GC/MS), and nuclear magnetic resonance

(NMR <sup>13</sup>C) are used for this purpose. These methods can be used to identify various hydrocarbon oxidation products such as alkylhydroperoxides ROOH, dialkyl peroxides ROOR, alcohols ROH, aldehydes RCHO, ketones RCOR<sup>1</sup>, diketones RCOCH<sub>2</sub>COR<sup>1</sup>, ketoaldehydes RCOCH<sub>2</sub>CHO, and hydroxyketones RCH(OH)CH<sub>2</sub>COR<sup>1</sup>.

The thermal oxidation stability of lubricating greases is tested using the classical method of oxidation in a pressure vessel (a so-called bomb) under specific temperature conditions for a specified period of time (usually at 99 or 100 °C for 100 hours) and the test result is given as the value of oxygen pressure drop. The standardized test methods for assessing the thermal oxidation stability of lubricating greases are: BS 2000–142:1993, FTM 791.3453, IP 142:2015, ASTM D 942–15, DIN 51808:2018–02, PN-C-04143:1956 [8–13]. For the first time in 2018, a new standardized method was introduced for the lubricating greases, the so-called rapid oxidation test, in which the time until the induction period (10% oxygen pressure drop) according to ASTM D 8206–18 is given as the result [14].

In their publications, Azad and Evans [15, 16] presented the benefits of combining the results of two research methods: oxidation using the ASTM D 942 method and the FTIR (Fourier Transform Infrared Spectroscopy) analysis when assessing the thermal oxidation stability of lubricating greases. When analyzing the samples of lithium and lithium complex lubricating greases, they pointed to the difficulties in comparing different lubricating greases based on the results obtained using the ASTM D 942 method. The authors also presented the way to obtain more precise information on the resistance of lubricating greases to

oxidation by performing the FTIR analysis on the samples with the modified ASTM D 942 test using smaller numbers of samples.

In his article [17], Edinger presented the results obtained for 11 samples in the oxidation resistance tests carried out with the RapidOxy (Rapid Small Scale Oxidation Test – RSSOT) device at 160 °C according to ASTM D 525 and the classic ASTM D 942 method for lubricating greases (at 99 °C, a 100- and 400-hours test). The comparison of the results allowed the author to conclude that there is a good correlation between these research methods. Nolan, on the basis of the research results presented in article [18], confirmed the existence of a correlation between the RSSOT method and the traditional lubricating grease oxidation resistance test method according to ASTM D 942.

## EXPERIMENTAL PART

The object of the study involved base oils of various chemical nature, as well as lithium, silica, and polyurea lubricating greases manufactured in the NLGI 2 consistency class on the basis of these oils.

## MATERIALS

The following base oils were used:

- A – a mineral paraffin oil of group I according to the API;
- B – a mineral naphthenic oil of group V according to the API;
- C and D – synthetic polyalphaolefin oils of group IV according to the API (1:1 ratio);
- E – a synthetic ester oil of group V according to the API.

The characteristics of these oils are presented in Table 2.

On the basis of the A, B, and E base oils, as well as the C and D oils (1:1 ratio), the following lubricating greases were manufactured: lithium (L), silica (K), and polyurea (P), in consistency 2 according to the NLGI. The lithium grease was produced in a classic saponification process of 12-hydroxystearic acid with lithium hydroxide. The silica grease was produced by dispersing hydrophobic silica in oil. The polyurea grease is produced by dispersing a ready-made thickener in the oil. Twelve samples of the lubricating greases were produced and their basic properties are presented in Table 3.

## TEST METHODS

The oxidation resistance of the lubricating greases was tested according to ASTM D 8206–18. A glass test vessel was filled with 4 g of lubricating grease at ambient temperature; the reaction vessel was initially rinsed with oxygen and then filled to the pressure of 700±5 kPa. The test chamber was heated to 140±0.1 °C. The pressure in the vessel dropped as oxygen was used to oxidize the sample and was recorded at 1 second intervals until the break point was reached – a 10% drop in pressure. The time elapsing from the beginning of the determination to the break point was an inductive period at the test temperature.

The infrared spectra (FTIR) of the lubricating grease samples were recorded with a Thermo Nicolet's IS5 camera. In the case of the transmission spectra, a ZnSe cuvette with a 0.1 mm thick spacer was used, whereas in the case of the ATR spectra, a diamond crystal was used. The spectra were recorded in the range of 4,000 cm<sup>-1</sup> to 550 cm<sup>-1</sup>.

**Table 2.** Determined physicochemical properties of the oils

| No. | Characteristics                                       | Unit               | A     | B     | C                                     | D     | E     |
|-----|---|--------------------|-------|-------|---------------------------------------|-------|-------|
| 1   | Kinematic viscosity [19]<br>- at 40 °C<br>- at 100 °C | mm <sup>2</sup> /s | 86.59 | 107.2 | 29.94                                 | 402.2 | 125.4 |
|     |   |                    | 10.24 | 8.927 | 5.768                                 | 40.58 | 14.40 |
| 2   | Viscosity index [20]                                  | -                  | 99    | 28    | 138                                   | 260   | 155   |
| 3   | Color [21]  | -                  | 1.5   | 2.0   | 0                                     | 0     | 0.5   |
| 4   | Pour point [22]                                       | °C                 | -12   | -30   | -66                                   | -42   | -27   |
| 5   | Acid value [23]                                       | mg KOH/g           | 0.015 | 0.010 | 0.045                                 | 0.145 | 1.569 |
| 6   | Flash point [24]                                      | °C                 | 220   | 214   | 245                                   | 287   | 297   |
| 7   | Resistance to oxidation [14]                          | min                | 420   | 77    | 59<br>(oils C and D in the 1:1 ratio) |       | 22.5  |

**Table 3.** The determined physicochemical properties of the lubricating greases

| Properties of the lithium lubricating greases  | Unit  | LA   | LB   | LCD  | LE   |
|--|-------|------|------|------|------|
| Worked penetration at 25 °C [25]               | mm/10 | 292  | 273  | 279  | 279  |
| Dropping point [26]                            | °C    | 196  | 192  | 198  | 190  |
| Resistance to oxidation [14]                   | min   | 27.3 | 25.8 | 23.5 | 17.5 |
| Properties of the silica lubricating greases   | Unit  | KA   | KB   | KCD  | KE   |
| Worked penetration at 25 °C [25]               | mm/10 | 297  | 259  | 273  | 289  |
| Dropping point [26]                            | °C    | >300 | >300 | >300 | >300 |
| Resistance to oxidation [14]                   | min   | 249  | 92   | 32   | 21   |
| Properties of the polyurea lubricating greases | Unit  | PA   | PB   | PCD  | PE   |
| Worked penetration at 25 °C [25]               | mm/10 | 263  | 277  | 275  | 299  |
| Dropping point [26]                            | °C    | 253  | 242  | 245  | 250  |
| Resistance to oxidation [14]                   | min   | 269  | 217  | 201  | 141  |

### DISCUSSION OF THE RESULTS – OXIDATION RESISTANCE

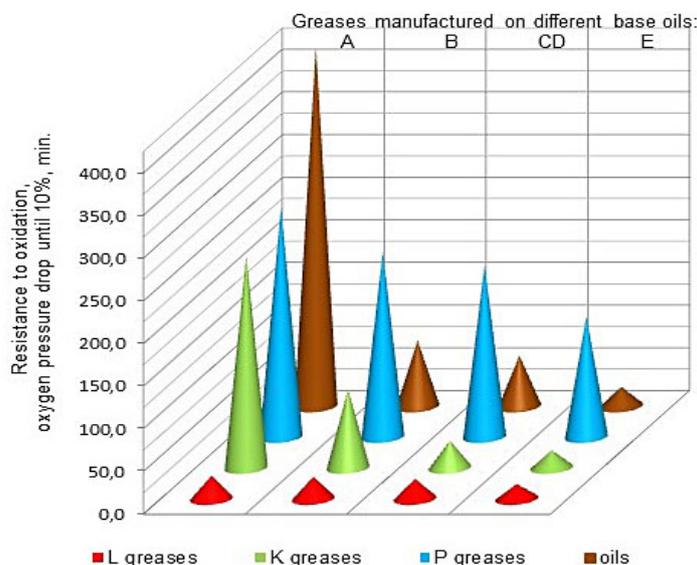
Under the conditions of the oxidation resistance test, the samples were only slightly degraded and the color of the samples did not change significantly. Most likely, during the test performed using the classical test method according to ASTM D942 (which results in a drop in oxygen pressure after 100 h of test), the test conditions would force strong oxidation of the lubricating greases. In the case of determination by accelerated oxidation, the measurement was completed after a 10% drop in pressure (from the maximum pressure) before intensive oxidation of the lubricating grease took place.

It was found that out of all the oils, the group I oil according to the API, i.e. the paraffin oil, was characterized by the best thermal oxidation stability, which is associated with its sulfur content.

The naphthenic and polyalphaolefin oils were characterized by 6 to 7 times lower thermal oxidation stability and the ester oil demonstrated the lowest thermal oxidation stability.

All the lubricating grease samples demonstrated a tendency to reflect the thermal oxidation stability of the oils – the lubricating grease made based on paraffin oil is the most stable, the lubricating grease made on the basis of naphthenic oil is less stable, followed by the lubricating grease made on the basis of PAO, with the ester oil being the least stable.

All lithium lubricating greases samples were characterized by similar, very low resistance to oxidation. In the case of this group of lubricating greases, the use of lithium 12-hydroxystearate as the thickener deteriorated the thermal oxidation stability of the base oils. It is most likely associated with the presence of lithium – a metal that is a catalyst for the oxidation process.



**Fig. 1.** Resistant to oxidation of the lubricating greases and the base oils

In the case of the silica lubricating greases, the use of silica as the thickener slightly improved the thermal oxidation stability of the naphthenic base oil, unlike in the case of the other oils used.

The polyurea lubricating grease produced on the basis of paraffin oil proved to be more susceptible to oxidation than the base oil that was used to produce it. The use of a polyurea thickener in other tested base oils increased their thermal oxidation stability 3 to 6 times.

### DISCUSSION OF RESULTS – SPECTRAL LUBRICATING GREASE DEGRADATION TESTS

The FTIR spectra were recorded and analyzed for all oils and lubricating greases as well as their oxidation products and, based on the results, the differential spectra were generated. The results of the FTIR differential spectra analysis of each of the tested lubricating greases are presented in Table 4. Three spectral ranges were analyzed. The spectral changes that were observed are discussed in detail using the example of the sample of LA – a mineral oil based lithium lubricating grease (Fig. 2).

In the FTIR spectrum of a fresh LA lubricating grease (Fig. 3 and 4, blue line) the COO<sup>-</sup> vibration bands typical for the higher carboxylic acids (lithium soaps) used as salt thickeners, 1,580 cm<sup>-1</sup>, 1,560 cm<sup>-1</sup>, 1,405 cm<sup>-1</sup>, and 1,340 cm<sup>-1</sup>,

and weak bands, 1,734 cm<sup>-1</sup> and 1,715 cm<sup>-1</sup>, originating from other carboxylic or carboxylic compounds, were observed. The spectrum also shows the hydrocarbon structure bands in the range of 3,000–2,800 cm<sup>-1</sup>, 1,450 cm<sup>-1</sup>, 1,377 cm<sup>-1</sup>, and 721 cm<sup>-1</sup>. At the same time, a wide band of the associated hydroxyl groups of about 3,330 cm<sup>-1</sup> is present.

In the LA lubricating grease spectrum, after the oxidation resistance test (Figs. 3 and 4, red line), a wide, strong band with a complex structure in the range of 1,800–1,660 cm<sup>-1</sup>, with a maximum of approx. 1,713 cm<sup>-1</sup>, typical of carboxylic acid structures, appeared. At the same time, the background was raised in the range of 1,300–1,000 cm<sup>-1</sup>, which indicated the formation of complex oxidation products containing the C-O bonds (alcohols, ethers, peroxides, carboxylic acids, and their derivatives). Due to the increase in the number of hydroxyl groups, the band intensity increased to approx. 3,340 cm<sup>-1</sup>.

These observations were confirmed by the analysis of the differential spectrum (Fig. 5), on which it is additionally possible to distinguish bands in the range of 1,200–1,000 cm<sup>-1</sup> connected to the structures containing single C-O bonds.

Analogous analyses were performed for other lubricating greases; however, it should be noted that for the lubricating greases containing the ester base oil (E) the analysis of the spectral range of 1,800–1,660 cm<sup>-1</sup> was difficult due to the very strong ester band of approx. 1,745 cm<sup>-1</sup>.

**Table 4.** Bands observed in FTIR differential spectra related to the oxidation processes of the tested lubricating greases

| Lubricant sample | Position of the bands, cm <sup>-1</sup> ; band intensity, abs/0.1 mm |  |             |  |
|------------------|--|--|-------------|--|
|                  | Bands in the range of 3,600–3,100 cm <sup>-1</sup>                   | Bands in the range of 1,800–1,660 cm <sup>-1</sup> |             | Bands in the range of 1,200–1,000 cm <sup>-1</sup> |
| LA               | 3,470/0.065  |  | 1,715/0.232 | 1,090/0.284  |
| LB               | 3,488/0.022  |  | 1,712/0.286 | 1,010/0.013  |
| LCD              | 3,506/0.030  | 1,749/-0.126                                       | 1,713/0.294 | 1,046/0.010  |
| LE               | 3,450/0.192  | 1,743*/-   | 1,719/0.983 | 1,165/-0.388                                       |
| KA               | 3,445/0.040  |  | 1,715/0.152 | -**  |
| KB               | 3,410/0.029  |  | 1,711/0.235 | -**  |
| KCD              | 3,405/0.054  |  | 1,712/0.226 | -**  |
| KE               | 3,516/0.095  | 1,746*/-   | 1,710/0.334 | -**  |
| PA               | 3,456/0.096  |  | 1,716/0.156 | 1,154/0.072  |
| PB               | 3,400/0.031  |  | 1,714/0.161 | 1,120/0.046  |
| PCD              | 3,465/0.031  | 1,748/-0.081                                       | 1,717/0.164 | 1,094/0.017  |
| PE               | 3,305/0.103  | 1,746*/-   | 1,716/0.179 | 1,074/0.140  |

\* Too strong bands in the transmission spectrum, the bands in the ATR differential spectrum were evaluated together with the indication whether the change in band absorbance is positive or negative.

\*\* Due to the very strong bands derived from SiO<sub>2</sub>, it was not possible to assess the bands in the 1,200–1,000 cm<sup>-1</sup> range.

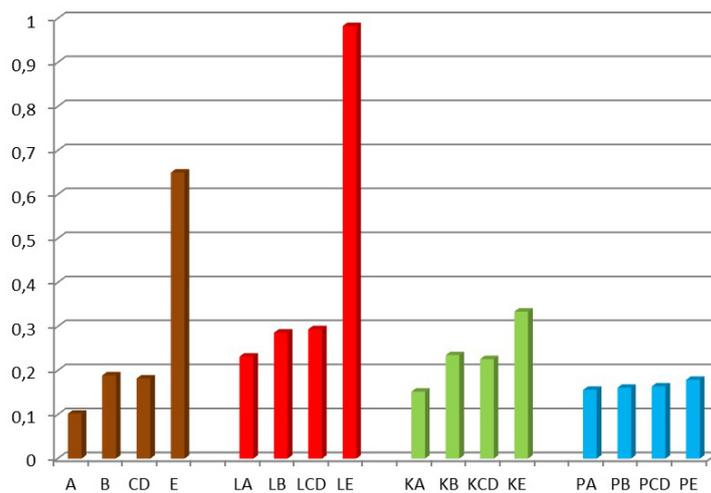


Fig. 2. Band intensity approx. 1,715 cm<sup>-1</sup>, abs/0.1 mm in the differential spectrum

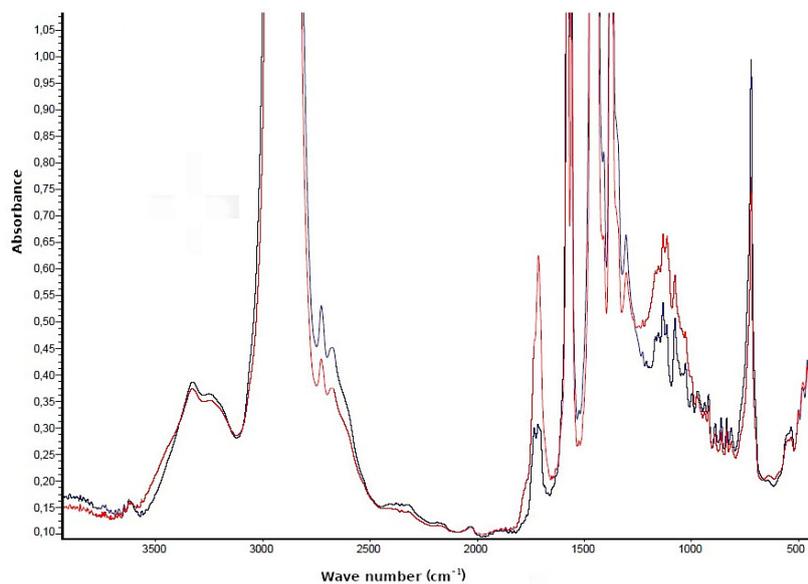


Fig. 3. FTIR spectra of the LA lubricating grease before (blue line) and after (red line) oxidation

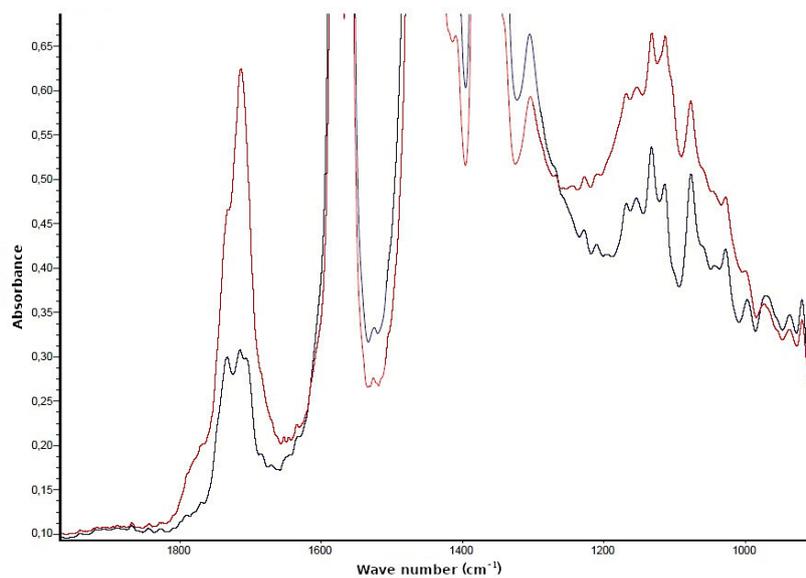


Fig. 4. FTIR spectra in the range of 2,000–1,000 cm<sup>-1</sup>, of the LA lubricating grease before (blue line) and after (red line) oxidation

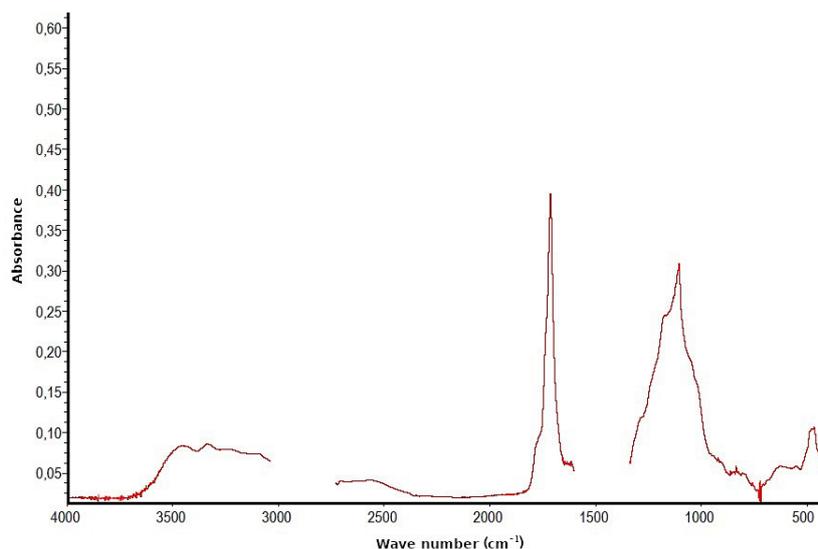


Fig. 5. Differential spectrum of the LA lubricating grease before and after oxidation

The formation of carboxylic structures (bands of about 1,715  $\text{cm}^{-1}$ ) was evaluated on the basis of the differential spectrum obtained by subtracting transmission spectra, whereas the analysis of the ATR spectra confirmed the loss of ester structures.

However, in the case of the lubricating greases containing silica (K) as a thickener, the analysis of the range of 1,200 – 1,000  $\text{cm}^{-1}$  was difficult due to very strong  $\text{SiO}_2$  bands that obscured the possible C-O bond vibration bands.

The results obtained for the base oils (Table 5) indicate that the mineral oil (A) undergoes the lowest degradation during the determination, the degree of degradation of the oils (B and CD) is slightly higher, and in the ester oil (E) a decrease in the intensity of ester bands with simultaneous strong oxidation of the oil was observed.

In the case of the lithium and silica lubricating greases, the weakest degradation during the test was observed in the paraffin oil-based lubricating grease (A). A similar degree of degradation was observed in the lithium (L) and silica (K) lubricating greases containing the B and CD

oils, while the ester (E) lubricating greases were oxidized to the greatest extent. In the case of the polyurea lubricating greases, all samples were similarly oxidized.

### CONCLUSIONS

1. Out of all the tested oils, the group I oil according to the API, i.e. the paraffin oil, was characterized by the best thermal oxidation stability, which is associated with its sulfur content. The naphthenic and polyalphaolefin oils were characterized by 6 to 7 times lower thermal oxidation stability and the ester oil demonstrated the lowest thermal oxidation stability.
2. The spectral analysis confirmed that the mineral paraffin oil is the most resistant to oxidation by the ASTM D 8602–18 method, while the ester oil is the least resistant.
3. All the lubricating grease samples demonstrated a tendency to reflect the thermal oxidation stability of the oils – the lubricating grease

Table 5. Bands observed in FTIR differential spectra related to the oxidation processes of the tested oils

| Oil sample | Position of the bands, $\text{cm}^{-1}$ ; band intensity, $\text{abs}/0.1 \text{ mm}$ |  |             |  |             |
|------------|---|--|-------------|--|-------------|
|            | Bands in the range of 3,600–3,100 $\text{cm}^{-1}$                                    | Bands in the range of 1,750–1,680 $\text{cm}^{-1}$ |             | Bands in the range of 1,200–1,000 $\text{cm}^{-1}$ |             |
| A          | 3,380/0.015   | 1,717/0.102  |             | 1,172/0.042  | 1,045/0.064 |
| B          | 3,410/0.041   | 1,713/0.189  |             | 1,165/0.077  | 1,120/0.078 |
| CD         | 3,405/0.051   | 1,748/-0.255                                       | 1,714/0.182 | 1,155/-0.050                                       |             |
| E          | 3,450/0.134   | 1,742*/-   | 1,716/0.650 |  | 1,065/0.111 |

\* Too strong bands in the transmission spectrum, the bands in the ATR differential spectrum were evaluated together with the indication whether the change in band absorbance is positive or negative.

made based on paraffin oil is the most stable, the lubricating grease made on the basis of naphthenic oil is less stable, followed by the lubricating grease made on the basis of PAO, with the ester oil being the least stable.

4. The spectral studies confirmed that in the case of the lithium and silica lubricating greases, the ester oil-based lubricating greases were characterized by the worst resistance to oxidation. In the case of the polyurea lubricating greases, this analysis does not allow distinguishing the level of degradation depending on the base oil used.

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